Hazardous Waste Analysis of Copper Azole, Alkaline Copper Quaternary, and Creosote Preserved Wood

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Hazardous Waste Analysis of Copper Azole, Alkaline Copper Quaternary, and Creosote Preserved Wood

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EXECUTIVE SUMMARY

Pressure-treated lumber is widely used in construction and landscaping. Toxicity concerns have led to arsenic-based wood preservatives being replaced by copper-based treatments, such as Copper Azole (CA) and Alkaline Copper Quaternary (ACQ).

Creosote, a coal-tar derivative, is another wood preservative, commonly used on utility poles and railroad ties. Creosote is a complex mixture of organic compounds, including toxic polycyclic aromatic hydrocarbons (PAHs) and cresols (methyl phenols). These materials may be classified as hazardous waste under the California Waste Control Law (Title 22 CCR, Section 66261.24, Characteristic of Toxicity) because of their total or leachable copper or phenolic content, or because of their aquatic toxicity. Currently, however, these preservative-treated wood wastes are regulated by the State of California under Alternative Management Standards (AMS), which, if certain conditions are met, do not require they be managed as hazardous waste.

This study evaluated the toxicity characteristics of CA, ACQ, and creosote-treated wood, using laboratory methods prescribed in Title 22 for waste classification. Representative samples of new CA- and ACQ-treated lumber and untreated controls were collected statewide. Used creosote-treated railroad ties were selected from a collection site in Nevada. Random sub-samples sawn from this wood were then composited and analyzed by the laboratory methods prescribed in Title 22.

Copper-treated wood was milled to a 2 mm particle size, and subject to the California Waste Extraction Test (WET); the WET extracts and the milled wood were analyzed for metals. The creosote-treated wood was cubed and milled. The cubes were extracted using the Toxicity Characteristic Leaching Procedure (TCLP); the TCLP extracts and the milled wood were analyzed for semivolatile organic compounds, including phenols and PAHs. The 96-hr aquatic screening bioassay was done on milled CA, ACQ, and creosote-treated wood. Untreated wood samples were analyzed as controls.

All of the copper-treated wood exceeded the STLC and TTLC toxicity criteria. (The CA-Douglas Fir (DF) mean total Cu was 2480 mg/Kg; however, the confidence interval of the mean was above the 2500 mg/Kg regulatory level.) No copper was detected in the control wood. Only the CA-Hemlock-fir lumber, which had the highest total and soluble copper levels, had an aquatic bioassay LC50 below the 500 mg/L regulatory level.

No TTLC-regulated compounds were detected in the creosote-treated oak and DF railroad ties; however, the pentachlorophenol quantitation limit was slightly above the 17 mg/Kg regulatory level. Other phenolic compounds and PAHs were found. The only TC-regulated compounds detected in TCLP extracts were cresols, but at concentrations well below the 200 mg/L regulatory level. The three creosote-treated DF samples had an LC50 below 500 mg/L; the oak-creosote sample and the untreated controls did not.

Based on this study, the CA-DF, ACQ-DF, and CA-Hem.-fir lumber, and creosote-DF RR ties would be classified as California hazardous waste; the creosote-oak would not.
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ABBREVIATIONS AND ACRONYMS

ACQ  Alkaline (Amine or Ammoniacal) Copper Quaternary
AMS  DTSC Alternative Management Standards for hazardous waste
ARF  Analysis Request Form, used to initiate sample analysis at ECL
AWPA American Wood Protection Association, an industry group that sets standards for treating wood with preservatives
CA   Copper Azole
CA-B Copper Azole, Type B (the type used in this study)
CCA  Chromated Copper Arsenate
Cu   Copper
CuO  Copper oxide
DF   Douglas fir, a softwood commonly used in construction in California
DTSC California Department of Toxic Substances Control
ECL  DTSC's Environmental Chemistry Laboratories in Berkeley and Los Angeles

g   gram
GC-MSD Gas chromatography with a mass-spectrometer detector, a laboratory instrumental technique used for trace organics analysis
GF/F Glass fiber filter used to filter TCLP extracts prior to analysis
HW   Hardwood (oak in this study); also, hazardous waste, as defined in U.S. and California statute and regulations

