

ASPECTS OF CHEMICAL CONTROL OF  
THE OLD HOUSE BORER,  
Hylotrupes bajulus (L.)  
(COLEOPTERA: CERAMBYCIDAE)

by

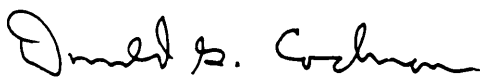
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APPROVED:



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(ABSTRACT)

The formulation of Dursban®, manufactured by Dow Chemical Corp., and its diluting agent were found to influence the penetration abilities of the final spray when it is applied to pine sapwood. The diluent of choice for providing the best penetration of the active ingredient, chlorpyrifos, is an oil-based carrier for the formulated product Dursban® WT. Dursban® TC, another product containing chlorpyrifos, will penetrate the wood surface better when diluted with water, not with an oil-based carrier such as kerosene. Residual amounts of Dursban® TC applied to pine sapwood will remain relatively constant six months after the initial treatment at depths of 400 - 600 microns below the surface.

The wood moisture content (WMC) of treated pine sapwood did not significantly influence the penetration abilities of Dursban® TC at 7.5% - 8.0% and 14.5% - 16.0% WMC. A 1.0% water-diluted formulation of Dursban® TC applied to pine

sapwood is predicted to be capable of penetrating the treated wood to a depth of 1320 microns.

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I dedicate this thesis to my first entomology professor, George W. Byers from the University of Kansas. He introduced me to the field of science that observes all of the unique characteristics of the diverse collection of insects. From our first acquaintance at Mt. Lake Biological Station in 1980 to the present, I have valued and appreciated the caring approach he has towards each and every student. I believe that this is why I have become an "entomologist".

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## 1.0 INTRODUCTION

The cerambycid, Hylotrupes bajulus (L.), the old house borer, is a pest of seasoned softwoods (pine, spruce, fir). Although native to Africa, H. bajulus has established a world-wide distribution (H. Becker 1968, 1979). St. George et al. (1957) first reported Hylotrupes bajulus in North America. Durr (1954) stated that wood infested by this species may be severely damaged due to the longevity of the larval stage and its feeding. Larvae will feed throughout the sapwood portions of infested wood, excavating galleries and creating structural as well as cosmetic damage.

Fertile adult females deposit eggs in cracks and crevices of coniferous woods. In the United States, the larval growth and development period ranges from 2 - 7 years (Cannon and Robinson 1983). The adult life stage is limited to 1 - 3 weeks (Durr 1956).

Lumber today is comprised of little heartwood and a considerable amount of sapwood. This sapwood provides a food supply for old house borer larvae. When young coniferous trees are cut into boards and kiln dried, the moisture content of the processed wood is usually between 12 - 18%, within the range suitable for old house borer infestations. Once an active infestation begins, destruction of timber may occur (Robinson 1986).

Previous studies have focused on the morphological, physiological, and ecological aspects of the old house borer (Durr 1954, 1956, Cymorek 1968, Serment 1976, Cannon and Robinson 1985, Mares and Robinson 1986). Chemical control of this species, both preventative and remedial, has received limited attention. Metzner et al. (1977) stated that the range of toxic effectiveness of chlorpyrifos, a chemical currently used to control old house borer infestations, is not known. By understanding the factors influencing the effectiveness of a chemical treatment, destructive infestations of Hylotrupes bajulus may be controlled.

The objectives of this research project were to:

1. Compare the wood penetration abilities of water-diluted formulations of chlorpyrifos, Dursban® Wood Treatment (WT) and Dursban® Termiticide Concentrate (TC) to the penetration abilities of kerosene-diluted formulations of Dursban® WT and TC;
2. Determine the six-month residual longevity of Dursban® TC applied to pine sapwood;
3. Determine the wood penetration abilities of Dursban® TC into wood at various moisture contents;
4. Predict the final endpoint of Dursban® TC penetration into pine sapwood.

## 2.0 LITERATURE REVIEW

### 2.1 GEOGRAPHIC DISTRIBUTION

The old house borer, Hylotrupes bajulus was first described by Linnaeus in 1758 as Cerambyx bajulus. Serville (1834) placed this beetle in the genus, Hylotrupes of the tribe Callidiini, subfamily Cerambycinae. Although native to Africa, H. bajulus has a world-wide distribution (H. Becker 1968, 1979). Infestations have been reported in Central Europe, South Africa, and China, as well as in much of southern England, including North Surrey and parts of Hampshire and Berkshire (Lea and Bravery 1978). The old house borer was first reported in Australia inhabiting imported timber (Hadlington and Campbell 1956). Howick and Carr (1971) reported that H. bajulus was not an established pest in Australia, but prevailing climatic conditions did favor establishment. In southern Norway, the old house borer is a known pest of wooden structures (Knudsen et al. 1969).

Hylotrupes bajulus was first reported in North America in 1841 (St. George et al. 1957). In the U.S., distribution of this species includes most states east of the Mississippi River, and states bordering the Gulf of Mexico (St. George et al. 1957, McIntyre and St. George 1961). The old house borer was discovered in Massachusetts in 1939 (Becker 1954), but New York state has collection specimens dating from 1900

(Simeone and MacAndrews 1955). Moore (1978) reported that North American populations of H. bajulus have not been established outside of man-made structures. Cannon (1979) tested the survival potential of the beetle when exposed to natural conditions, and suggested that the old house borer may have the potential for establishing natural populations.

## 2.2 BIOLOGY

Biological studies of H. bajulus have provided detailed information on its biology and habits. Durr (1954, 1956), Cymorek (1968), Serment (1976), Cannon and Robinson (1983, 1985), Mares and Robinson (1986) discussed morphological, physiological, and nutritional aspects of this cerambycid. The duration of the life stages of H. bajulus vary from continent to continent (Cannon and Robinson 1983). The reported periods for egg hatch is two weeks; larval stage several years, and pupal stage two weeks (St. George et al. 1957, McIntyre and St. George 1961).

Durr (1956) reported that old house borer eggs, oviposited by fertile females, have a thin chorion. Fertile females will deposit their eggs in available cracks and crevices of seasoned softwoods. The eggs are laid in 2 - 5 batches, each batch contains about 35 eggs (Cannon and Robinson 1983). Females may be attracted to the coniferous woods by  $\alpha$  and  $\beta$  pinenes (Becker 1943). Mares et al. (1986)

provided a detailed report of the morphological structure and function of the adult female's ovipositor. Cannon and Robinson (1983) evaluated egg incubation and percentage hatch.

Optimal development conditions for larvae include temperatures ranging from 82.4 - 84.2° F and relative humidity ranging from 40 - 50% (Becker 1943). Cannon and Robinson (1985) stated that temperature significantly influences larval growth and wood consumption. Larvae must maintain their body moisture content, with uptake exceeding the amount of moisture lost to the environment by evaporation and excretion. Larvae are able to derive water from the environment through absorption through the cuticle from the substrate, and absorption from air pockets, atmosphere, or galleries in the substrate (Durr 1956). In addition, metabolic water resulting from the oxidation of foodstuffs provides another source of moisture for the wood boring larvae (Durr 1956). A moisture content in pine sapwood of 8 - 10%, corresponding to 40 - 50% relative air humidity, represents the lower tolerance limit for old house borer larvae (Schuch 1937). Weight gain of larvae is dependent upon the moisture content of the nutrients (Schuch 1937). Vongkaluang et al. (1982) reported that a wood moisture content of 10% or less had adverse effects on first-instar larval growth and development.

