Determination of As, Cr, and Cu Concentrations in CCA-treated Wood Poles Using Acid Decomposition in Closed Flasks, Oven Heating, and ICP-MS Analysis

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ABSTRACT

To prevent the degradation of wood used for utility poles, a chromated copper arsenate (CCA) preservative is introduced to increase the wood's resistance to degradation. The components of CCA are highly toxic and may leach into the environment, contaminating the soil as well as surface and groundwater. The aim of the present study was to determine the As, Cr, and Cu concentration in samples of CCA-treated wood poles using acid decomposition in closed flasks, oven heating, and the ICP-MS technique. For this purpose, milled wood samples (particle size $\leq 425 \mu\text{m}$) were decomposed in a mixture of concentrated HNO_3 and H_2O_2 for 24 hours at 95°C . The reference material NIST SRM 1575a Trace Elements in Pine Needles was used to evaluate the accu-

racy of the method, and the results obtained for As, Cr, and Cu were in agreement with the certified values. For the analysis of real samples, wood poles with different characteristics were used: poles submitted to impregnation under laboratory conditions, poles exposed to industrial treatment with preservatives, and poles removed from service. In the comparison of the results with those obtained for decomposition with microwave heating, the paired *t*-test showed no significant difference between the two methods. In addition, linear regression of the data yielded a high *R* value and the slope of the regression close to 1 for all of the elements, indicating that the proposed method is nearly identical with microwave heating for the determination of As, Cr, and Cu in wood pole samples.

which has been used since the 1950s, is to-date the most widely applied wood preservative worldwide. It is accepted as one of the most effective treatments for protection of wood against fungi, insects, and marine borers (7).

In the CCA treatment process, wood products such as dimensional lumber, plywood, and poles are preserved by impregnating the wood with an aqueous solution containing CrO_3 , CuO , and As_2O_5 in a pressurized treating cylinder. After addition of the preservative, the wood is removed from the cylinder and allowed to dry. Arsenic and copper act as biocides, and chromium acts as a "fixing" agent to bind the metals to the wood. The CCA fixation process refers to the chemical reactions that take place when hexavalent chromium is reduced to trivalent chromium in the wood (8).

The composition of CCA is typically 34% chromic oxide, 13% cupric oxide, 25% arsenic pentoxide, and 28% water and inert materials (9).

From a commercial standpoint, CCA is an excellent alternative to increase wood durability, evidenced by numerous reports proving its efficiency. Nevertheless, from an environmental and public health perspective, its use is controversial due to its high toxicity. The components present in this preservative, particularly As and Cr, are highly toxic and several countries have restricted its use. These restrictions

INTRODUCTION

Wood is employed for a variety of purposes due to its numerous advantages and utilities (1). One of the most important uses of wood is in construction, and particularly in the United States, Canada, Australia, and South Africa also for the manufacture of utility poles. In Brazil, it is used for this purpose primarily in the southern part of the country (2).

The biological origin of wood sets it apart from other industrial materials; however, its major drawback is its tendency to decay (3–4). The agents responsible for deterioration can be physical, chemical, or biological in nature and include factors such as heat, fire, pollution, humidity, climate effects, the presence of highly acidic or basic substances, friction with other materials and biological agents. Of these, biological agents such as fungi, bacteria, and insects are considered the most harmful (5–6).

The wood preservative of chromated copper arsenate (CCA),

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are based on the loss of CCA components from wood over time through leaching or volatilization, leading to contamination risks for humans and the environment (10). In addition to the occupational and environmental problems related to the production and use of CCA-treated wood, an even greater challenge is the final disposal of the waste generated at the end of its useful life. Utility poles removed from service are an example of this type of waste since they cannot be discarded together with other solid waste, owing to the danger involved (2, 11–12). As such, it is essential to develop efficient methods for quantifying As, Cr, and Cu in CCA-treated poles and using these data to devise treatment and removal methods that enable adequate disposal of out-of-service poles.

The quantification of As, Cr, and Cu in wood treated with CCA and other preservatives is typically achieved using X-Ray fluorescence (XRF), flame and graphite furnace atomic absorption spectrometry (FAAS and GFAAS), inductively coupled plasma optical emission spectrometry (ICP-OES), and inductively coupled plasma mass spectrometry (ICP-MS) (13–21). The ICP-OES technique is recommended by the American Wood Preservatives Association – AWWA (A21-08, 2008) for analyzing treated wood and its solutions (22). However, in contrast to ICP-OES, ICP-MS is also multi-elemental, offers a wider dynamic range, significantly lower detection limits, and the ability to measure isotopic ratios; therefore, ICP-MS has wider fields of application (23–24).