kg   kilogram
ICP-AES Inductively coupled plasma-atomic emission spectrometry, a laboratory instrumental technique used for trace metals analysis
LC\textsubscript{50} Concentration lethal to 50 percent of the bioassay test organisms
<table>
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<tr>
<th>Abbreviation</th>
<th>Description</th>
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<td>LCS</td>
<td>Laboratory control sample, a QC sample with a known concentration of analyte(s) of interest</td>
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<td>Matrix spike and Matrix spike duplicate</td>
<td>QC samples made by adding a known quantity of analyte(s) of interest to sample replicates</td>
</tr>
<tr>
<td>Method blank</td>
<td>A QC sample containing no added analyte(s) of interest</td>
</tr>
<tr>
<td>mg</td>
<td>milligram</td>
</tr>
<tr>
<td>PAH</td>
<td>Polyaromatic hydrocarbon, also referred to as polynuclear aromatic</td>
</tr>
<tr>
<td>PCF</td>
<td>Pounds (of preservative) per cubic foot of treated lumber, an AWPA standard for retention based on species and end use of lumber</td>
</tr>
<tr>
<td>PFTE</td>
<td>Poly(tetrafluorethylene), a fluorine-containing polymer</td>
</tr>
<tr>
<td>QA/QC</td>
<td>Quality assurance/quality control, laboratory system and protocols used to assess accuracy, precision, and other data quality criteria</td>
</tr>
<tr>
<td>STLC</td>
<td>Soluble Threshold Limit Concentration (Title 22, Div. 4.5)</td>
</tr>
<tr>
<td>SVOC</td>
<td>Semi-volatile organic compounds, a class of organic compounds that can be volatilized when heated, as in a GC/MSD instrument</td>
</tr>
<tr>
<td>TC</td>
<td>Toxicity Characteristic, a hazardous waste regulatory level</td>
</tr>
<tr>
<td>TCLP</td>
<td>Toxicity Characteristic Leaching Procedure, SW-846 Method 1311</td>
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<td>Title 22 CCR</td>
<td>California Code of Regulations, Title 22</td>
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<td>TTLC</td>
<td>Total Threshold Limit Concentration (Title 22, Div. 4.5)</td>
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<td>TWW</td>
<td>Treated Wood Waste</td>
</tr>
<tr>
<td>U.S. EPA</td>
<td>United States Environmental Protection Agency</td>
</tr>
<tr>
<td>WF</td>
<td>White fir, a member of the Hemlock-fir (HF) species group</td>
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</table>
ACKNOWLEDGEMENTS

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INTRODUCTION

Arsenic-based wood preservatives, such as Chromated Copper Arsenate (CCA), have been phased out because of concerns over the toxicity of arsenic (1), although significant amounts are still in service. Alternative waterborne pressure-treatment preservatives include Copper Azole Type B (CA-B) and Alkaline Copper Quaternary (ACQ) (2). The U.S. EPA has not established a federal regulatory level for copper, the primary biocide in these preservatives. Copper, however, is regulated by the State of California as a Persistent and Bioaccumulative Toxic Substance (Title 22 California Code of Regulations, Division 4.5, Chapter 11, Section 66261.24). Wastes exceeding the Title 22 Soluble Threshold Limit Concentration (STLC) or Total Threshold Limit Concentration (TTLC) regulatory levels for soluble (extractable) or total or copper must be managed in accordance with the California Hazardous Waste Control Act (Health and Safety Code, Division 20, Chapter 6.5).

Railroad ties are typically preserved with creosote, a complex mixture of organic compounds (3), including polyaromatic hydrocarbons (PAHs) and cresols (methyl phenols), derived from coal-tar distillate. Cresols, trichlorophenol, and pentachlorophenol have regulatory levels based on the U.S. EPA Toxicity Characteristic Leaching Procedure (TCLP) (22CCR, Section 66261.24(a) (1)).

The Department of Toxic Substances Control (DTSC) has adopted Alternative Management Standards (AMS) for certain treated wood wastes (22CCR, Division 4.5, Chapter 34) that are deemed protective of human health and the environment, but exempt these materials from hazardous waste requirements. So that DTSC can evaluate and develop its standards for the management of treated wood wastes, it asked its Environmental Chemistry Laboratory (ECL) to characterize CA-B, ACQ, and creosote treated wood using laboratory methods for hazardous waste testing specified by the California Code of Regulations, Title 22, Sec. 66261.24, “Characteristic of Toxicity.”

To evaluate CA-B and ACQ wood wastes, new preserved and untreated lumber was analyzed for total copper and other metals using U.S. EPA methods for acid digestion and analysis. Soluble metals were determined using the Title 22 California Waste Extraction Test (WET), a 48-hour citrate buffer extraction. Results from these tests were compared to the Title 22 TTLC and STLC, respectively, for copper.

Used creosote-treated railroad ties were tested for total semivolatile organic compounds (SVOC) using U.S. EPA solvent extraction and analysis methods. Soluble SVOCs were determined using the federal Toxicity Characteristic Leaching Procedure (TCLP), a 16-hour extraction with acetate buffer. The total and leachable SVOC results were compared to the regulatory levels for target analyte compounds, specifically the cresols and chlorophenols.