The feeding habits of H. bajulus have been closely examined by Pallaske (1984). He reported that larvae will

support themselves both with the dorsal and ventral sides of their bodies while excavating galleries. They are incapable of tolerating unequal forces on their dorsal and ventral sides, subsequently, they seldom break the the surface of the wood while feeding. Larvae will feed within an annual ring and the tunnels created by their feeding habits are located beneath the surface of the wood (Pallaske 1984).

Rasmussen (1967) reported that the conclusion of larval life occurs at the limit of growth in the last ecdysis. The results from Rasmussen's work suggested that H. bajulus has nine larval molts and one pupal molt. Cymorek (1968) reported that long periods of moderate cooling, 8 weeks at 5° C, influenced the percentage of successful pupation and adult emergence more than the humidity conditions of 56, 76, and 90 percent relative humidity.

The adult life stage is relatively short compared to the duration of the larval form. Longevity for adult males usually ranges from 14 - 18 days, whereas females may live from 9 - 14 days (Durr 1956, Cannon and Robinson 1983). Adult H. bajulus are strong fliers, with an optimal flying temperature of 30° C (Cymorek 1968).



## 2.3 CONTROL

### 2.3.1 BIOLOGICAL CONTROL

Several insect species parasitize H. bajulus. A female Rhoptrocentrus piceus Marsh (Hymenoptera: Braconidae) can parasitize and kill four old house borer larvae, but is capable of paralyzing others (G. Becker 1979). Beetles in the family Cleridae are larval predators that can limit the population of H. bajulus larvae (Durr 1954, Serment 1976). Linsley (1964) stated that biological control agents probably have only a limited effect on restraining old house borer populations.

### 2.3.2 MECHANICAL CONTROL

The removal and burning of H. bajulus infested wood is a control method preferred by several European scientists (Durr 1954). Moore (1978) and Durr (1954) reviewed the temperature parameters required for eliminating infestations by heat sterilization. A cooling agent, dry ice (solidified carbon dioxide), was tested for possible lethal effects on accessible populations of H. bajulus (Vongkaluang 1978). She concluded that applying dry ice to surfaces, such as infested joists may be an inexpensive and safe method for controlling old house borer infestations. Knudsen et al. (1969) observed

that storing timber in fresh or salt water did not protect the wood from old house borer attack.

### 2.3.3 CHEMICAL CONTROL

Durr (1954) stated that H. bajulus infestations in coniferous wood may result in severe damage to the wood, due to the duration of the larval stage and its mode of feeding. He also discussed pretreatment of wood before construction by dipping the wood in or otherwise impregnating it with different preservatives, including creosote, arsenical liquids, pentachlorophenol, copper, and zinc naphthenate. In a mortality test reported by Robinson et al. (1981), pentachlorophenol (5.0%) and lindane (0.5%) provided 100% control against the egg, first-instar larva, and adult stages. In 1984 the U.S. Environmental Protection Agency (EPA) imposed restrictions on the use of creosote, pentachlorophenol and inorganic arsenicals (U.S. Federal Register 1984).

The United Kingdom in 1950 enacted Model Bye-Laws to protect new housing from old house borer infestation (Lea and Bravery 1978). Australia's Commonwealth Department of Health ordered that timber-framed houses infested with H. bajulus be fumigated (Hadlington and Campbell 1956).

Structural woods may be pretreated with wood preservatives prior to construction. Jacquot and Serment

(1971) investigated larval survival after a surface insecticide treatment test. They concluded that the vapor phase of the insecticide was responsible for killing larvae located beneath the point of penetration of the liquid phase. Residual activity of liquid insecticides is affected by the type of surface treated (Slominski and Gojmerac 1972) and humidity of the wood (Reichenbach and Collins 1984). Metzner et al. (1977) reported that the type of insect to be controlled influences the toxicity of the insecticide of choice more than does the categorization of that insecticide (hydrogen chloride, thiophosphate, or carbamate).

Cypermethrin was found to be more toxic to H. bajulus larvae than was permethrin (Berry 1984). Doppelreiter (1982, 1980) tested the effect of diflubenzuron upon eggs and newly hatched larvae of the old house borer. He concluded that diflubenzuron reduced egg hatch by 90% and caused complete kill or marginal survival of treated larvae. Taylor (1967) reported that boric acid at concentrations above 0.4 kg/m<sup>3</sup> (0.1%) were sufficient to prevent survival of newly hatched old house borer larvae.

Fumigation with an insecticidal gas can immediately reduce insect populations, but this method offers no residual activity against future infestations. Durr (1954) tested methyl bromide and reported that all H. bajulus larvae died 24 hours after the fumigation. Kenaga (1957) compared methyl bromide and sulfuryl fluoride in toxicity and penetration tests. Mori (1974) reported sulfuryl fluoride to be superior

to methyl bromide in capabilities for penetration into wood. The toxicity of the ovicidal effects of sulfuryl fluoride has been reviewed by Outram (1970) and Mori (1974).

Chlorpyrifos, [O,O-diethyl-O(3,5,6-trichloro-2-pyridyl)] phosphorothioate, is currently labeled for the control of H. bajulus. The insecticidal properties of chlorpyrifos were first described by Kenaga et al. (1965). The vapor pressure of this compound is relatively low,  $1.87 \times 10^{-5}$  mm Hg at 25° C (Worthing 1979). However, in spite of the low vapor pressure, Smith (1966) has stated that volatility is an important factor in decreasing the amount of residues on such surfaces as wood, metal, glass and paper. Smith (1966) also stated that the rate of chemical breakdown of chlorpyrifos by either hydrolysis or oxidation increases with an increase in temperature or pH. The stability of chlorpyrifos in ultraviolet light is influenced by the moisture content of the sample, with a very dry sample maintaining the greatest stability in ultraviolet light (Smith 1966).

## 3.0 CHAPTER I

### 3.1 INTRODUCTION

Hylotrupes bajulus, the old house borer, is a wood-destroying pest of seasoned softwoods (pine, spruce, fir). This cerambycid is considered an important pest throughout the world (Cannon and Robinson 1982, Becker 1962, White 1962, St. George et al. 1957, Durr 1956, 1954). In the U.S., populations of H. bajulus are known to inhabit only man-made structures; they apparently cannot survive in unprocessed woods (Moore 1978, Snyder 1955).

Pine lumber is composed primarily of sapwood, the portion of wood which contains the nutrients necessary for larval development. When larvae excavate galleries beneath the surface of the wood, loss of structural integrity may result. The rasping sounds made as the larvae tunnel through the sapwood, and the exit holes carved on the wood's surface by late-instar larvae, are additional nuisance aspects of old house borer infestations.

H. bajulus larvae may feed below the surface for 2 - 11 years (Cannon and Robinson 1983), making it difficult for the surface application of insecticides to effectively control the target pest. Therefore, the insecticide chosen as the control agent must have good wood penetration characteristics. The choice of a carrying agent (water or a

suitable base oil) and the formulation of the technical grade material may influence the penetration abilities of any insecticide applied to the wood surface.