Methods used to prepare wood samples for subsequent quantification of Cu, Cr, and As are underreported in the literature. Geng et al. (25) described an analytical methodology developed to quantify As in coal samples using oxygen

flask combustion (OFC), followed by hydride generation atomic absorption spectrometry (HG-AAS), subsequently using the same technique to quantify this element in CCA-treated wood wastes.

The American Wood Protection Agency (AWPA) recommends four decomposition procedures to prepare wood (AWPA A7-04) for chemical analysis (26), two in an open system using Erlenmeyer flasks and two in a closed system with a microwave oven. However, none of these proposed decomposition methods describe the type/species of wood, particle size, extraction time, or temperature used, merely indicating the reagents employed and the mass of the sample. As such, the objective of this study was to develop a method to determine As, Cr, and Cu in samples of CCA-treated wood poles using acid decomposition in closed flasks with heating in an oven and ICP-MS analysis. The comparison of the results with a method employing microwave heating is also presented.

EXPERIMENTAL

Instrumentation

All measurements were performed using the ELAN® 6000 inductively coupled plasma mass spectrometer (PerkinElmer, Inc., Shelton, CT, USA). Argon (99.996%, White Martins, São Paulo, Brazil) was used as the carrier gas. The instrumental parameters are listed in Table I. Instrument optimization was achieved by adjusting the gas flow rate of the nebulizer and aligning the mass spectrometer with the torch (x-y adjustment) in order to achieve maximum M^+ and minimum M^{++} , MO^+ ion production and background signal at $m/z = 220$ using a conventional pneumatic nebulizer. The mathematic correction of $-3.127^*(Se\ 77 - (0.874*Se\ 82))$ was applied to correct the spectral interference of the polyatomic $^{40}Ar^{35}Cl$ ion under ^{75}As measurement.

The wood samples were ground using two different analytical mills: A11 IKA - Basic (IKA, Germany) and TE 600 (Tecnal, Brazil).

TABLE I
ICP-MS Instrumental Operating Parameters

Instrument	PerkinElmer® ELAN® 6000 ICP-MS
RF power	1100 W
Gas flow rate:	
Plasma	15 L min ⁻¹
Auxiliary	1.2 L min ⁻¹
Nebulizer	0.90 L min ⁻¹
Sampler and skimmer	Pt
Sweeps per reading	3
Reading per replicate	1
Replicates	5
Unit of measurement	Counts per second (cps)
Isotopes	⁶³ Cu, ⁵³ Cr, ⁷⁵ As
Signal measurement	Peak hopping
Resolution	0.7 amu (10% of peak height)
Spray chamber	Scott-type
Nebulizer	Cross-flow
Auto lens	On

Sample decomposition was carried out in a drying oven with exhaust (MA 035 – Marconi, Italy) and in a microwave oven (ETHOS PLUS – High Performance Microwave, Milestone, Italy).

Reagents and Certified Material

All glassware and polypropylene flasks were previously washed with a 5% (v/v) neutral detergent solution (EXTRAN®, Millipore Corporation, Bedford, MA, USA), then decontaminated using a 10% (v/v) HNO₃ solution, and rinsed abundantly with deionized water. The PTFE flasks used in the oven and in the microwave were decontaminated with the same acid mixture used for sample decomposition. This was carried out in accordance with the equipment cleaning procedure for microwave and, in the case of decomposition in the oven, following the same decomposition procedure applied for the samples.

All reagents used were of analytical reagent grade. Nitric acid and hydrogen peroxide (Merck, Darmstadt, Germany) were used. Nitric acid was sub-boiling distilled in a quartz still (Kürner Analysentechnik, Rosenheim, Germany). The water was purified in a Milli-Q® system (Millipore Corporation USA), resulting in water with a resistivity of 18.2 MΩ.cm. The multi-element calibration solutions were prepared by appropriate dilution of 1000 mg L⁻¹ single-element Titrisol® stock solutions (Merck) in 5% (v/v) HNO₃. The acid concentration in the calibration solutions was varied in order to match that of the sample solution. A rhodium solution at a concentration of 5 µg L⁻¹ was used as the internal standard to minimize instrument fluctuations during the measurements.