The Title 22 mandated 96-hour acute aquatic bioassay was also done, at a contract laboratory, on the CA, ACQ, and creosote preserved wood and untreated controls.
Wood samples were collected and processed under a contract by the University of California Cooperative Extension, and according to a sampling plan (Appendix I) developed by U. C. and DTSC. Wood samples were cut, randomly sampled, and put in containers at the U.C. Richmond Field Station (RFS) in Richmond, California. The samples were then taken to ECL in Berkeley for further processing and analysis.

Part I: Copper Azole (CA) and Alkaline Copper Quaternary (ACQ) Treated Lumber

BACKGROUND

Douglas fir and Hemlock fir lumber is stacked, then pressure-treated in a cylinder (retort). The lumber is first incised to aid penetration into the interior of the board by the preservative. The industry specification (4) for DF and HF penetration is 10 mm (0.4 in) and 90 percent of the sapwood, but no more than one-half the width or thickness of the board. The specified ground-contact retention (or preservative loading) for CA-B is 0.21 pounds per cubic foot (pcf), including 0.16 pcf as Cu and 0.0066 pcf as the azole co-biocide. For ACQ, the specified retention is 0.40 pcf, including 0.21 pcf as CuO (0.17 pcf as Cu) and 0.11 pcf as the quaternary ammonium co-biocide. (Preserved lumber for above-ground use has lower specified retention; only ground-contact lumber was considered in this study.) The retention specifications are based on the preservative concentration in the “assay zone”; for the dimensional lumber used in this study, the assay zone is 15 mm (0.6 in) from the treated surface toward the center of the cross section of the board.

FIELD INFORMATION

As described in the Sampling Report, CA Type B CA-B and ACQ treated 2”x8”x8’ boards were purchased at home centers and lumberyards throughout California. Twenty boards were obtained for each of three species-treatment combinations: Douglas fir (DF)-CA, DF-ACQ, and Hem-fir (HF)-CA. In California, White fir (WF) is the typical species in the Hemlock-Fir (HF) species group; these terms are used here interchangeably. No HF-ACQ was collected. For quality assurance controls, untreated DF 2x8s and WF 2x4 mill end-cuts were obtained (untreated WF boards are not commonly available).

For each of the four composite replicates, 0.25 in. slices were taken from three locations in the interior of 20 boards (two and four feet from the ends). To facilitate processing in the laboratory, the slices were quartered. For each replicate, one quartered specimen, randomly allocated, from each of the 20 boards was aggregated to make a composite sample. Four composite samples were prepared for each species-treatment combination. These were put into sample jars provided by ECL, and labeled accordingly: CADF1…CADF4, CAHF1…CAHF4, and ACQDF1…ACQDF4. The untreated control wood was similarly cut into small pieces. Sufficient untreated Douglas
fir was provided for mill (equipment) blanks, and for replicate control composite samples prepared in the laboratory.

The samples were transported to ECL by ECL staff. After additional preparation (grinding and sub-sampling), the wood samples were logged-in and assigned ECL sample numbers. The samples were then distributed to the ECL Inorganic section and the contract laboratory for analysis. Sample management was documented with the ECL Authorization Request Form (ARF) and the Sample Analysis Request (SAR) form. The SAR also serves as the ECL chain of custody document.

ANALYTICAL PROCEDURES

Particle Size Reduction
The lumber samples were reduced in size to the dimensions specified in the hazardous waste testing regulations and applicable laboratory methods. Title 22 CCR, Chapter 11, App. II, Section (c)(1) states that for total and extractable regulated inorganic elements (i.e. for which TTLCs and STLCs exist) a “millable solid…shall be milled to pass through a No. 10 (two millimeter) standard sieve before it is analyzed.”

Regarding the aquatic bioassay, Title 22 CCR, Ch. 11, Sec. 66261.24(a)(6) says to use “…test sample prepared or meeting conditions for testing as prescribed in subdivisions (c) and (d) of Appendix II.” Thus, the 2 mm particle size was deemed appropriate for the WET (STLC), total metals (TTLC), and the 96-hr LC50 aquatic bioassay procedures.