Chlorpyrifos formulated as Dursban® Wood Treatment or as Dursban® Termiticide Concentrate may be toxic to target organisms beneath the surface of the wood if the chemical is capable of penetrating the surface of the wood. These insecticides are currently labeled for the prevention and control of H. bajulus infestations and may be diluted with water or a suitable base oil to prepare a final formulation suitable for surface application.

The objectives of the research reported here were to 1) compare the wood penetration abilities of a water-diluted formulation of chlorpyrifos, Dursban® Wood Treatment (WT), to the penetration abilities of a kerosene-based formulation of Dursban® WT; 2) compare the wood penetration abilities of a water-based formulation of chlorpyrifos, Dursban® Termiticide Concentrate (TC), to the penetration abilities of a kerosene-based formulation of Dursban® TC; and 3) determine the six-month residual longevity of Dursban® TC when applied to pine sapwood.

## 3.2 MATERIALS AND METHODS

### 3.2.1 PREPARATION OF SAMPLE

Two formulations of chlorpyrifos, the macroemulsion Dursban® WT, and the microemulsion Dursban® TC, were diluted with white kerosene or distilled water to prepare 0.5% and 1.0% (A.I.) final formulations. Wooden samples were cut into blocks (5 cm × 3 cm × 1.3 cm) from planed sapwood of southern yellow pine (Pinus spp.) with 10% - 15% moisture content. Wood moisture content was determined by a Delmhorst moisture meter (Delmhorst Instrument Comp., Boonton, N.J.). The blocks of sapwood contained no structural irregularities. Each formulation was brushed onto the pine blocks using two strokes in the direction of the wood grain. A one-inch wide paintbrush was first dipped into the insecticide formulation, and excess formulation was allowed to drain off the brush. The paintbrush was pulled across the wood surface at a 60° angle for three seconds per stroke. The paintbrush applied approximately 0.33 ml of Dursban® (approx. 0.16 g chlorpyrifos) onto each wooden block. The amount of chlorpyrifos was estimated by weighing each wooden block before and immediately after treatment, and with the assumption that an evenly dispersed mixture of chlorpyrifos and solvent was applied, the average amount of active ingredient was calculated. Morgan and Purslow (1973) stated that the application method of brushing an insecticide onto

a surface does not easily control the amount of active ingredient applied, although additional loadings do not guarantee improved penetration.

The treated blocks were allowed to dry at 70 - 74° F in a room in which 5 hours of fluorescent lighting were followed by 19 hours of darkness. A total of five blocks were treated with each prepared formulation.

Additional pine blocks were treated as described above with Dursban® TC (0.5% and 1.0%) and placed in an attic or a crawl-space area, both devoid of sunlight, from October (1985) to April (1986). The roof over the attic area was covered by asphalt shingles.

The temperature and relative humidity was recorded for a two day period at each location every other month during the field tests. The average temperature (°C) and relative humidity (%) for the months of October, December, and February were 24 and 82; -4 and 80; and 2 and 95, respectively, for the crawl-space area. For the attic area, the average recorded temperature (°C) and relative humidity (%) for the months of October, December, and February were 25 and 70; 3 and 66; and -6 and 65, respectively. After a six-month period the treated blocks were removed from both of these locations and prepared for analysis.

The center cubic centimeter of each treated wooden block was removed and sectioned with a microtome (40 or 50 microns/slice) from the top surface down. This provided a series of slices representing different depths to allow



determination of insecticidal penetration within each wooden block (Fig. 1).

Dursban® TC is a microemulsion of chlorpyrifos. This emulsion should penetrate deeper into a wooden surface than a macroemulsion (Dursban® WT), due to the difference in micelle size. Accounting for this physical attribute, samples were analyzed at greater depths for chlorpyrifos contents of Dursban® TC than for contents of Dursban® WT.

Each wooden slice from the treated blocks was weighed and placed in a glass vial containing 3 ml of analytical-grade hexane (redistilled), and stored in a freezer at -10° C until analysis. All analyses were performed within 1.5 months after sectioning.

### 3.2.2 RESIDUE ANALYSIS

Analysis of the chlorpyrifos content in each wooden slice was performed by gas chromatography using an electron capture detector (Guinivan et al. 1981, Greenhalgh and Cochrane 1980). Gas chromatograph conditions and analytical procedures were modified from the guidelines presented by the Pesticide Analytical Manual (Marcotte 1977), and by Claborn et al. (1968).

A Tracor 540 gas chromatograph, equipped with an electron capture detector employing Ni<sup>63</sup> as an ionization source, was used for all analysis. Nitrogen was used as the carrier gas with a flow rate of 40 ml/min. Operating conditions for the

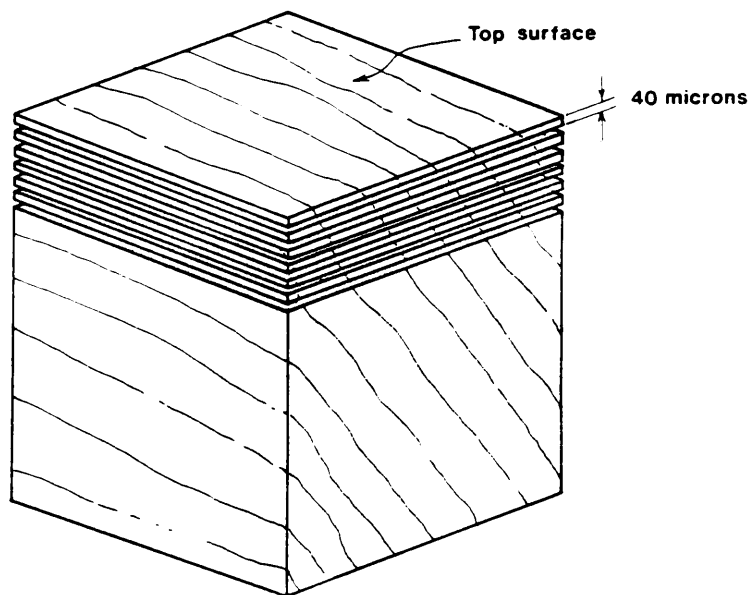
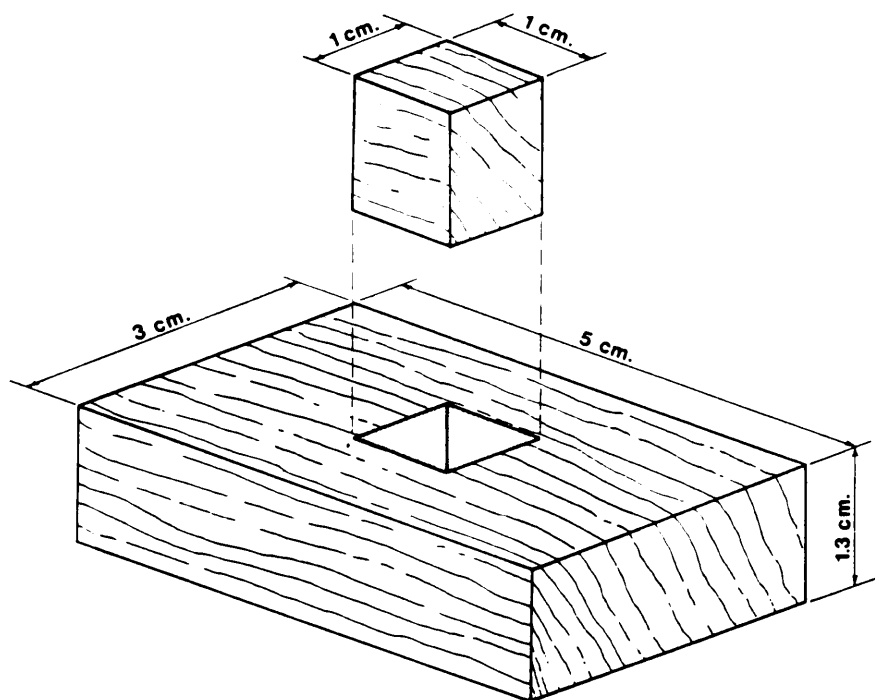


Figure 1. Preparation of wood sample for extraction of chlorpyrifos.

gas chromatograph were: oven temperature 210° C, injector temperature 215° C, and detector temperature 350° C. The samples were injected into a 2 mm ID, six-foot glass column that was packed with 10% SP-2100 on 100/120 Supelcoport. Peak areas were determined with a Hewlett-Packard model 3392A integrator.

