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*Aquatic Plant Control Research Program*

# Herbicide Concentration/Exposure Time Requirements for Controlling Submersed Aquatic Plants: Summary of Research Accomplishments

*by Kurt D. Getsinger, Michael D. Netherland*

WES



APCRP

Prepared for Headquarters, U.S. Army Corps of Engineers

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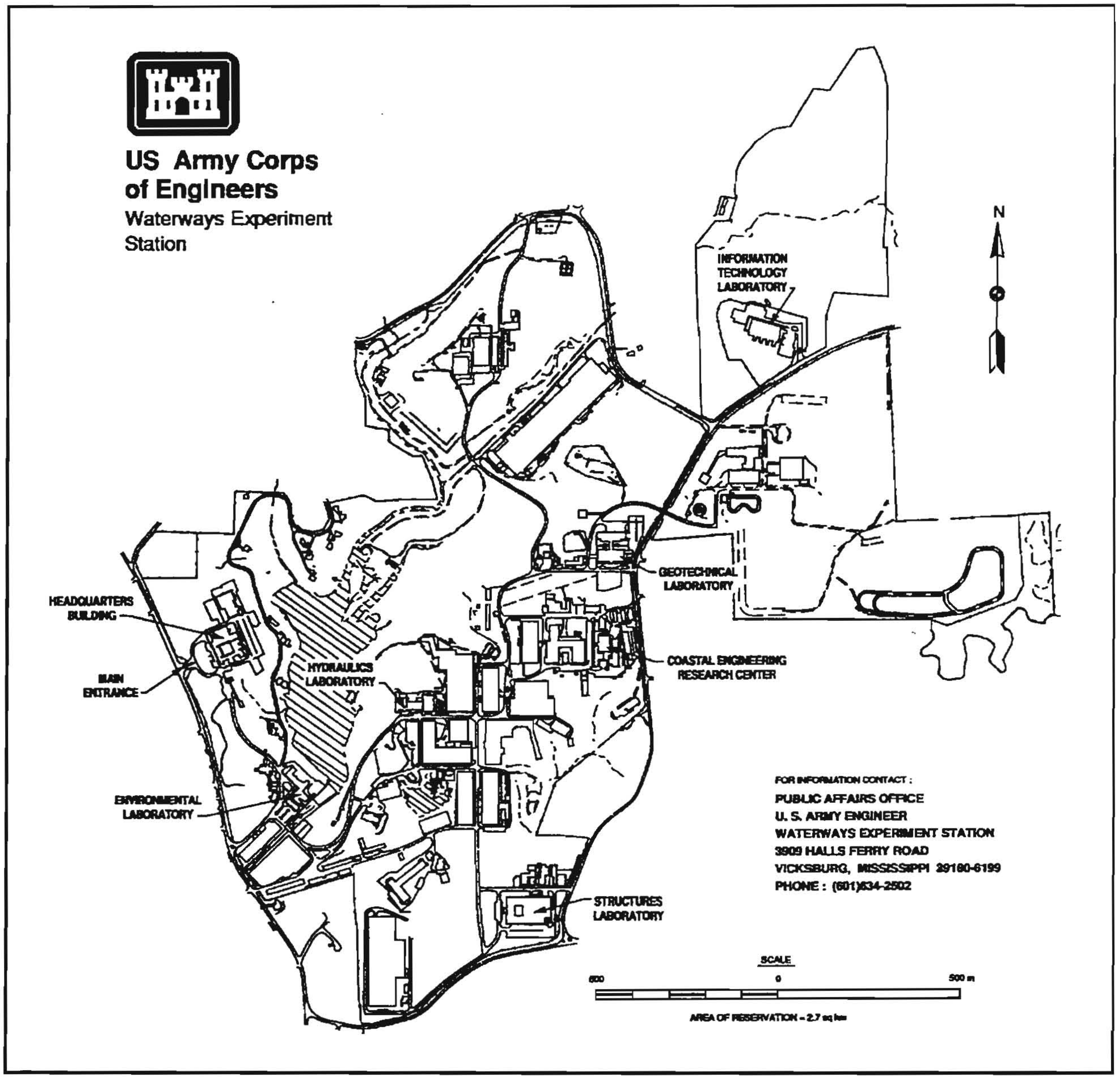