The lumber samples were ground with a laboratory mill (Thomas-Wiley Model 4, Thomas Scientific) (Figure 1), fitted with a 2 mm sieve at the outlet. Before use, and after each composite replicate was processed, the mill was cleaned using brushes, laboratory spatulas, and compressed air. Once clean of particulate matter, the mill was wiped with acetone using cotton swaps and laboratory wipes. To check for analyte carry-over between replicates, approximately 50 g DF (from the same sources as the control DF) was milled, then discarded. For the CA-DF replicates, another 50 g DF was milled and retained as a mill blank (MB). One MB was prepared before the first sample, and after each treatment replicate. The composite samples were milled in this order: DF control, CA-DF, ACQ-DF, CA-HF, and WF control. Four DF, but only one WF, control replicates were prepared.

The quartered wood sections were fed into the mill in small handfuls (Figure 2). The milled wood (about 700-800 g for each composite replicate) was mixed in an aluminum pan using a plastic scoop until the sample appeared homogenous, as indicated by a uniform distribution of the light colored, untreated core wood and the darker colored treated exterior wood (Figures 3-7). About one-half of each of the composited samples was transferred to large and small pre-cleaned sample jars (Figure 8) that were distributed for analysis. The remaining sample was retained in sealed polyethylene bags.
Figure 1. Laboratory mill, with 2mm sieve

Figure 2. Milling wood pieces

Figures 3-5. Treated 2x8 quartered-sections as received at laboratory and after milling

Figure 6-7. Untreated Douglas fir and Hem. fir controls

Figure 8. Composited milled samples, ready for analysis
Sample Analysis
Total metals and WET analyses were done by the ECL-Berkeley Inorganic Section. For metals analysis, the wood samples were acid-digested using U.S. EPA Method 3050B and analyzed using Method 6010B, Inductively Coupled Plasma-Atomic Absorption Spectrometry (ICP-AES).

WET analysis was done using ECL SOP 910, California Waste Extraction Test, with 10 g sample and 100 mL pH 5 citrate buffer. Extraction was for 48 hr with constant agitation, followed by filtration through a 0.45 micron filter. The WET filtrate was analyzed by Method 6010B for metals; the filtrate was not digested, but was diluted (1:10) with water because of the high salt content of the extractant. The treatment composites were extracted and analyzed separately, as submitted, but the four Douglas fir control composites were further composited by the Inorganic Section to yield one sample for the WET. Only one Hemlock-fir control sample was milled, extracted, and analyzed.

Aquatic bioassays (5) were done at Associated Laboratories (Orange, CA) using fathead minnows (Pimephales promelas) as the test organism. The bioassays were of the screening type, at three concentrations: 250 mg/L, 750 mg/L, and 500 mg/L (the California 96-hr LC50 toxicity characteristic level). A subsample of the milled wood samples (e.g. 5.0 g to give a final concentration of 500 mg/L in a 10 L aquarium) was mixed with 300 mL of the same water used in the aquaria and shaken for six hours (6). This suspension was added to the aquarium water, which was then made up to 10 L. The 500 mg/L concentration was run in duplicate. An undosed control aquarium was also run. During the 48-hr test period, dissolved oxygen, temperature, pH, and fish survival were monitored. A slow air bubble stream was introduced to maintain dissolved oxygen levels.

Quality Control and Quality Assurance
Sampling Quality Control
Quality control during sample collection and processing are described in the Sampling Plan (Appendix I) and the Study Report (Appendix II). Treated lumber and untreated DF boards were purchased from different outlets throughout the state, to ensure a representative sample. To prevent cross-contamination, a new cross-cut saw blade was used for each species-treatment combination (i.e. after 20 boards were processed), and the blades were wiped with alcohol between boards. The locations on the board of the 0.25” sections that made up each of the four composite replicates for each species-treatment were randomly allocated. A new band saw blade was used for each composite replicate (four per species-treatment) to quarter the pieces for laboratory preparation. The samples were then put into clean quart jars provided by ECL. As described above, the laboratory mill was thoroughly cleaned before each composite replicate was processed, and equipment (mill) blanks were milled between replicates to check for cross-contamination. All 13 mill blanks were analyzed for total metals; one composite made of the 13 was subjected to the WET. The milled wood was thoroughly mixed to ensure a homogenous and representative sample, and subsampled into labeled jars for analysis.
Analytical Quality Control
Standard U.S. EPA SW-846 and ECL quality control procedures were followed for the metals analysis and the WET. Method blanks, a solid laboratory QC sample (LCS), matrix spikes (MS) and matrix spike duplicates (MSD) were analyzed. Triplicate extractions and analyses were done on two of the treated wood composites for both the WET and total metals. Matrix spikes for the WET were done after extraction and dilution, and before instrumental analysis. Daily multi-point ICP-AES calibration standards and a reagent blank were run to establish response linearity, and calibration verification standards were analyzed after every ten samples.