Standard solutions of 0.10, 0.25, 0.50, and 1.00 ppm were made from technical grade chlorpyrifos of 99.8% purity (EPA Reference Standard, Batch #A821, Research Triangle Park, N. C.) in redistilled hexane. Two  $\mu$ l aliquots from each of the four standards were injected individually into the gas chromatograph. Calculations of the peak areas of replicate standards determined the linearity of detector response (average  $R^2 = 0.9953$ ).

Each glass vial containing a wood slice and hexane was shaken for 20 seconds on a Vortex mixer prior to an injection of a 2 $\mu$ l aliquot solution. Peak areas of replicate 2  $\mu$ l sample injections were compared to peak areas of standard solutions to determine the amount of chlorpyrifos contained in each sample. The recovery rate for high and low concentration values of chlorpyrifos was 71% with S.D.=  $\pm 9\%$ .

### 3.2.3 DATA ANALYSIS

Data obtained in the Dursban® WT, Dursban® TC and Dursban® TC longevity tests were statistically analyzed by a profile analysis technique (Ott 1984). In the analysis of

Dursban® WT and TC, the Least Significance Difference Test ( $p < 0.05$ ) was employed. The association between the amount of chlorpyrifos, time of treatment, and location of treated sample in the longevity test was measured using the procedure General Linear Models (GLM) on SAS (Statistical Analysis System 1982).

### 3.3 RESULTS AND DISCUSSION

#### 3.3.1 DURSBAN® WT

Formulations of Dursban® WT and kerosene carried significantly more chlorpyrifos below the surface of the wood than did the water-based formulations (Tables 1, 2). At a depth of 120 microns from the surface of the wood, 0.721 and 1.554  $\mu\text{g}$  chlorpyrifos/mg wood were detected in the 0.5% and 1.0% kerosene-based treatments, respectively. Lower levels, 0.337 and 0.879  $\mu\text{g}$  chlorpyrifos/mg wood, were detected in the 0.5% and 1.0% water-based treatments, respectively. The kerosene-based formulations of both concentrations of Dursban® WT continued to enhance chlorpyrifos penetration at depths of 440 microns.

At the 1.0% concentration level of Dursban® WT, the kerosene-based formulation carried almost twice the amount of chlorpyrifos detected at all the depths sampled than in the 0.5% kerosene-based treatment. The average amount of chlorpyrifos, 2.263  $\mu\text{g}$  chlorpyrifos/mg wood, detected at the

Table 1. Penetration of 0.5% Dursban® WT into pine blocks.

( $\bar{X}^1$  of  $\mu\text{g}$  chlorpyrifos/mg wood)

	Depth (Microns)					
	40	120	200	280	360	440
<b>Diluent</b>						
<b>Water</b>	0.537a <sup>2</sup>	0.337a	0.181a	0.121a	0.102a	0.115a
S.D. $\pm$	0.183	0.165	0.043	0.074	0.029	0.052
<b>Kerosene</b>	0.694a	0.721b	0.539b	0.461b	0.427b	0.457b
S.D. $\pm$	0.171	0.270	0.159	0.222	0.088	0.079

<sup>1</sup> G.C. analysis represent a recovery rate of 71%  $\pm$  9%

<sup>2</sup> Means in each column followed by the same letter are not significantly different (Profile Analysis; Least Significant Difference,  $p < 0.05$ ,  $n = 4$ )

Table 2. Penetration of 1.0% Dursban® WT into pine blocks.

( $\bar{X}^1$  of  $\mu\text{g}$  chlorpyrifos/mg wood)

	Depth (Microns)					
	40	120	200	280	360	440
<u>Diluent</u>						
<b>Water</b>	2.153a <sup>2</sup>	0.879a	0.325a	0.323a	0.138a	0.050a
S.D. $\pm$	1.178	0.500	0.231	0.438	0.095	0.045
<b>Kerosene</b>	2.263a	1.554b	1.160b	0.974b	0.772b	0.654b
S.D. $\pm$	0.825	0.329	0.151	0.291	0.270	0.146

<sup>1</sup> G.C. analysis represent a recovery rate of 71%  $\pm$  9%.

<sup>2</sup> Means in each column followed by the same letter are not significantly different (Profile Analysis; Least Significant Difference,  $p < 0.05$ ,  $n = 5$ )

surface of the wood (40 microns) at the 1.0% concentration level is considerably more than the amount of chlorpyrifos, 0.694  $\mu\text{g}$  chlorpyrifos/mg wood, detected at the 0.5% level.

Although the use of oil-based formulations has decreased since 1966 (Metzner et al. 1977), Berry (1984) and De Groot (1984) stated that the use of oils in emulsion formulations would yield an evenly distributed concentration of insecticide through a greater depth of wood than would the use of water. The data in this study indicate that the selection of a suitable base oil to dilute the preservative Dursban® WT would probably provide better control of H. bajulus larvae located beneath the surface of the wood than would water, due to superior penetration capabilities of the oil-based insecticide formulation.

### 3.3.2 DURSBAN® TC

From the upper-most layer of the wood's surface to a depth of 100 microns, chromatographic analysis showed higher concentrations of chlorpyrifos, 2.264 and 4.432  $\mu\text{g}$  chlorpyrifos/mg wood in 0.5% and 1.0% water-based formulations, respectively, than the comparatively lower concentrations of 0.869 and 1.254  $\mu\text{g}$  chlorpyrifos/mg wood detected in 0.5% and 1.0% oil-based formulations, respectively (Tables 3, 4). At depths of 250 microns to 600 microns, both the water and kerosene diluted formulations carried approximately the same amount of chlorpyrifos into

Table 3. Penetration of 0.5% Dursban® TC into pine blocks.

( $\bar{X}$ <sup>1</sup> of  $\mu\text{g}$  chlorpyrifos/mg wood)

	Depth (Microns)					
	50	100	250	400	500	600
<u>Diluent</u>						
<b>Water</b>	2.265a <sup>2</sup>	1.410a	0.515a	0.265a	0.163a	0.107a
S.D. $\pm$	0.850	0.413	0.151	0.116	0.078	0.058
<b>Kerosene</b>	0.869b	0.637b	0.702a	0.287a	0.245a	0.244a
S.D. $\pm$	0.251	0.178	0.257	0.046	0.034	0.090

<sup>1</sup> G.C. analysis represent a recovery rate of 71%  $\pm$  9%.