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# Preface

The work reported herein was conducted as part of the Aquatic Plant Control Research Program (APCRP), Work Unit 32352. The APCRP is sponsored by the Headquarters, U.S. Army Corps of Engineers (HQUSACE), and is assigned to the U.S. Army Engineer Waterways Experiment Station (WES) under the purview of the Environmental Laboratory (EL). Funding was provided under Department of the Army Appropriation No. 96X3122, Construction General. The APCRP is managed under the Center for Aquatic Plant Research and Technology (CAPRT), Dr. John W. Barko, Director. Mr. Robert C. Gunkel was Assistant Director for the CAPRT. Program Monitor during this study was Ms. Denise White, HQUSACE. Additional funding and/or logistical support were provided by the Tennessee Valley Authority, the U.S. Bureau of Reclamation, and the U.S. Department of Agriculture.

The Principal Investigator for this report was Dr. Kurt D. Getsinger, Ecosystem Processes and Effects Branch (EPEB), Environmental Processes and Effects Division (EPED), EL, WES. The report was prepared by Dr. Getsinger and Mr. Michael D. Netherland, EPEB. Technical reviews of this report were provided by Dr. Susan Sprecher and Ms. Linda Nelson, EPEB. In addition, many individuals from Federal, State, and local agencies, universities, and the private sector made possible much of the research summarized in this report. DowElanco, DuPont, Elf Atochem, Griffin, Rhone-Poulenc, SePRO, and Zeneca chemical companies provided the herbicides used in these studies.

The investigation was performed under the general supervision of Dr. John Harrison, Director, EL, and Dr. Richard E. Price, Chief, EPED.

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# 1 Introduction

## Background

The focus of the Aquatic Plant Control Research Program's (APCRP) Chemical Control Technology Area is to develop environmentally compatible techniques for herbicides and plant growth regulators (PGRs) that provide improved tools for managing nuisance aquatic vegetation (Getsinger and Decell 1992). In recent years, several lines of research have been pursued within this technology area, including the following: (a) Herbicide Concentration and Exposure Time Studies; (b) Herbicide Application Technique Development for Flowing Water; (c) Field Evaluation of Selected Herbicides for New Aquatic Uses; (d) PGRs for Aquatic Plant Management; (e) Herbicide Delivery Systems; (f) Species-Selective Use of Aquatic Herbicides and PGRs; and (g) Coordination of Control Tactics with the Phenology of Aquatic Plants. Studies in these work units are conducted at the U.S. Army Engineer Waterways Experiment Station (WES) in Vicksburg, MS, the WES's Lewisville Aquatic Ecosystem Research Facility in Lewisville, TX, and at selected field locations throughout the country.

Although the chemical control work units function as individual research efforts, they have been carefully designed and scheduled to act as integral components for the successful development of improved application techniques. As structured, these integrated work units collectively support the development and evaluation of safe and effective chemical formulations and application techniques for the aquatic environment. Consequently, aquatic plant managers are provided with effective operational techniques that minimize chemical dose, while maximizing the control of target plants, reducing the amount of chemicals placed in the environment, minimizing effects on nontarget plant species, and decreasing the effort and costs associated with aquatic applications.

An important outcome of the chemical control research activities has been the close working relationship that WES scientists have developed with the chemical industry and the U.S. Environmental Protection Agency (USEPA). This cooperation enables chemical control researchers to stay informed of the latest developments in aquatic pesticides and regulation requirements. In addition, interaction with U.S. Army Corps of Engineers Districts and other Federal agencies responsible for aquatic plant management activities, such as the Tennessee Valley Authority, the U.S. Bureau of Reclamation, and the U.S. Department of Agriculture, is necessary to coordinate and focus resources on regional and national problems. Finally, cooperation with State and local aquatic plant management programs and institutional research facilities is maintained to augment WES's laboratory and field research capabilities.

In response to recent reductions in APCRP funding levels, four of the Chemical Control Technology Area work units were terminated prematurely in Fiscal Year 1996: (a) Herbicide Concentration and Exposure Time Studies; (b) Herbicide Application Technique Development for Flowing Water; (c) Field Evaluation of Selected Herbicides for New Aquatic Uses; and (d) PGRs for Aquatic Plant Management. As work in these areas is completed, a series of reports will be published to summarize and document the final accomplishments in these terminated work units. This report is a product in that series.