RESULTS AND DISCUSSION

Laboratory results for the total copper and copper by WET are shown in Figures 9 and 10, and in Table 1. All three species-preservative combinations exceeded the California Toxicity Characteristic regulatory levels for total and soluble copper. The extraction efficiency of copper by the WET (WET/total Cu) was over 87 percent. Copper was not detected in the untreated control samples.

The 96-hr acute aquatic bioassay results are shown in Table 1. Only the copper azole-treated Hemlock-fir composites, which had the highest total and soluble copper, had a 96-hr acute aquatic bioassay LC50 concentration less than the 500 mg/L toxicity characteristic level. The LC50 results were >750 mg/L for the CA and ACQ treated Douglas fir, and the control Douglas fir and Hemlock-fir; all the fish survived those trials.

![Figure 9. Preservative Treated Wood Total Copper](image1)

![Figure 10. Preservative Treated Wood CA Waste Extraction Test (WET)](image2)

**Preservative Treated Wood Total Copper**

- **Douglas fir - Copper Azole**
- **Hem fir - Copper Azole**
- **Doug. Fir - Alkaline Copper Quaternary**

**Total Threshold Limit Concentration (TTLC)** = 2500 mg/Kg wet wt. (CA Title 22 Toxicity Characteristic, Sec. 66261.24)

**Preservative Treated Wood CA Waste Extraction Test (WET)**

- **Doug Fir-Copper Azole**
- **Hem Fir-Copper Azole**
- **Doug Fir - Alk. Copper Quaternary**

**Soluble Threshold Limit Concentration (STLC)** = 25 mg/L
Table 1. Hazardous Waste Analysis of CA and ACQ Treated Lumber

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<tr>
<th>Sample</th>
<th>ECL No.</th>
<th>Cu mg/Kg</th>
<th>MDL</th>
<th>Cu mg/L</th>
<th>MDL</th>
<th>percent</th>
<th>96-hr Aquatic Bioassay LC50</th>
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ND = not detected, CA = copper azole, DF = Douglas fir, COMP = composite, MB = mill blank, ACQ = alk.copper quant., HF = Hemlock-fir, CTRL = control

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<tr>
<th>Regulatory Level:</th>
<th>Total Copper TTLC 2500 mg/Kg</th>
<th>Cu-WET STLC 25 mg/L (Tot. Cu) (10x WET)</th>
<th>96-hr Aquatic Bioassay LC50 500 mg/L</th>
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<td>Cu-WET (10x WET)</td>
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<td>STLC</td>
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Although the industry retention specifications (4) for ground-contact use for CA-B and ACQ are nearly the same (0.16 as Cu for CA; 0.21 as CuO = 0.17 as Cu, for ACQ), the experimental concentrations of the three species-treatments were very different: The CA Hem.-fir Cu concentration was 95 percent greater than CA-DF. The ACQ mean Cu value was between the two CA means. The methodology used for this study, however, was different than the AWPA procedure for measuring preservative retention in the 15 mm (0.6 in) deep assay zone using core samples. The goals of this study did not include determining if the sampled lumber met specifications for preservative retention, although visually it appeared that some of the cross-sections did not have the required 10 mm (0.4 in) penetration by the preservative.

Low concentrations (< 6 mg/L WET; < 50 mg/Kg total) of barium and zinc were found in all the wood samples. Presumably, these are naturally occurring trace elements.

All the QC results for accuracy (percent recovery) and precision (relative percent difference and relative standard deviation) were within the established control limits for the determinative methods (Appendix 4). The relative standard deviations for the treatment–species composite replicates were less than 10 percent for total and TCLP copper.

Part II: Creosote Treated Railroad Ties

BACKGROUND

According to the American Wood Protection Association (AWPA) 2005 Standards (4), creosote-treated railroad ties have a specified retention of 8.0 pcf or refusal for Douglas-fir and 7.0 pcf or refusal for oak. A Swiss study (7) estimated that during the 20-30 yr service life of railroad ties, 5 Kg creosote, 0.5 Kg PAHs, and 10 g phenolic compounds are emitted. When taken out of service, one disposal option is as a fuel source in co-generation plants. The treated ties used in this study were from this waste stream.

FIELD INFORMATION

Railroad tie samples were collected at a collection yard in Flannigan, NV, 60 mi north of Reno (Appendix III). Bundles of ties were randomly selected from open railcars used to transport the out-of-service ties. Sixty-two softwood (Douglas fir) and 18 hardwood (oak) ties were sampled; this reflected the distribution of ties in the yard. Using a chainsaw, two sections were sawn from each tie; each cross-cut section was approximately 3 in. thick (Figure 11). One section from each tie was retained at RFS; the other was further sawn at RFS, and became part of three DF and one oak composite sample that were submitted to ECL for analysis. The prepared and retained samples were randomly allocated. Eighteen oak tie sections were used for the oak composite, 19 DF tie
sections were used for DF replicate 1; 20 DF tie sections each were used for DF composite replicates 2 and 3.