<sup>2</sup> Means in each column followed by the same letter are not significantly different (Profile Analysis; Least Significant Difference,  $p < 0.05$ ,  $n = 5$ )



Table 4. Penetration of 1.0% Dursban® TC into pine blocks.

( $\bar{X}$ <sup>1</sup> of  $\mu\text{g}$  chlorpyrifos/mg wood)

	Depth (Microns)					
	50	100	250	400	500	600
<u>Diluent</u>						
<b>Water</b>	4.434a <sup>2</sup>	3.080a	1.106a	0.409a	0.313a	0.172a
S.D. $\pm$	0.985	0.691	0.412	0.406	0.328	0.139
<b>Kerosene</b>	1.254b	1.093b	0.838a	0.546a	0.415a	0.347a
S.D. $\pm$	0.242	0.370	0.341	0.190	0.146	0.139

<sup>1</sup> G.C. analysis represent a recovery rate of 71%  $\pm$  9%.

<sup>2</sup> Means in each column followed by the same letter are not significantly different (Profile Analysis; Least Significant Difference,  $p < 0.05$ ,  $n = 5$ )

the wood. Baker and Taylor (1967) tested the action of certain insecticides against H. bajulus, and they reported that 50% of the larvae were distributed in the outer centimeter of the untreated wood blocks. If chlorpyrifos is capable of penetrating 600 microns (0.06 cm) below the wood's surface, then this insecticide could come in contact with a majority of the larval population.

The selection of water as the carrying agent for Dursban® TC is a significantly better choice than is a suitable base oil for yielding a high concentration of chlorpyrifos at the surface of the wood.

Dursban® TC was initially formulated by Dow Chemical as an insecticide restricted for use by certified applicators. The selection of a flammable and odorous compound, such as kerosene, as the diluting agent may not be advantageous for the applicator or the client when applying chlorpyrifos formulations to wooden surfaces. The selection of water as a carrier for Dursban® TC instead of an oil would lower the cost of treatment, ultimately reducing the cost of the treatment for the client. Thus a consideration of the economics, as well as the penetration capabilities of water-diluted formulations of Dursban® TC and the undesirable characteristics of kerosene in surface application of insecticides, water is the diluent of choice for Dursban® TC.

### 3.3.3 LONGEVITY OF DURSBAN® TC

There was a significant difference between chlorpyrifos concentration and wood depth detected at each test location (Table 5, 6). At the laboratory location, a 24-hour analysis of chlorpyrifos penetration was conducted. At both the attic and crawl-space locations, a six-month analysis of chlorpyrifos content was conducted. Various depths were sampled from all of the treated blocks at each location.

There was a significant difference in chlorpyrifos content observed between the application concentrations (0.5% and 1.0%) and the depths sampled (50 - 600 microns).

There was a significant difference between the analysis at 24 hours and the attic and crawl-space analyses at six months at depths of 50 - 100 microns for both concentrations. At a depth of 100 microns in the 1.0% formulation, the 24 hour analysis detected 3.082  $\mu\text{g}$  chlorpyrifos/mg wood. The analyses of the attic and crawl-space blocks at six months detected lower amounts of chlorpyrifos at 100 microns, showing 0.394 and 0.630  $\mu\text{g}$  chlorpyrifos/mg wood, respectively. At the depths of 250 - 600 microns, no significant differences between the locations were measured.

The amounts of chlorpyrifos in the blocks placed in the attic and crawl-space areas, resulting from the two application concentrations, were not significantly different. At the 1.0% concentration, the amount of chlorpyrifos detected at depth 400 microns for the attic and

Table 5. Longevity of 0.5% Dursban® TC.

( $\bar{X}$ <sup>1</sup> of  $\mu\text{g}$  chlorpyrifos/mg wood)

	Depth (Microns)					
	50	100	250	400	500	600
<b>Location</b>						
<b>Lab<sup>2</sup></b>	2.265	1.410	0.515	0.265	0.163	0.107
S.D. $\pm$	0.850	0.413	0.151	0.116	0.078	0.058
<b>Attic<sup>3</sup></b>	0.141	0.117	0.207	0.191	0.227	0.215
S.D. $\pm$	0.068	.040	.098	0.103	0.144	0.040
<b>Crawl<sup>3</sup></b>	0.210	0.385	0.200	0.192	0.182	0.165
S.D. $\pm$	0.061	0.127	0.027	0.115	0.054	0.103

<sup>1</sup> G.C. analysis represent a recovery rate of 71%  $\pm$  9%, with n = 5

<sup>2</sup> 24 hour analysis

<sup>3</sup> 6 month analysis

Table 6. Longevity of 1.0% Dursban® TC.

( $\bar{X}$ <sup>1</sup> of  $\mu\text{g}$  chlorpyrifos/mg wood)

	Depth (Microns)					
	50	100	250	400	500	600
<u>Location</u>						
<b>Lab</b> <sup>2</sup>	4.434	3.080	1.106	0.409	0.313	0.172
S.D. $\pm$	0.985	0.691	0.412	0.406	0.328	0.139
<b>Attic</b> <sup>3</sup>	0.191	0.394	0.539	0.538	0.370	0.374
S.D. $\pm$	0.080	0.148	0.380	0.066	0.075	0.107
<b>Crawl</b> <sup>3</sup>	0.612	0.631	0.450	0.548	0.330	0.307
S.D. $\pm$	0.247	0.272	0.128	0.325	0.294	0.246

<sup>1</sup> G.C. analysis represent a recovery rate of 71%  $\pm$  9%, with n = 5

<sup>2</sup> 24 hour analysis

<sup>3</sup> 6 month analysis

crawl-space locations were 0.540 and 0.550 µg chlorpyrifos/mg wood, respectively.

The longevity of the larval stage of *H. bajulus* requires longevity of the active ingredient in an insecticide in order for that insecticide to be an effective controlling agent. These results suggest that during a six-month time period, a certain amount of chlorpyrifos is lost from the immediate surface of the wood. Some time after application, the Dursban® TC formulation of chlorpyrifos might fail to prevent egg hatch or to provide adequate control of first-instar larvae as they bore into treated surfaces. Late-instar larvae might not be adversely affected by these lower amounts of chlorpyrifos as they approach the surface of the wood when excavating exit holes. Fertilized female *H. bajulus* might not be able to detect minute quantities of chlorpyrifos and would therefore not be repelled from potential ovipositioning sites if certain amounts of chlorpyrifos have repellent properties. An insecticide might, however, mask the odor of pinenes, attractants emitted from pine sapwood that Becker (1943) found to enhance oviposition.

Losses of insecticide from treated wood may occur through volatilization, leaching with water, and by chemical change (Morgan and Purslow 1973). Orsler and Stone (1981) stated that the outermost 2 mm is the zone from which the majority of the volatile losses of insecticides takes place.

The amounts of chlorpyrifos detected at depths of 400 - 600 microns did not significantly change between the 24 hour

analysis and the six-month analyses. The concentrations of chlorpyrifos detected will essentially remain constant during a six-month interval (Fig. 2). If an infested structure is treated in April, the quantity of chlorpyrifos should be equal in amount six months later, in October. Schaefer et al. (1970) and Wade (1968) showed the high degree to which chlorpyrifos adsorbs to organic solids.

No statistically significant difference was found in the amount of chlorpyrifos detected in the treated wood six months after the application of 0.5% and 1.0% formulations of Dursban® TC. Without toxicological data, it is difficult to suggest if these reported amounts of chlorpyrifos differ in relative toxicity to H. bajulus larvae.