## Herbicide Concentration/Exposure Time (CET) Relationships

The success or failure of a herbicide treatment designed to control submersed plants will primarily depend upon two related factors: (a) the concentration of herbicide that comes in contact with the target plant, and (b) the length of time a target plant is exposed to dissipating concentrations of herbicide in the water column. Chemical applications to entire water bodies or small static-water systems are generally successful, since target plants are exposed to lethal concentrations of herbicides for sufficient time periods. However, flowing-water systems and large reservoirs, in which a high rate of water exchange can occur over a relatively short period of time, present unique problems to the applicator. As the rate of water exchange increases in these situations, both the concentration of the herbicide and the length of exposure time decrease, potentially resulting in poor plant control. Therefore, basic relationships must be defined between herbicide concentration and the exposure time required to achieve desired plant control against major nuisance species, such as Eurasian watermilfoil (*Myriophyllum spicatum* L.) and hydrilla (*Hydrilla verticillata* Royle).

To complicate matters, the unique properties of the active ingredient (ai) in each aquatic herbicide require that CET relationships be developed individually for each target plant. These characteristic properties include rate of application, mode of action, environmental half-life, plant uptake rate, biomass, growth stage, and plant susceptibility (species selectivity). For example, use rates and exposure periods for contact herbicides (used as milligrams ai per liter per hour) can differ dramatically from some systemic herbicides (applied as micrograms ai per liter per week). The principle guiding these CET studies was the expectation that as exposure time was increased, lower concentrations of herbicide would be required to achieve desired plant control.

In 1986 a research work unit was initiated to (a) define, under controlled conditions, CET relationships for the most widely used aquatic herbicides, registered by the USEPA or under development via a USEPA Experimental

Use Permit, that are effective against Eurasian watermilfoil and/or hydrilla, and (b) use specific CET relationships to provide guidance for improving the control of those nuisance submersed plants in a variety of field situations. These research objectives were accomplished through a series of small-scale laboratory, growth chamber, and greenhouse studies; mid-scale outdoor mesocosm studies; and large-scale field verification and operational demonstrations. Products evaluated included the systemic herbicides 2,4-D, fluridone, triclopyr, and bensulfuron methyl and the contact herbicides endothall, diquat, and copper.

Using the previously described integrated work unit approach, critical information from the Herbicide Application Technique Development for Flowing Water work unit (Getsinger, Fox, and Haller 1996) was matched with results from many of these CET studies to design improved application techniques for specific flowing water situations. The methodologies spawned during this long-term research effort have substantially improved the management of Eurasian watermilfoil and hydrilla in rivers, lakes, and reservoirs throughout the United States in a fashion that effectively and efficiently uses herbicides, while minimizing impacts on nontarget species. Moreover, the determination of herbicide CET relationships has allowed for improved predictability of plant control in hydrodynamic systems. At present, these innovative treatment techniques are being used to manage other target species in a variety of field situations.

Although results from these studies were primarily used to establish effective CET relationships for each aquatic herbicide and selected target plant, this information was also used in the Herbicide Delivery Systems work unit to assist in the development of innovative application techniques (Netherland 1991b; Netherland and Getsinger 1991). These techniques are designed to minimize the amount of herbicide needed for a particular situation, while maximizing efficacy. Data obtained from this effort were also used in the herbicide fate model developed in the APCRP Simulation Technology Area.

This report describes the critical phases of this CET work unit while summarizing major research findings and accomplishments. These summaries are organized and presented by chemical compounds: the systemic herbicides 2,4-D, fluridone, triclopyr, and bensulfuron methyl and the contact herbicides endothall, diquat, and copper. Details of individual studies conducted under this work unit can be found in specific publications cited throughout this report and listed in the Reference section.