Composite samples were prepared in two ways, for two types of laboratory analyses. First, the tie sections were sawn into representative sub-sections. Then, with a bandsaw, the sub-sections were reduced in size for milling at ECL (as with the CA and AQC lumber) or were cut into 0.9 cm cubes for TCLP. In order to expose a fresh wood surface, a thin section was removed from each section prior to cutting them for milling or TCLP. Composites were prepared of both types of sub-samples; approximately one-half of each section went into the milling composite for that replicate; the other half was used for the TCLP cubes for that same numbered replicate.

Oak control wood was cut from twenty 1” board cut-offs from a Berkeley lumber yard. The DF-creosote controls came from the same source as the DF-CA and DF-ACQ controls.

The processed wood samples were put into jars provided by ECL, and labeled: DF Creosote 1, DF Creosote 2, DF Creosote 3, and HW Creosote. The samples were transported to ECL by laboratory staff, where they were prepared and distributed for analysis.

ANALYTICAL PROCEDURES

Particle Size Reduction
Composite samples for semi-volatile organic analysis and aquatic bioassay were milled as described in Sec. 4.1 for CA- and ACQ-treated lumber. For TTLC compliance for organic compounds, Title 22, Chapter 11, Appendix II, states that particle size should be < 1 mm. The sample extraction Method 3540 for semi-volatile organic compounds in solid samples (by Soxhlet extraction) also calls for 1 mm particle size.

However, the 2 mm sieve was used for grinding the creosote samples (Figures 12, 13). When milling the control oak, some charring was noticed. Grinding the samples further, to < 1 mm, may have degraded the sample and likely would not have yielded better extraction efficiency. The extraction Method 3540 says that for fibrous samples, particle size reduction should be sufficient to ensure contact with the solvent. Title 22, Chapter 11, Appendix III states that SW-846 should be consulted on appropriate methods for each “specific sample analysis situation.” It was therefore determined that the 2 mm sieve size used for the CA and ACQ lumber was also appropriate for the creosote-treated wood sample preparation.

The mill was again cleaned between replicates with scrapers, brushes, compressed air, and acetone. Approximately 50 g DF control wood was then milled and discarded, but no mill blanks were analyzed.
Samples that were to be subject to the TCLP (U.S. EPA SW-846 Method 1311) were sawn into 0.9 cm cubes at the RFS, and did not require further size reduction. Scheduling was coordinated so that samples could be extracted in the laboratory within 14 days of being cubed, in keeping with the method hold time.

**Toxicity Characteristic Leaching Procedure (TCLP)**

Approximately 100 g cubed DF and oak controls, and creosote-treated DF and oak were extracted with 2 L (20:1 ratio) buffer solution. Based upon the preliminary evaluation of the pH of a mixture of the milled sample and water, TCLP extraction fluid #1 was used. This is an acetate buffer at pH 4.93 +/- 0.05. The sample and extractant were put into PFTE bottles, which were put into the TCLP rotary agitation device (Associated Design and Manufacturing, Alexandria, VA) (Fig. 13). After 18 hrs, the samples were filtered using a pressure filtration device (Millipore, Inc., Bedford, MA) and GF/F glass fiber filters (Figs. 14, 15). The filtrates (light brown for the controls, and dark amber for the creosote samples) were transferred to labeled bottles, and then sent, packed in ice, by overnight courier to ECL - Los Angeles for analysis.
Sample Extraction and Analysis
The aqueous TCLP extracts were extracted within the seven days specified by Method 1311, using Method 3510, separatory funnel liquid-liquid extraction. The milled wood samples were extracted with Method 3540, Soxhlet extraction. The wood extracts were subjected to Method 3640, a gel permeation column cleanup to remove interferences.

The TCLP and wood extracts were analyzed by gas chromatography with a mass selective detector (GC-MSD) using Method 8270C. Although the initial study design only considered organic compounds for which a Toxicity Characteristic (TC) or a Total Threshold Limit Concentration (TTLC) regulatory level have been set, the decision was made to report and quantify, if possible, all compounds on the Method 8270C target list.