#### 3.4 CONCLUSION

The diluent of choice for Dursban® WT is an oil-based carrier, whereas the diluent of choice for Dursban® TC is water, for providing best penetration of the active ingredient, chlorpyrifos. The formulation of Dursban® and its diluting agent influences the penetration abilities of the final spray when it is applied to pine sapwood.

Residual amounts of Dursban® TC applied to pine sapwood will remain relatively constant six months after the initial treatment under tested conditions at depths of 400 - 600 microns below the treated surface.

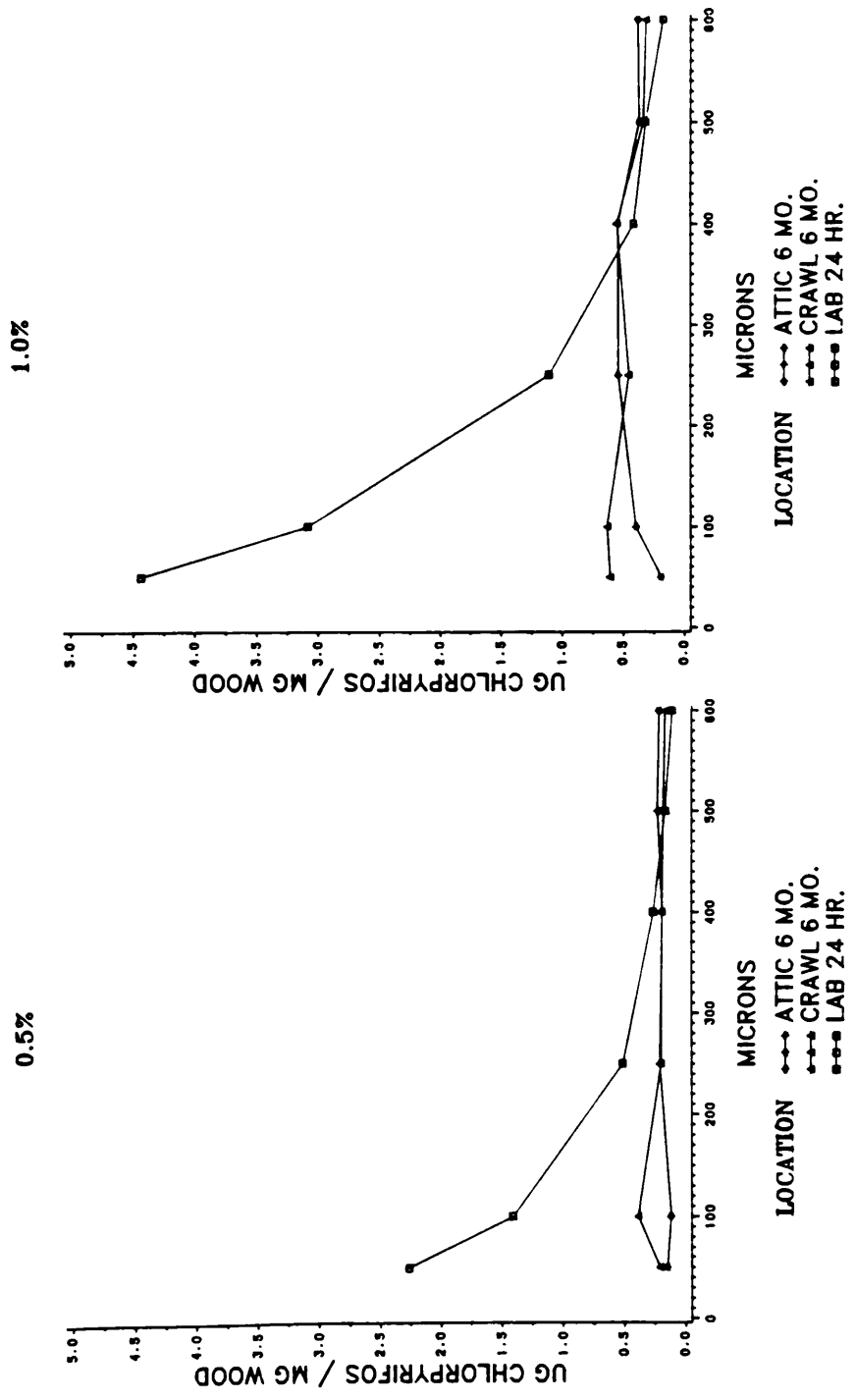


Figure 2. Depth and location effect on chlorpyrifos penetration.



## 4.0 CHAPTER II

### 4.1 INTRODUCTION

The old house borer, Hylotrupes bajulus, is an important pest of seasoned softwoods. This cerambycid has established a world-wide distribution (H. Becker 1968, 1979). In the United States, H. bajulus, the old house borer, is considered second only to termites as the most important insect pest in the eastern and Gulf Coast States (St. George et al. 1957, McIntyre and St. George 1961).

The larval stage of this longhorned beetle feeds in seasoned softwoods (pine, spruce, fir). This life stage may feed in infested wood for 2 - 11 years (Cannon and Robinson 1983). The growth and development of larvae is influenced by the moisture content of the infested wood; the optimal moisture content is 10 - 12 percent (Becker 1949). Durr (1956) reported that larvae were able to develop in timber with a moisture content as low as 7.5%. Becker (1977) stated that natural populations of H. bajulus in Germany may be located in wooden posts at least 0.5 meters above the ground, away from the wet area of the pole contacting the ground.

The moisture content of a piece of structural wood is influenced by the geographic area in which it is located; the season of the year; the operation of climate control units (air conditioners, electric heaters); and the location of the

wood within the structure (Robinson 1986). The thickness of the piece of wood (Kubler 1982), and whether the wood is finished also affects wood moisture content (Verrall 1965).

Relative humidity and temperature influence wood moisture content (WMC). Wood is a hygroscopic material; it absorbs and gives off water vapor in order to establish an equilibrium between the moisture content of the wood and the humidity of the surrounding air. Barlow and Hadaway (1968), observed the interactions between insecticides, cellulose, and water. They reported that the efficiency of a spray application may vary with time as humidity changes throughout a day or from season to season. When humidity changes from 20% to 80%, the amount of water held by the wood fibers increased by 9.4% (Barlow and Hadaway 1968). Bois (1959) reported that the moisture content of wood located in basements was approximately 9% greater during the summer months than the winter months.

Insecticide residues on cellulose materials, such as wood, are often influenced by the interactions between the relative humidity of the atmosphere and the moisture content of the wood (Barlow and Hadaway 1968). They reported that surface diffusion influences the depth and uniformity of penetration of the insecticide's active ingredient.

The objectives of the research presented here were to 1) determine the wood penetration abilities of Dursban® TC into pine sapwood at various moisture contents; and 2) predict the final endpoint of Dursban® TC penetration into pine sapwood.