# 2 Systemic Herbicides

## 2,4-D

Preliminary investigations on determining CET relationships for controlling Eurasian watermilfoil (hereafter referred to as milfoil) with the systemic herbicide 2,4-D (2,4-dichlorophenoxy acetic acid) were initiated in an aquarium-type diluter system housed in a controlled-environment greenhouse (Hall et al. 1982; Westerdahl and Hall 1983; Westerdahl et al. 1983). These studies showed that 2,4-D threshold concentrations required to produce 50-percent injury of milfoil ranged from 0.05 mg/L for 3.5-weeks exposure to 0.10 mg/L for 1-week exposure.

More extensive 2,4-D CET studies were conducted against milfoil in a modified and improved version of this diluter system under controlled-environment conditions (Westerdahl 1986; Westerdahl 1987; Green 1988; Green and Westerdahl 1988; Green 1989; Green and Westerdahl 1990; Netherland 1991a). In these evaluations, plant injury increased and biomass decreased with increasing 2,4-D concentrations and exposure times, to a threshold above which satisfactory plant control was achieved. Severe milfoil injury occurred with exposure to 0.5 mg acid equivalent (ae)/L for 72 hr, 1.0 mg ae/L for 36 hr, and 2.0 mg ae/L for 24 hr. Threshold levels for milfoil control were established in the 1.0 mg ae/L for 48-hr exposure and 2.0 mg ae/L for the 36- and 48-hr exposures.

A series of laboratory and field evaluations for developing controlled-release (CR) 2,4-D formulations using the laboratory-derived CET relationships were conducted as summarized by Netherland and Getsinger (1991). These formulations included lignin pellets, acrylic polymers, clay pellets, rubber-based elastomers, all of which were shown to deliver 2,4-D to the water column at relatively constant rates. However, problems associated with scale-up procedures to produce operational quantities of the formulations and/or limitations with maintaining adequate herbicide delivery periods (exposure times) ended further evaluations of these products.

Based on results obtained in the small-scale CET studies described earlier, milfoil control in the field should be a predictable event. Acceptable control of milfoil should be achieved when plants are exposed to a minimum 2,4-D concentration of 0.5 mg ae/L for greater than 72 hr, 1.0 mg ae/L for greater than 36-48 hr, and 2.0 mg ae/L for greater than 24-36 hr. However, actual herbicide exposures in the field, with dissipating concentrations over time, differ from that of static exposures conducted in the laboratory. Plants treated in field applications will be exposed to dissipating concentrations of herbicides over time, versus static exposures used in small-scale CET studies. Also, field concentrations will be influenced by the rate of herbicide application and the type of formulation used. The rate of dissipation and the magnitude of plant exposure will also be influenced by water exchange characteristics and herbicide diffusion, uptake, adsorption, and degradation. Nevertheless, these laboratory-derived CET relationships conform well with 2,4-D field treatments reported in the literature. Green and Westerdahl (1990) showed that calculated dissipation rates resulting from aqueous concentrations of 2,4-D residues in field applications in Lake Seminole, GA, and in lakes of the Okanagan Valley, British Columbia, Canada, verified the CET relationships, with respect to milfoil control. Results from studies conducted in outdoor, hydraulic flumes also verified these 2,4-D dose/response requirements (Turner et al. 1996).

## Fluridone

Initial studies to determine low-dose CET relationships for fluridone (1-methyl-3-phenyl-5-[3-(trifuoromethyl)phenyl-4(1H)-pyridinone) against milfoil and hydrilla were conducted under controlled-environment conditions (Hall 1984; Hall 1985; Hall, Westerdahl, and Stewart 1984; Westerdahl 1986; Westerdahl 1987; Van and Conant 1988). Results from these early studies indicated that fluridone contact time requirements for controlling these target plants were much longer (days to weeks) than exposure times required to control the plant with other aquatic herbicides (hours).

Both milfoil and hydrilla were controlled 75-80 percent when exposed to fluridone levels of 15 to 30 µg/L for 20 to 40 days. Data from these studies were then used to design and evaluate low-dose CR formulations (polycaprolactone fibers and pellets) in the laboratory and the field (Westerdahl, Getsinger, and Hall 1984; Westerdahl 1986; Dunn et al. 1988; Westerdahl, Getsinger, and Green 1988; Netherland and Getsinger 1991). However, although most of these CR formulations delivered near predicted target rates of fluridone, field efficacy performance was inconsistent.