The toxicity of creosote-contaminated water has been attributed to several classes of compounds, such as phenols, PAHs, and N-heterocyclic aromatics (8). PAHs and N-heterocyclics have also been found in laboratory leachates of creosote-treated wood, using deionized water, pH 4.7 buffer, and humic acid solutions (9). Therefore, it was considered worthwhile to identify and quantify compounds in creosote-treated wood and their TCLP leachates that were of interest as environmental contaminants, but for which regulatory levels had not been established under the Characteristics of Toxicity (Title 22, Section 66261.24).

Quality Assurance and Quality Control
Sampling Quality Control
Quality control during sample collection and processing, in the field and at RFS, are described in the Sampling Plan and the Sampling Report. Eighty ties were selected from various bundles from different railcars at the Flanigan, NV yard. Two cross-sections of each tie were removed using a chain saw, and one of those, randomly allocated, was further sawn at RFS. The 80 ties resulted in four composite samples, one oak and three Douglas fir, each representing approximately 20 ties.

Treated and control (untreated) wood was processed with the same equipment, at both RFS and ECL. Representative specimens of each tie cross-section were sawn and cubed. As with the copper-preserved lumber, saw blades were changed or cleaned with alcohol to minimize carryover between samples. In the laboratory, the mill used for grinding the wood was cleaned between replicates.

Representative composites were made by thoroughly blending the milled wood by hand. Representative sub-samples of the cubed wood containing dark, treated and light, untreated wood were used for the TCLP extractions. Duplicates, reagent (extraction solution) blanks, and cubed oak and DF controls were carried through the TCLP.

Analytical Quality Control
For the solvent extraction and GC-MS analysis, standard ECL and SW-846 QC practices were followed. Method blanks, method standards, surrogates, matrix spikes and matrix spike duplicates were analyzed to assess bias (accuracy) and precision. For quantitation, multi-point calibrations were done using commercially available reference
mixtures. Response factors were verified by continuing check standards. Analyte identification is by comparison of the unknown and reference compound spectra, using characteristic ions.

For the aquatic bioassay, the contract laboratory used three sample dilutions; one dilution was run in duplicate. A control was run for each batch using unspiked waste. In accordance with the California Dept. of Fish and Game procedure, the milled wood sample was shaken for 6 hr with water (50 g: 50 mL) to disperse the sample before an aliquot was taken and added to the aquarium water.

RESULTS AND DISCUSSION

Toxicity Characteristic Leaching Procedure
The complete TCLP-creosote GC/MS laboratory reports are in Appendix 2. The results for TC analytes in the TCLP extracts are in Table 2; all analytes are in Table 3. The oak-creosote composite and one of the Douglas fir composite replicates had 0.42 mg/L and 1.4 mg/L methyl phenols (cresols), respectively, well below the toxicity characteristic regulatory level of 200 mg/L. No other TC compounds were detected above the 0.04 mg/L quantitation limit (0.50 mg/L for pentachlorophenol and three nitro-phenols). Trace amounts of 2- and 3-ring polyaromatic hydrocarbons (PAHs) were found in all treated wood samples. Phenols and two heterocyclic compounds, carbazole and dibenzofuran, were reported in the oak and one DF composite sample.
# Table 2  TCLP Creosote-Treated Wood Semivolatile Organics

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<th>AQ-</th>
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<th>AQ-</th>
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<td>HW (oak)</td>
<td>TCLP</td>
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ND = not detected  
Sample: 1,2,3 = composite replicate ; A,B = TCLP replicate  
NR = not reported
### Table 3: Toxicity Characteristic Leaching Procedure

**Creosote Treated Wood Semivolatile Organics Method 8270C GC/MS Results**

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<th>Sample:</th>
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<th>TCLP blank control creosote-2A blank control</th>
<th>TCEP blank control creosote-2B blank control</th>
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</table>

**Estimate** **Quant. Limit = 0.5 mg/L; all other analytes = 0.04 mg/L**

Sample: 1,2,3 = composite sample replicates; A,B = TCLP duplicates

*yellow = PAH  blue = phenolic  bold = TC rule compound (reg. level mg/L)  orange = heterocyclic aromatic*
Aquatic Bioassay of Creosote Treated Wood

As indicated in Table 4, the oak control composite sample was non-hazardous (LC 50 > 500 mg/L), as were the four Douglas fir control composites tested with the CA and ACQ treatments. All three Douglas fir-creosote composite samples had LC50 values below the 500 mg/L regulatory level. The hardwood (oak) creosote-treated composite sample had an LC50 above the regulatory level. Fish survival, however, was 60 and 70 percent for the duplicates, indicating some toxic effect at concentrations below the regulatory threshold.