In order to determine the accuracy of the method, the certified reference material SRM 1575a Trace Elements in Pine Needles (National Institute of Standards and

Technology, USA), with a granulometry of < 100 µm, was used. This material was chosen because it showed greater similarity to the wood under study, also no other material could be found that exhibited similar characteristics. The present study used wood pole samples with different characteristics such as poles submitted to impregnation under laboratory conditions (see sample impregnation method), poles exposed to industrial treatment with preservatives, and poles removed from service.

Sample Impregnation Method

A Tanalith® CCA solution (purchased from Arch Química Brasil Ltda, Brazil), with a composition of 28.70 % CrO₃, 11.17% CuO, and 20.54% As₂O₅, was used for sample impregnation. Wood impregnation was carried out using two different methods: (a) The first employed a 3.5% (w/w) CCA solution with the same concentration used in industry, and prepared by dilution of the solution as described above. For this purpose, approximately 30 small samples of Eucalyptus (species unknown) were selected and dried in an oven at 95 °C ± 5 °C for four hours. After cooling, the samples were divided into groups of three, weighed to obtain almost equal mass, and transferred to beakers. Then 75 mL of CCA solution (3.5% w/w) was added. The beakers were covered with Parafilm and agitated for one hour. After the excess solution was removed, the samples were left in a dry environment with controlled temperature and humidity (23 °C and 65% relative humidity) for two weeks. At the end of this period, they were ground and decomposed in an oven.

(b) The second impregnation procedure employed a more diluted CCA solution (1% w/w) (27), and dilution of the same solution aforementioned to treat samples of new poles from three different species. Two were from

the Eucalyptus genus (*E. saligna* and *E. grandis*) and one from the Corymbia genus (*C. citriodora*). A total of 14 samples were used for each impregnation in blocks measuring 19x19x19 mm. For the *C. citriodora* species, five different regions of the pole were impregnated (A, B, C, D, and E), whereas only three areas (B, C, and D) were selected for the other two species (Figure 1) (28). The total number of impregnations was 11.

Prior to impregnation, the samples were allowed to dry for two weeks at a temperature of 23 °C and 65% relative humidity. After weighing, the samples (blocks) underwent vacuum impregnation, as shown in Figure 2. An initial vacuum process was performed to open the wood fibers and facilitate the introduction of the preservative. A CCA solution (1% w/w) was then gradually introduced until all 14 blocks were submersed, after which the vacuum was slowly removed over a period of 15 minutes. Next, the beaker containing the samples was removed and allowed to rest for 30 minutes. The excess preservative was then extracted and the samples were weighed and stored for two weeks to finalize the binding process. The same procedure was repeated for the 10 remaining groups of blocks.

Sample Decomposition Method

A hammer and chisel were used to reduce the size of the wood samples, which were then ground in mills. CO_{2(s)} was used during the grinding process to avoid possible losses due to volatilization of the elements.

Sample decomposition with oven heating was based on an adaptation of the American standard for wood preservatives (26). The influence of parameters such as decomposition time (2, 6, 12, and 24 hours), sample amount (0.10, 0.25, and 0.50 g), and temperature (70,

95, and 150 °C) were investigated. For all optimizations, decomposition was carried out in closed PTFE flasks. After grinding, the samples were weighed, transferred directly to the flasks, and treated with 8 mL of HNO₃ and 3 mL of H₂O₂. The flasks were left half open to allow the vapors to escape and then submitted to heating. After cooling, the resulting solutions were quantitatively transferred to polypropylene flasks, and deionized water was added until a volume of 50 mL was

reached. The samples were diluted with 5% (v/v) nitric acid before analysis by ICP-MS.

For decomposition in the microwave oven, the samples were weighed to an approximate mass of 0.10 g for the wood sample or 0.20 g for the certified reference material, and a mixture of 5 mL of HNO₃ and 2 mL of H₂O₂ was added. The samples were then heated in accordance with the manufacturer's recommendations (Table II). After cooling, the resulting solutions

were quantitatively transferred to the polypropylene flasks and the volume was adjusted with deionized water. The samples were diluted with 5% (v/v) nitric acid before ICP-MS analysis.