A second series of controlled-environment static water studies were conducted to better define fluridone CET relationships against milfoil and hydrilla (Netherland 1991a; Netherland 1992; Netherland, Getsinger, and Turner 1993; Netherland and Getsinger 1955a). In addition, controlled-environment studies were carried out to determine potential control of these plants under various fluridone half-lives, simulating dissipation scenarios that might occur in the field (Netherland and Getsinger 1995b). Results from these static and half-life evaluations confirmed and refined earlier CET findings and suggested that the threshold of milfoil and hydrilla growth inhibition occurs at fluridone concentrations from 1 to 3 µg/L with exposure periods of greater than 30 days. Initial treatment rates of less than 4 µg/L inhibited growth compared with untreated controls, but did not result in biomass reduction. These studies also showed that fluridone rates of 10 to 15 µg/L (order of magnitude below maximum label rate), for exposure periods of greater than 60 days, were required for

90-95 percent control of both plants, and that exposure time is the most critical factor for optimizing treatment efficacy. However, following high initial treatment rates (>10 µg/L), rates as low as 1 to 2 µg/L completely inhibited regrowth of hydrilla and milfoil.

Large-scale field treatments were conducted, coupling laboratory-derived CET data and site-specific water exchange information, to verify the CET relationships and to confirm their use for improving the control of milfoil and hydrilla. Using this combined strategy, successful treatments were accomplished in the hydrilla-infested portions of the St. Johns and Withlacoochee rivers in Florida (Haller, Fox, and Shilling 1990; Fox, Haller, and Shilling 1994). This technique was also used to control hydrilla in Foster Creek, South Carolina (De Kozlowski 1994), and as part of a hydrilla eradication program in Pipe and Lucerne lakes, Washington (McNabb and Marquez 1996). Similar field-verification studies were conducted in milfoil-infested systems including sections of the Columbia River, Washington (McNabb 1993), and Long and Sacheen lakes, Washington (Farone and McNabb 1993; Getsinger 1993; McNabb 1995).

## Triclopyr

A series of evaluations to determine CET relationships for controlling milfoil using triclopyr (3,5,6-trichloro-2-pyridinyloxyacetic acid) were conducted under controlled-environment conditions (Netherland 1990; Netherland 1991a; Netherland 1992; Netherland and Getsinger 1992; Netherland and Getsinger 1993). Results from these static water studies showed that excellent milfoil control was achieved at CET combinations of 0.25 mg ae/L for 72 hr, 0.5 mg ae/L for 48 hr, 1.0 mg ae/L for 36 hr, 1.5 mg ae/L for 24 hr, and 2.0 and 2.5 mg ae/L for 18 hr. However, treatments of 2.5 mg ae/L for 2 hr, 1.0 mg ae/L for 6 hr, and 0.25 and 0.5 mg ae/L for 12 hr were ineffective, providing only partial control of milfoil.

Data from these laboratory efforts were also used to develop unique CR carriers for triclopyr, including protein and gypsum matrices, which were evaluated in small- and mesocosm-scale systems (Turner et al. 1993; Netherland and Sisneros 1994; Rodgers, Dunn, and Jones 1994; Netherland et al. 1994). Of the two formulations, the gypsum-based carrier proved to be the most consistent in maintaining required triclopyr release rates. Companion studies using the conventional liquid amine formulation of triclopyr verified the laboratory-derived CET principles (Turner et al. 1993; Netherland et al. 1994; Smart et al. 1995; Turner et al. 1996).

Large-scale CET field verification studies were conducted in selected locations around the country. These sites included flowing-water situations on the Columbia and Pend Oreille rivers in Washington (McNabb 1993; Getsinger et al. 1996; Getsinger et al. in press) and more quiescent water conditions in Guntersville Reservoir, Alabama (Turner, Getsinger, and Burns 1996), and Lake Minnetonka, Minnesota (Getsinger 1995; Madsen and Getsinger 1995; Fox and Haller 1995).