## 4.2 MATERIALS AND METHODS

### 4.2.1 PREPARATION OF SAMPLE

A formulation of chlorpyrifos, Dursban® TC, was diluted with distilled water to prepare a 1.0% (A.I.) final formulation. Wooden blocks (5 cm × 3 cm × 1.3 cm) were cut from matched samples of southern yellow pine (Pinus spp.). The blocks were free from obvious defects, such as fungal stains and knots. An Eppendorp pipette was used to apply 250 µl of 1.0% chlorpyrifos solution to the center of the horizontal wood surface. A total of ten blocks were treated: five blocks had a WMC of 7.5% - 8.0%, and five blocks had a WMC of 14.5% - 16.0%. The WMC was determined by a Delmhorst moisture meter (Delmhorst Instrument Comp., Boonton, N.J.). The low WMC represented the WMC of structural timber located inside a house in the mountain geographical area in eastern U.S. during the winter, and the high WMC represented the WMC of the same structural wood during the summer (Bois 1959). The treated blocks were allowed to dry at 72° F for a 24-hour period in a darkened room.

The center cubic centimeter of each treated wooden block was removed and sectioned immediately after the 24-hour drying period. Sectioning was performed with a microtome at 50 microns/slice from the top surface down to a depth of 950 microns, providing a series of slices representing different depths in order to allow determination of insecticidal

penetration within each block (Fig. 1, p. 16). Each wooden slice collected from the treated blocks was weighed and placed in a glass vial containing 3 ml of analytical-grade hexane (redistilled), and stored in a freezer at  $-10^{\circ}$  C until analysis. All analyses were performed within 1 month after sectioning.

#### 4.2.2 RESIDUE ANALYSIS

Analysis of the chlorpyrifos content in each wooden slice was performed by gas chromatography, as described in section 3.2.2. The analysis procedure was modified by using standard solutions of 0.10, 0.25, 0.50, 1.00, 1.50, 2.00, and 2.50 ppm of technical grade chlorpyrifos in hexane. A 2  $\mu$ l aliquot of the appropriate standard solution was injected into the gas chromatograph. The amount of chlorpyrifos in each sample extract was quantified by the integration of the previously injected standard peak area. The recovery rate for high and low concentration values of chlorpyrifos was 71% with S.D. =  $\pm 9\%$ .

#### 4.2.3 DATA ANALYSIS

The association between wood moisture content and depth of penetration was statistically analyzed by a profile analysis technique (Ott 1984) using the procedure General Linear Models (GLM) on SAS (Statistical Analysis System

1982). The prediction of the final endpoint of Dursban® TC penetration was determined using an two parameter exponential (Myers 1986).

#### 4.3 RESULTS AND DISCUSSION

##### 4.3.1 WOOD MOISTURE CONTENT

Depth of penetration of chlorpyrifos was not significantly related to the moisture content of the treated wood (Table 7). At a depth of 100 microns, analysis of the treated wood with low moisture contents (7.5 - 8.0%) and high moisture contents (14.5 - 16.0%) detected 4.028 and 3.770  $\mu\text{g}$  chlorpyrifos/mg wood, respectively. At depths of 700 - 950 microns the quantity of chlorpyrifos in sample extracts from both WMC levels differed only slightly. The concentrations of chlorpyrifos were essentially constant at similar depths in samples with varying wood moisture contents.

The majority of the chlorpyrifos applied to the surface of the wood remained in the outermost wood cells, as compared to the amount which penetrated to depths of 600 - 950 microns (Fig. 3). Orsler and Stone (1982) reported that the bulk of lindane insecticide in surface applications remained near the surface of the wood.

When processed wood is drying, movement of water throughout the wood may occur through minute channels in the wood cell. Water bound to the cell wall may move through

Table 7. Penetration of 1.0% Dursban® TC into pine sapwood of various moisture contents.

( $\bar{X}$ <sup>1</sup>  $\mu\text{g chlorpyrifos/mg wood}$ )

	Depth (Microns)									
	50	100	250	400	500	600	700	800	900	950
<b>Low</b>	4.072	4.027	2.288	1.667	0.422	0.421	0.173	0.078	0.093	0.013
<b>S.D. <math>\pm</math></b>	0.293	0.427	2.110	1.877	0.254	0.304	0.128	0.045	0.093	0.023
<b>High</b>	4.392	3.769	2.921	1.340	0.695	0.296	0.168	0.112	0.093	0.038
<b>S.D. <math>\pm</math></b>	1.202	1.428	1.266	0.470	0.328	0.162	0.175	0.147	0.129	0.046

<sup>1</sup> G.C. analysis represent a recovery rate of 71%  $\pm$  9%, with n = 4

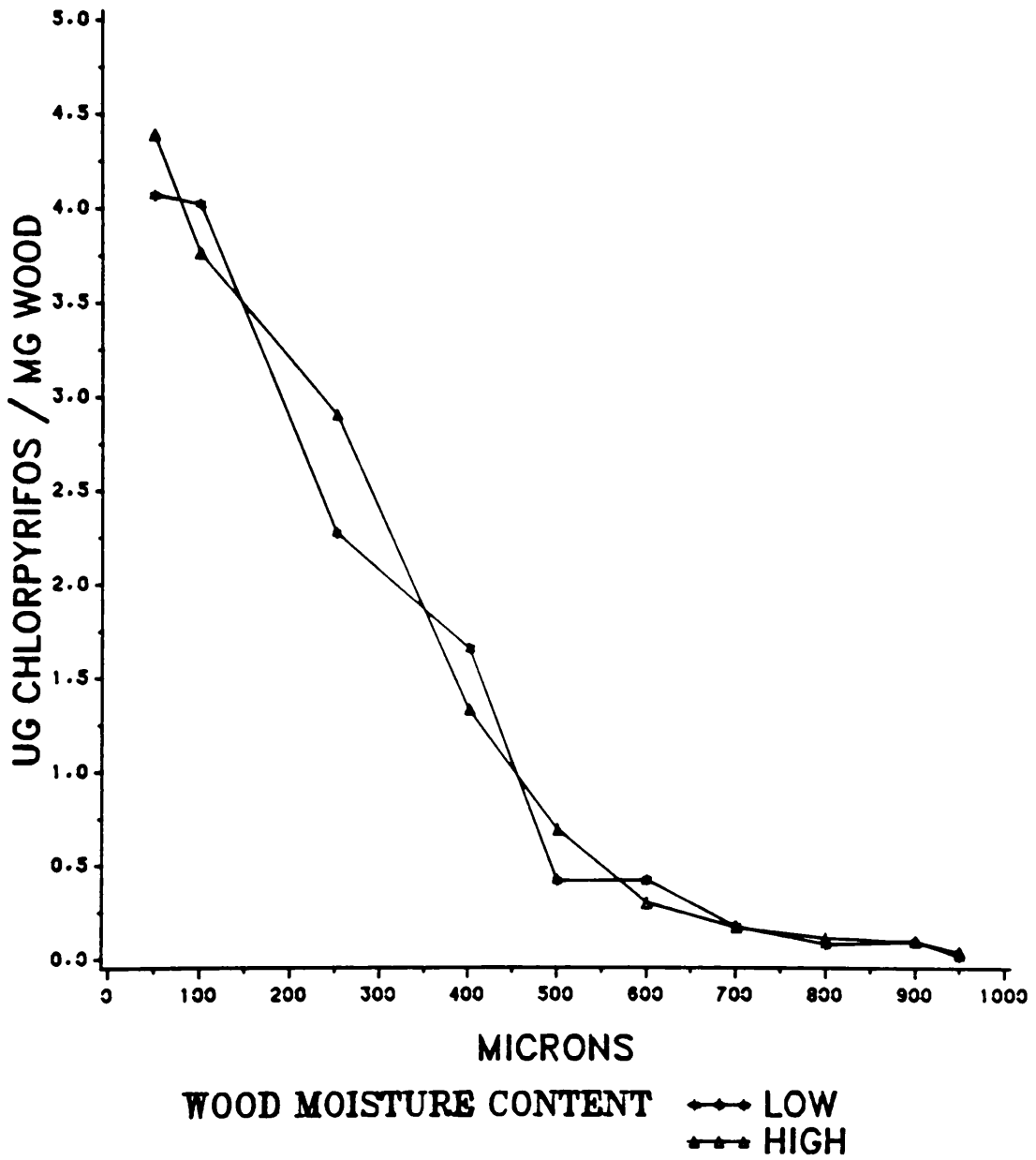


Figure 3. Wood moisture effect on Dursban® TC penetration.