TABLE II
Heating Program of Microwave Oven Used for Sample Digestion

Stage	Time (s)	Temp. (°C)
1	150	90
2	300	130
3	210	200
4	900	200
Cooling	-	-

RESULTS AND DISCUSSION

Decomposition Methodology for the Samples Using PTFE Flasks and Oven Heating

Several factors were investigated to ascertain their influence on sample decomposition in closed flasks with oven heating such as decomposition time, sample quantity, and decomposition temperature. For optimization of the decomposition time and sample quantity, samples of Eucalyptus (species unknown) impregnated with 3.5% (w/w) CCA solution were used. Since the majority of samples analyzed in this study were from the Eucalyptus genus, the results obtained for the optimizations presented below can be considered representative for all of the samples. It should be noted that *C. citriodora* is generally known as *Eucalyptus citriodora*.

For all optimizations, a particle size of ≤ 425 μm was used. According to studies reported in the literature, in general the particle sizes of the analyzed treated wood samples ranged between 425 and 213 μm (21, 27, 29–30). The choice of this particle size may be due to the difficulty in grinding the samples and

Fig. 1. Schematic highlighting the regions where samples (blocks) were collected from the new poles.

Fig. 2. Vacuum impregnation apparatus; (A) vacuum desiccator; (B) beaker for treatment; (C) test wood blocks; (D) treating solution; (E) polyethylene tubing; (F) three-way stopcock; (G) vacuum pump; (H) flask for vacuum trap; (I) stopcock to atmosphere; (K) vacuum source line.

the high toxicity exhibited primarily by As in the sample. Working with larger particle sizes reduces excessive handling of the sample, thereby lowering preparation time.

Influence of Decomposition Time

Figure 3 shows the influence of time on the extraction of As, Cr, and Cu. The results suggest that a higher decomposition time favored the extraction of the three elements, causing a gradual increase in their concentration.

The same figure also indicates that a decomposition time of 24 hours increased the standard deviations, particularly for As and Cr. The results obtained for 12 hours decomposition were similar to those recorded at 24 hours (increases of 0.7% for Cu, 2.4% for Cr, and 6.4% for As); however, several wood fragments remained in the solution after 12 hours of heating. A longer decomposition time (48 hours), tested on another type of impregnated sample (result not shown), did not cause a significant increase in the element concentrations. We also observed a substantial reduction in the standard deviation at 24 hours compared to 48 hours, possibly due to volatilization losses at longer times. A decomposition time of 24 hours

was therefore chosen for subsequent tests.

Influence of Sample Quantity

Figure 4 shows the effect of sample quantity on extraction of the elements. The highest values were obtained with a mass of 0.25 g for all elements. Concentration values were lower for Cu and Cr for a mass of 0.50 g, and the standard deviation between the replicates was higher than that recorded for the other masses tested. This result can be related to the smaller contact surface between the sample and the acid, leading to less efficient element extraction and lower repeatability. Additionally, the resulting solution after decomposition using a mass of 0.5 g showed high turbidity, indicating incomplete decomposition of the sample.

Influence of Decomposition Temperature

For investigation of this effect, a wood sample from an out-of-service pole (Eucalyptus genus) was analyzed. As shown in Figure 5, the temperature exhibited no significant influence on extraction of the elements. Although the concentrations obtained for Cu and As were similar at 70 and 95 °C, the standard deviations were lower at the latter temperature. Moreover,

a temperature of 70 °C resulted in incomplete sample decomposition with several fragments of wood remaining in the solution. At 150 °C, the solution had almost entirely evaporated, so the decomposition was completed in 6 hours. As a result of these experimental observations, the temperature of 95 °C was chosen.

Analysis of the Certified Reference Material

To determine the accuracy of the methodology developed, it was applied to the decomposition of the certified reference material NIST SRM 1575a Trace Elements in Pine Needles (sample mass of 0.25 g, decomposition time of 24 hours, and decomposition temperature of 95 °C). The certified material was also decomposed by microwave heating in accordance with the AWWA standard for wood samples (26) to establish a comparison between the methods. The results of the analyses are listed in Table III.

The concentration values found for Cu and Cr were in line with the reference material and informed values, respectively. The value obtained for As was larger than that of the reference material, but the Student's *t*-test (31) showed that the difference was not significant for a confidence interval of 95%,

Fig. 3. Influence of time on the extraction of As, Cr, and Cu. Analysis of the wood sample impregnated with CCA solution (3.5% w/w) by ICP-MS. Each value represents the mean of three determinations.

Fig. 4. Influence of sample quantity on the extraction of As, Cr, and Cu. Analysis of the wood sample impregnated with CCA solution (3.5% w/w) by ICP-MS. Each value represents the mean of three determinations.