## Bensulfuron Methyl

The efficacy of bensulfuron methyl (methyl 2-[[[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl]methyl]benzoate) at various concentration and exposure time combinations was evaluated on hydrilla and milfoil under controlled-environment conditions (Nelson and Van 1991; Nelson and Netherland 1993; Nelson, Netherland, and Getsinger 1993; Nelson et al. 1996). Results of these studies indicated that, similar to CET relationships developed for the herbicide fluridone, exposure periods exceeding 42 days at concentrations greater than 25 µg/L bensulfuron methyl were required to provide control of hydrilla and milfoil. With this systemic herbicide, increasing exposure time was more efficacious than increasing chemical concentration.

Static-exposure studies of bensulfuron methyl CET requirements for controlling milfoil were conducted in outdoor mesocosms at treatment rates ranging from 25 to 100 µg/L for periods of 6 to 12 weeks (Getsinger et al. 1994a). At the long exposure periods, milfoil was adequately controlled, verifying the exposure time principles demonstrated in previous laboratory studies. Field evaluations of bensulfuron methyl on hydrilla and milfoil in Guntersville Reservoir, Alabama (Turner, Getsinger, and Burns 1996), and on hydrilla in Lake Seminole, Georgia (Getsinger et al. 1994b), produced mixed results with respect to efficacy. Poor efficacy observed in some of these treatments may have been caused by inadequate bensulfuron methyl (BSM) exposure periods, due to water exchange processes.

# 3 Contact Herbicides

## Endothall

Evaluations to determine CET relationships for controlling milfoil and hydrilla with the contact herbicide endothall (the dipotassium salt of 7-oxabiocyclo [2,2,1] heptane-2,3-dicarboxylic acid) were conducted under controlled-environment conditions (Van and Conant 1988; Green 1989; Netherland 1990; Netherland 1991a; Netherland, Green, and Getsinger 1991a,b; Netherland, Green, and Getsinger 1991b). Results from these studies showed that milfoil and hydrilla control (biomass knock-down) increased with increasing endothall concentrations and/or exposure times. Lower concentrations and shorter exposures provided initial plant injury, but generally allowed substantial regrowth during the study period. Severe milfoil injury (>85 percent biomass reduction) occurred when plants were exposed to endothall levels of 0.5 mg ae/L for 48 hr, 1.0 mg ae/L for 36 hr, 3.0 mg ae/L for 18 hr, and 5.0 mg ae/L for 12 hr. Severe hydrilla injury occurred when plants were exposed to 2.0 mg ae/L for 48 hr, and 3.0, 4.0, and 5.0 mg ae/L for 24 hr.

Information generated in these laboratory-derived CET studies were used for preliminary evaluations of several new endothall formulations. These included a CR clay pellet (Dunn et al. 1988), a 27 percent ai conventional-release clay pellet (Turner et al. 1993; Turner, Getsinger, and Burns 1996), a gypsum-based slow-release matrix device (Netherland and Sisneros 1994; Netherlandet al. 1994), and a protein matrix and 45 and 63 percent ai superabsorbent polymer (Netherland et al. 1994; Netherland and Turner 1995). None of these prototype formulations progressed beyond the experimental evaluation stage. However, a 65 percent ai superabsorbent polymer formulation of endothall is being field tested and evaluated for USEPA aquatic registration (Fox and Haller 1996).

Endothall CET relationships for controlling hydrilla were verified in large-scale field trials in Lake Washington, Florida (Fox and Haller 1990), and in the Crystal River, Florida (Fox and Haller 1992; Fox, Haller, and Getsinger 1993). In addition, CET relationships for controlling milfoil using endothall were verified in outdoor hydraulic flume studies (Netherland et al. 1994; Netherland and Turner 1995).

## Diquat

Limited evaluations to determine CET relationships for controlling milfoil and hydrilla with the contact herbicide diquat (6,7-dihydrodioyrido [1,2-α:2',1'-c]pyrazinediiumion) were conducted under laboratory and controlled-environment conditions (Westerdahl 1987; Van and Conant 1988; Netherland 1994). Excellent milfoil control was obtained with diquat at treatment rates of 0.25 to 0.5 mg/L for 1- to 12-hr exposure times. A minimum of 6 hr contact to 2.0 mg/L diquat was required to achieve adequate control of hydrilla.