Table 4 96-hour Acute Aquatic Bioassay Creosote-Treated Railroad Ties

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<th>AR-</th>
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<td>00117</td>
<td>00118</td>
<td>00119</td>
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<tr>
<td>Sample:</td>
<td>oak control</td>
<td>HW (oak) creosote</td>
<td>Doug. fir controls*</td>
<td>DF creosote Comp-1</td>
<td>DF creosote Comp-2</td>
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<tr>
<td>LC50 (mg/L)</td>
<td>&gt; 750</td>
<td>&gt; 500</td>
<td>&gt; 750</td>
<td>&lt; 250</td>
<td>&lt; 250</td>
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<tr>
<td>percent survival at 500 mg/L, 96-hrs (duplicates)</td>
<td>100,100</td>
<td>60,70</td>
<td>100,100</td>
<td>20,10</td>
<td>10,20</td>
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</table>

* same for all DF controls

Total Semivolatile Organics in Creosote Railroad Ties

The results for creosote treated wood and controls semivolatile organic compounds by SW-846 Method 8270C are summarized in Table 6. The complete laboratory reports are in Appendix IV. Pentachlorophenol (TTLC=17/mg/Kg) was not detected. Other phenols (primarily cresols), PAHs, carbazole (dibenzopyrrole), and dibenzofuran were reported in all treatment samples, but not in the controls.

The PAHs included 4- and 5-ring compounds not in the TCLP extracts: anthracene, fluoranthene, pyrene, crysene and benzo (b) fluoranthene. The ratio of total to TCLP concentrations (Table 5) demonstrates the low aqueous solubility of the tricyclic PAHs compounds (aceanapthene, florene and phenanthrene) and the relatively higher solubility of naphthalene.

Table 5. Total vs. soluble (TCLP) PAHs in Creosote-Treated RR Ties

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<tr>
<th>PAH:</th>
<th>naphthalene</th>
<th>acenaphthene</th>
<th>fluorene</th>
<th>phenanthrene</th>
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<tr>
<td>Total (mg/Kg mean, n=4):</td>
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<tr>
<td>TCLP (mg/L mean, n=6):</td>
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<tr>
<td>Total/TCLP</td>
<td>750</td>
<td>5300</td>
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Table 6. Creosote Treated Wood SVOCs by GC/MS Method 8270C

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ND = non-detect, NR = not reported, **bold** = TCLP-regulated compound, blue = phenolic, orange = heterocyclic aromatic, yellow = PAH, TTLC = 1mg/Kg, TCLP = 17mg/Kg
CONCLUSIONS

The copper azole (CA-B) and alkaline copper quaternary (ACQ) treated new 2x8s exceeded the California Title 22 hazardous waste Toxicity Characteristic STLC and TTLC levels for soluble (WET) and total copper. The Douglas fir CA-B and ACQ lumber had a 96-hour acute aquatic bioassay LC$_{50}$ > 500 mg/L; CA-B treated Hemlock fir had a LC$_{50}$ < 500 mg/L, which is below the Toxicity Characteristic level. The CA-B Hemlock fir had the highest total and soluble copper levels; this may account for the higher aquatic toxicity in these samples.

Low concentrations of PAHs, heterocyclic aromatics, and phenols were measured in used creosote-treated railroad ties and their TCLP extracts. The TCLP concentrations for cresols were well below the Title 22 Toxicity Characteristic regulatory level. Pentachlorophenol was not detected in the railroad ties or the TCLP extracts. The oak railroad ties had a 96-hour acute aquatic bioassay LC$_{50}$ > 500 mg/L; however, fish survival was 60 and 70 percent at the 500 mg/L concentration, indicating a toxic effect below the regulatory threshold. Douglas fir ties had a LC$_{50}$ < 500 mg/L, below the Toxicity Characteristic level.

The untreated oak, Douglas fir, and hemlock fir control samples did not exhibit aquatic toxicity, or have detectable concentrations of regulated elements or compounds in the wood or their WET and TCLP extracts.

The methods and findings described in this report were incorporated into the department’s Draft Report: Sampling and Analysis Study on Treated Wood, and presented at a DTSC public workshop on Sept. 11, 2008. The draft report, workshop presentations, laboratory reports, and other material related to this study and the regulation of treated wood wastes are on the DTSC website under the Emerging Issues tab at:

http://www.dtsc.ca.gov/HazardousWaste/Treated_Wood_Waste.cfm

REFERENCES


(3) Kohler, M.; Känniger, T.; Schmid, P.; Gujer, E., Crockett, Rowena, Wolfensberger, M. Inventory and emission factors of creosote, polycyclic


APPENDICES

Appendix 1: Sampling plan (Stephen Quarles)

Appendix 2: Laboratory Reports with Table of Contents