Fig. 5. Influence of temperature on the extraction of As, Cr, and Cu. Analysis of the wood sample from out-of-service pole by ICP-MS. Each value represents the mean of three determinations.

TABLE III
ICP-MS Analysis of NIST 1575a Trace Elements in Pine Needles Using Acid Decomposition With Oven Heating and Microwave Heating

Element	Oven ($\mu\text{g g}^{-1}$)	Microwave ($\mu\text{g g}^{-1}$)	Certified ($\mu\text{g g}^{-1}$)
Cu	2.60 (0.04) ^a	2.60 (0.01)	2.8 ^b (0.2)
Cr	0.40 (0.04)	0.50 (0.08)	0.3–0.5 ^c
As	0.048 (0.013)	0.038 (0.005)	0.039 ^d (0.002)

^a Values in parentheses are standard deviations of three determinations (n=3).

^b Certified value. ^c Informed value. ^d Reference value.

TABLE IV
Total Oxides Obtained by Oven Decomposition and ICP-MS Measurement for Different Eucalyptus Species During Vacuum Impregnation Using 1% (w/w) CCA Solution

Concentration (kg m ⁻³)				
Sample	CuO	CrO ₃	As ₂ O ₅	Total Oxides
<i>C. citriodora</i>				
A	0.32	1.93	1.15	3.41
B	0.31	1.63	1.60	3.54
C	0.30	2.10	1.26	3.66
D	0.24	2.03	1.29	3.56
E	0.32	2.16	1.33	3.81
<i>E. grandis</i>				
B	0.21	1.41	1.41	3.03
C	0.48	2.82	0.65	3.95
D	0.45	2.38	0.58	3.41
<i>E. saligna</i>				
B	0.19	1.76	0.52	2.47
C	0.26	1.91	0.57	2.74
D	0.25	1.77	0.51	2.52

confirming the efficiency of the proposed methodology.

The concentration values obtained for As, Cr, and Cu by microwave heating were also in agreement with those provided by the NIST sample. It should be noted that the low concentration value for As present in the SRM 1575a Trace Elements in Pine Needles justifies use of the ICP-MS technique for these determinations.

Figures of Merit

The limit of detection (LOD) was calculated as three times the standard deviation (3σ) of 10 consecutive measurements of the blank signal divided by the slope of the calibration curve. For 0.25 g of sample in a 50-mL solution, the detection limits were 0.006, 0.007, and 0.003 $\mu\text{g g}^{-1}$ for Cu, Cr, and As, respectively. It is noteworthy that the correlation coefficients (R) were all above 0.9999 in the calibration range investigated (1 to 100 $\mu\text{g L}^{-1}$). The limits of detection (LOD) of all analytes were lower than the lowest concentrations obtained for each analyte in the decomposed samples. The relative standard deviations (RSD) were lower than 10% for all of the elements.

Wood Sample Analysis

In order to characterize the samples impregnated in the laboratory in terms of retention rate (total oxides) and also the effectiveness of the impregnation along the pole, the samples coming from three species (*C. citriodora*, *E. grandis*, and *E. saligna*) were submitted to the developed decomposition methodology. Table IV shows the concentration values of the individual oxides and also the total oxides obtained for the three different species.

It was found that impregnation generally occurred evenly along the pole. Moreover, retention was simi-

lar for *C. citriodora* and *E. grandis*, likely due to the similar densities (763 kg m^{-3} and 819 kg m^{-3} , respectively) of these species. Despite indicating uniform distribution along the pole, the value for *E. saligna*, whose density was 586 kg m^{-3} , was lower in relation to the other species.

To evaluate the accuracy of the results obtained by the method developed in this work, section B of each of the impregnated species was sent to the laboratory of the São Paulo Institute of Research Technology (IPT), a benchmark in wood analysis in Brazil. Table V shows this comparison, indicating a high degree of similarity between

the results. Relative error (related to total oxides), calculated using the values found by IPT as reference, was lower than 10% for all samples, except *C. citriodora*, indicating agreement between the results. The reason for the difference between the total oxides values in *C. citriodora* is, principally, because the As_2O_5 concentration found by IPT is lower than that obtained by this method. Since IPT determines As by AAS, some losses could occur during the procedure due to As volatilization.