These laboratory-based results should be tempered with some degree of caution. In the field, suspended sediment and other micelles, and particulate material covering plants, are likely to account for an immediate loss of diquat, which is readily bound to negatively charged particles and subsequently inactivated. In contrast, these laboratory experiments minimized any turbidity in the water column, and the plants were generally free of particulate material. This minimal loss of diquat during the exposure periods may underestimate the CET relationship required for controlling submersed plants in field situations.

Information derived from some of the laboratory CET studies was used to develop CR formulations of diquat, including several generations of polycaprolactone fibers (Dunn et al. 1988). Although several versions of this CR fiber system provided rates and durations of diquat release that are required in field treatments, no additional evaluations were conducted. Field verification of laboratory-derived diquat CET requirements were conducted in Orange Lake, Florida (Fox and Haller 1994; Langeland et al. 1994).

## Copper

Pilot studies were conducted in under controlled-environment conditions to evaluate a chelated formulation (ethylenediamine complex) of copper, Komeen, against milfoil (Netherland et al. 1994; Netherland 1994). Milfoil injury symptoms were noted within hours following treatment at rates of 1 and 3 mg/L copper and exposures of 1, 3, and 12 hr; however, plants recovered from all treatments within 1 week following application. Results suggested that milfoil may be somewhat tolerant of Komeen and other copper-based compounds.

# 4 Conclusions and Recommendations

## Conclusions

The basic relationships between herbicide concentration and exposure time required to provide control of milfoil and hydrilla were more thoroughly defined for the chemicals 2,4-D, fluridone, triclopyr, bensulfuron methyl, endothall, diquat, and copper in a series of small-scale, controlled-environment studies. Generally, results showed that improved control of these two target plants can be achieved by coupling appropriate herbicide concentrations with exposure times. With most products that means herbicide concentration must be increased to compensate for short exposure times, but can be decreased if exposure times are lengthened. Depending upon the nature of the herbicide active ingredient, the concentrations and exposure times required for acceptable control range from micrograms/liter to milligrams/liter, and from hours to weeks. Many of the laboratory CET requirements developed in this work unit have been verified in large-scale field applications. When herbicide-specific CET information is coupled with site-specific water-exchange data, guidance can be developed for improving the control of milfoil and hydrilla using the minimum dose of herbicide.

## Recommendations

Based on the results of this work, the following actions are recommended:

*a.* Continue to refine herbicide CET relationships for target plants, and initiate similar studies for nontarget vegetation.

*b.* Use CET information on nontarget plants for developing selective control techniques and for determining effects of off-target movement of herbicides.

*c.* Apply CET relationships and site-specific water-exchange information to develop prescription treatment strategies for selectively managing nuisance plants and restoring desirable native vegetation.

*d.* Develop environmentally compatible controlled-release carriers/ formulations to assist in providing prescription herbicide treatments.

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Efficacy of herbicides used to control submersed aquatic plants depends upon herbicide concentration in the water column and exposure time of herbicide concentrations surrounding target plants. Furthermore, the unique properties of aquatic herbicides require that concentration/exposure time (CET) relationships be developed for each product and target plant. Improved control of exotic species such as Eurasian watermilfoil (*Myriophyllum spicatum*) and hydrilla (*Hydrilla verticillata*), using lower levels of active ingredients, can be achieved through the characterization of herbicide CET relationships.

From 1986 through 1995, studies were conducted under controlled conditions to define CET relationships for the most widely used U.S. Environmental Protection Agency (USEPA)-registered aquatic herbicides that are effective against Eurasian watermilfoil and hydrilla, including 2,4-D, fluridone, endothall, diquat, and copper. The USEPA Experimental Use Permit aquatic herbicides triclopyr and bensulfuron methyl were also evaluated. These research objectives were accomplished through a series of laboratory, growth chamber, greenhouse, mesocosm, and field verification studies.

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Coupling pertinent CET information with water exchange data, prescription treatment scenarios can be developed to achieve desired levels of target plant control, while using low doses of herbicides and minimizing damage to nontarget plants. Results from these CET evaluations have provided guidance for the cost-effective and environmentally compatible use of aquatic herbicides in a variety of field situations, including the development of innovative delivery techniques for flowing water conditions. This report summarizes major research findings and accomplishments of the CET work effort.

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