



**US Army Corps  
of Engineers**  
Waterways Experiment  
Station

Technical Report A-98-2  
July 1998

*Aquatic Plant Control Research Program*

# **Field Evaluation of Low-Dose Metering and Polymer Endothall Applications and Comparison of Fluridone Degradation from Liquid and Slow-Release Pellet Applications**

*by Michael D. Netherland, WES*

*David Sisneros, U.S. Bureau of Reclamation*

*Alison M. Fox, William T. Haller, University of Florida*

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by Michael D. Netherland

U.S. Army Corps of Engineers  
Waterways Experiment Station  
3909 Halls Ferry Road  
Vicksburg, MS 39180-6199

David Sisneros

U.S. Bureau of Reclamation  
Denver Federal Center  
6th and Kipling, Building 56  
Denver, CO 80225

Alison M. Fox, William T. Haller

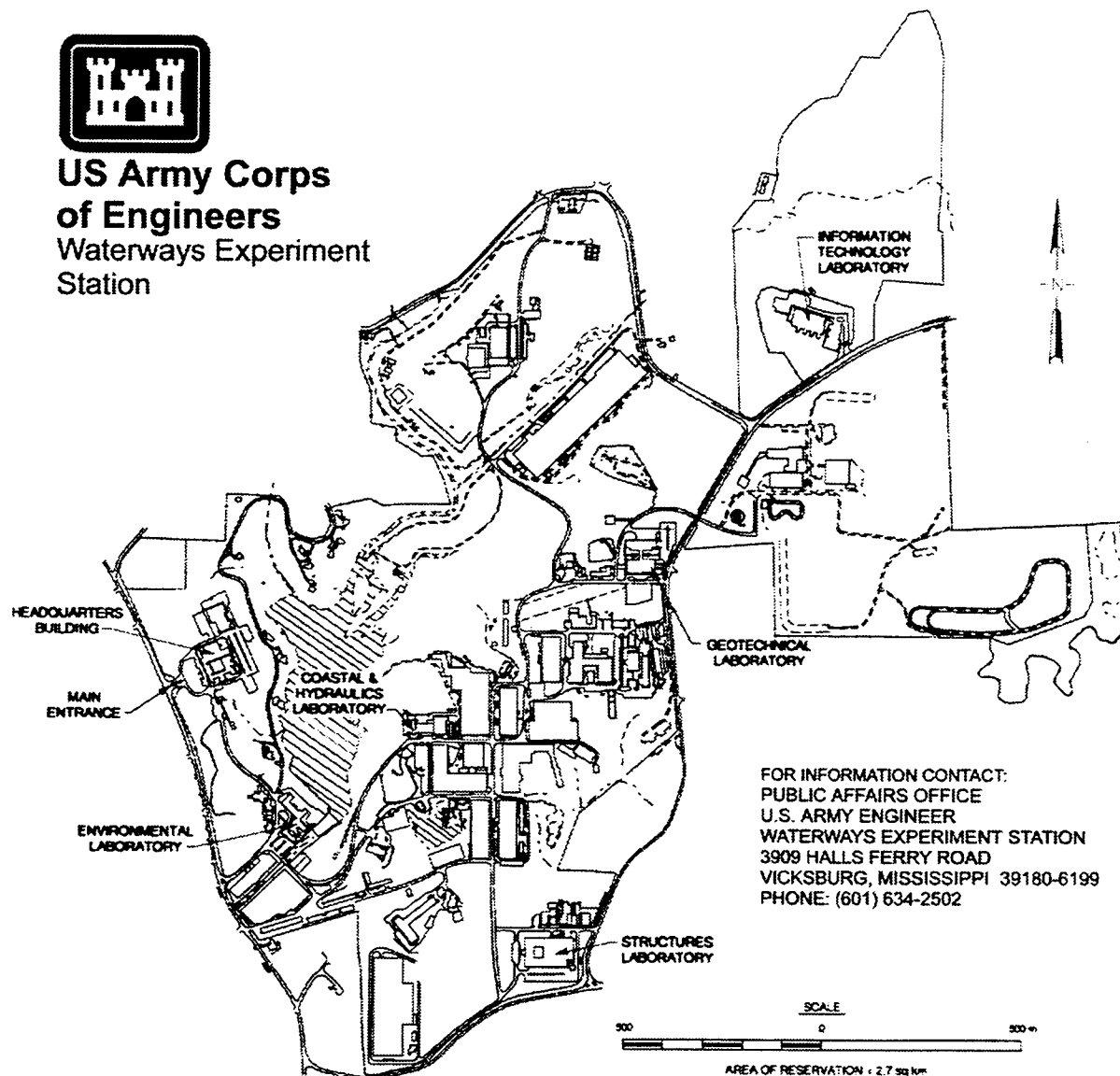
University of Florida  
Center for Aquatic Plants  
7922 NW 71st Street  
Gainesville, FL 32653