The optimized methodology was applied to the analysis of different wood samples: (a) one pole removed from service, (b) two samples from new poles (1 and 2) industrially impregnated with 3.5% (w/w) CCA solution (under pressure in an autoclave), (c) three samples from new poles (*C. citriodora*, *E. saligna*, *E. grandis*) impregnated under laboratory conditions with 1% (w/w) CCA solution and as described in Figure 2 according to the AWWPA recommendation (26). The concentration values determined for As, Cr, and Cu in the analysis of all CCA-treated wood samples are presented in Table VI. This table also shows the comparison of the results with those obtained for decomposition with microwave heating. The relative standard deviations (RSD) were lower than 8% for both methods.

The paired Student's *t*-test (31) was applied to the results obtained using both methods to verify if there was a difference in the two methods at the 95% confidence level. For all the elements in the different samples, the *t*-value calculated was lower than the critical *t*-value, indicating no significant differences between the two methods. In addition (as shown in Figure 6), a significant linear relationship ($R = 0.999, 0.991, \text{ and } 0.998$) with the slope of 1.17, 1.06, and 1.17, for Cu, Cr and As, respectively,

TABLE V
Comparison Between the Results Obtained by
Acid Decomposition With Oven Heating and Results From IPT

	CuO		CrO ₃		As ₂ O ₅		Total Oxides		Relative Error
	(kg m ⁻³)		(kg m ⁻³)		(kg m ⁻³)		(kg m ⁻³)		(%)
	Oven	IPT	Oven	IPT	Oven	IPT	Oven	IPT	E*/IPT
C. citriodora B	0.31	0.30	1.63	1.70	1.60	1.00	3.54	3.00	18
E. grandis B	0.21	0.30	1.41	1.80	1.41	1.00	3.03	3.10	2.2
E. saligna B	0.19	0.30	1.76	1.50	0.52	0.90	2.47	2.70	8.5

IPT = Sao Paulo Institute of Research and Technology.

*E = Absolute error = |Oven - IPT|

TABLE VI
Analysis of CCA-treated Wood Samples Using Acid Decomposition
With Oven Heating and Microwave Heating by ICP-MS

Sample	Element	Oven (μg g ⁻¹)	Microwave (μg g ⁻¹)
Pole removed from service	Cu	522 (15) ^a	545 (8)
	Cr	820 (4)	1100 (2)
	As	484 (10)	479 (9)
New pole 1	Cu	2407 (75)	2045 (100)
	Cr	3348 (102)	2947 (149)
	As	3628 (83)	3004 (143)
New pole 2	Cu	3389 (34)	2883 (27)
	Cr	4707 (13)	4533 (49)
	As	4879 (39)	4229 (32)
Impregnated pole <i>C. citriodora</i>	Cu	368 (8)	293 (5)
	Cr	1044 (36)	869 (16)
	As	816 (18)	670 (6)
Impregnated pole <i>E. saligna</i>	Cu	186(15)	160 (3)
	Cr	788 (63)	707 (29)
	As	550 (40)	535 (16)
Impregnated pole <i>E. grandis</i>	Cu	490 (3)	343 (19)
	Cr	1869 (10)	1564 (74)
	As	1533 (4)	1188 (60)

^a Values in parentheses are standard deviations of three determinations (n=3).

exists between the methods. These comparisons indicate the viability of the oven decomposition method for estimating As, Cr, and Cu concentrations in pole samples and assessing the environmental impact by using CAA as a preservative.

Table VI also shows a difference between concentration ranges for the samples from new poles, those from industrially impregnated poles (under pressure in an autoclave), and samples impregnated in the laboratory. The reason is that the industry uses a 3.5% (w/w) CCA solution, whereas the solution used in the laboratory had a concentration of 1% (w/w).

The differences in concentration values for the new poles and those removed from service (Table VI) highlight the losses of the three elements into the environment with time of service. As previously discussed, these losses are likely related to the volatilization and leaching of CCA constituents over time.

CONCLUSION

Acid decomposition in closed flasks with oven heating using a mixture of nitric acid and hydrogen peroxide was efficient in decomposing wood samples. The ICP-MS technique, using internal standard calibration, was suitable for determining Cu, Cr, and As with low limits of detection, good accuracy, and a relative standard deviation lower than 8%. Factors such as decomposition time, sample quantity, and temperature exert an influence on analyte extraction.

A comparison between the method proposed in this study and that recommended by the AWWPA (microwave heating) using the paired *t*-test and also a regression analysis demonstrated the validity of the former in estimating As, Cr, and Cu concentrations in pole samples. The method developed is

Fig 6. Relationship between acid decomposition with oven heating and microwave heating.

simple and also allows an evaluation of the environmental consequences of using CCA as a preservative.

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