various passageways by diffusion of water vapor resulting from the change of relative humidity inside the wood. Water found inside the cell lumen will flow primarily through cell cavities and small openings in the cell wall by capillary action (Rasmussen 1961). The different degrees of WMC (low and high) of the blocks in this evaluation might not have differed enough to significantly influence the amount of movement of water-diluted chlorpyrifos by diffusion or capillary action.

#### 4.3.2 ENDPOINT PREDICTION

A model-based prediction was made to determine the maximum penetration of chlorpyrifos by a nonpressure application of the insecticide, Dursban® TC. With a 95% lower confidence level, a "trace" amount (0.0125 µg) of chlorpyrifos will penetrate to a depth of approximately 1320 microns when Dursban® TC is applied to planed pine sapwood (Fig. 4). A "trace" amount of chlorpyrifos represents the lower limit of detection of chlorpyrifos by previously reported gas chromatograph analysis. This conservative prediction of the final endpoint of penetration of 1.0% Dursban® TC is based on the data collected in section 4.3.1. and applied to the non-linear model:  $y = ae^{\beta \times \text{depth}} + E$ .

By employing this non-linear model, with  $R^2 = 84\%$  and a lower confidence interval of 95%, the prediction that the



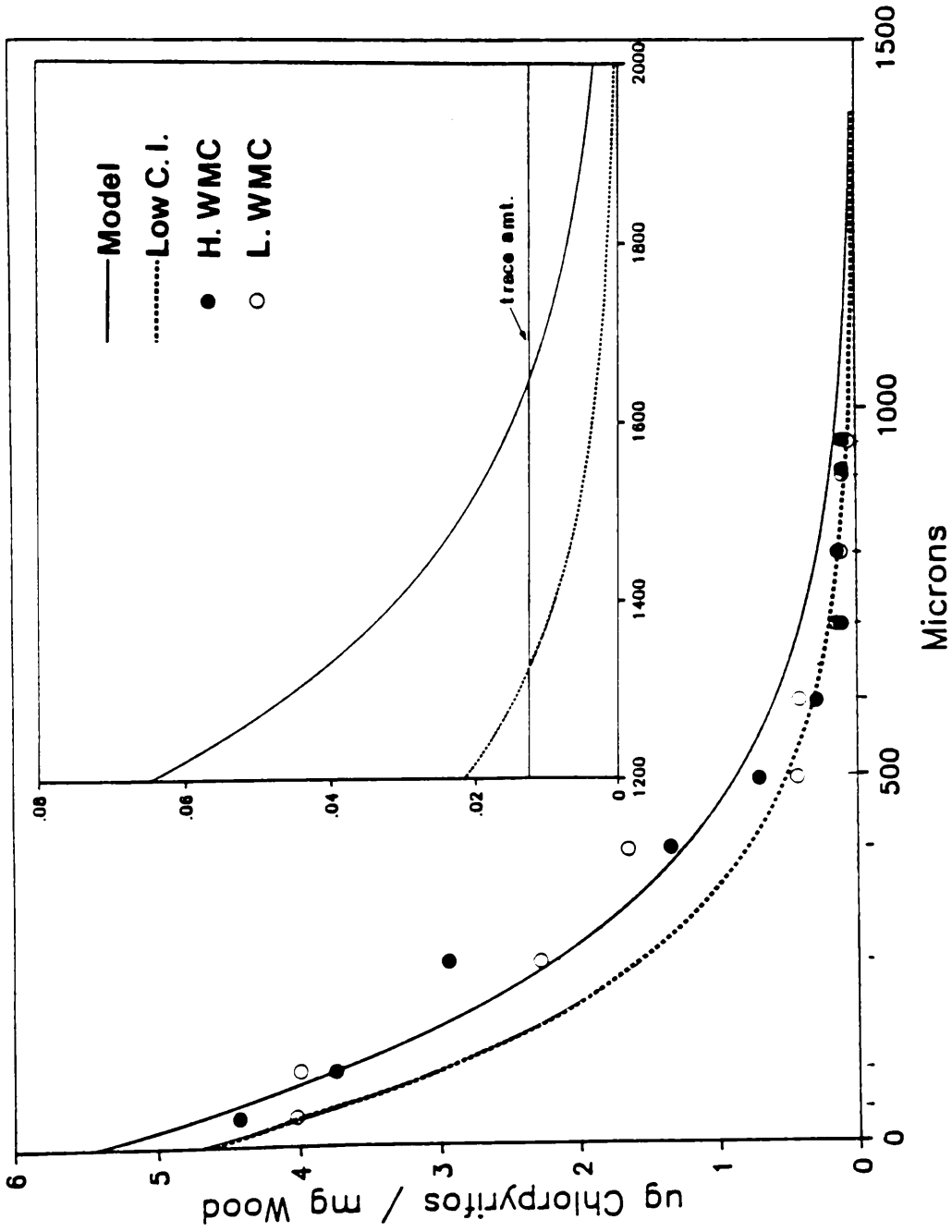


Figure 4. Prediction of the penetration endpoint of Dursban® TC employing the model  $y = \alpha e^{\beta \times \text{depth} + E}$  with  $\alpha = 5.422888$  and  $\beta = -0.003687$ .

limited detection of chlorpyrifos will occur at the depth of 1320 microns is made assuming that the model extrapolates from the observed depths of penetration. When variability due to experimental error and recovery percentage are taken into account, the predicted endpoint of penetration has a relative good fit to the observed data.

The prediction of the final endpoint excludes the movement of insecticides through galleries excavated by tunneling beetle larvae, resin canals, knots and checks, and through other defects in treated wood. The endpoint data does not presume uniform distribution of chlorpyrifos at the deeper zone of penetration.

#### 4.3.3 CONCLUSION

The wood moisture content of treated pine sapwood did not significantly influence the penetration abilities of Dursban® TC at tested WMC. The lower tolerance level of WMC (8 - 10%) for the growth and development of *H. bajulus* larvae (Schuch 1937) was included in this evaluation. High WMC, approaching fiber saturation (approximately 26%), was not tested. Becker (1977) reported that larvae were not located in the wet areas of an infested wooden pole, suggesting that these larvae avoid extremely wet wood.

A 1.0% water-diluted formulation of Dursban® TC applied to planed pine sapwood may penetrate 1320 microns away from the treated surface. The untreated wood below the predicted

point of penetration may provide a secure feeding site for surviving H. bajulus larvae. Orsler and Stone (1982) stated that the ability of an insecticide to provide an initial kill will be directly related to the penetration pattern of that insecticide into the treated lumber.

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