**Final report**

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Station



FOR INFORMATION CONTACT:  
PUBLIC AFFAIRS OFFICE  
U.S. ARMY ENGINEER  
WATERWAYS EXPERIMENT STATION  
3909 HALLS FERRY ROAD  
VICKSBURG, MISSISSIPPI 39180-6199  
PHONE: (601) 634-2502

**Waterways Experiment Station Cataloging-in-Publication Data**

Field evaluation of low-dose metering and polymer endothall applications and comparison of fluridone degradation from liquid and slow-release pellet applications / by Michael D. Netherland ... [et al.] ; prepared for U.S. Army Corps of Engineers.

64 p. : ill. ; 28 cm. — (Technical report ; A-98-2)

Includes bibliographic references.

1. Aquatic plants — Control — Environmental aspects. 2. Aquatic herbicides — Testing. 3. Hydrilla — Testing. 4. Sago pondweed — Testing. I. Netherland, Michael D. II. United States. Army. Corps of Engineers. III. U.S. Army Engineer Waterways Experiment Station. IV. Aquatic Plant Control Research Program (U.S. Army Engineer Waterways Experiment Station) V. Series: Technical report (U.S. Army Engineer Waterways Experiment Station) ; A-98-2.

TA7 W34 no.A-98-2

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# Preface

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The work reported herein was conducted as part of the Aquatic Plant Control Research Program (APCRP), Work Unit 32437. 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 the Department of the Army Appropriation Number 96X3122, Construction General. In addition, funding for the chemical metering studies was provided by the Bureau of Reclamation's Water Research Program: Project EE007, Improved Aquatic Site Pest Management Methods Program and Reclamation's Tech Maturation Funds. The APCRP is managed under the Center for Aquatic Plant Research and Technology (CAPRT), Dr. John W. Barko, Director. Mr. Robert C. Gunkel, Jr., was Assistant Director for the CAPRT. Program Monitors during this study were Mr. Timothy Toplisek and Ms. Cheryl Smith, HQUSACE.

Principal Investigator for this work unit was Mr. Michael D. Netherland, Ecosystems Processes and Effects Branch, Environmental Processes and Effects Division (EPED), EL, WES. The Principal Investigator for the chemical metering studies was Mr. David Sisneros, Bureau of Reclamation's Ecological Research and Investigation Work Group, Bureau of Reclamation, Denver, CO. The report was reviewed by Drs. Kurt Getsinger and Susan Sprecher, EPED. Messrs. Netherland and Sisneros prepared this report along with Drs. Alison M. Fox and William T. Haller of the University of Florida.

For work conducted in the western canals, the authors would like to thank Mr. E. G. Turner of the AScI Corporation, Mr. Fred Amator of Elf Atochem, Mr. Pat McGordy of the Idaho Water Users Association, and Mr. Mark Lichtwardt and Ms. Tracie Greene of Reclamation for technical assistance.

The polymer study conducted in Lake Weohyapapka, Florida, was carried out with the much appreciated cooperation of Messrs. Bill Moore and Chet DeSavigny, Elf Atochem, Mr. Glenn Turner, AScI, Mses. Margaret Glenn and Jan Miller, University of Florida, and personnel from the South Florida Water Management District and Polk County, Florida, Environmental Operations. In addition, the cooperation of Elf Atochem, Inc., for providing herbicide and analytical services is greatly appreciated.

The fluridone formulation study conducted at the Austin Cary Forest near Gainesville, FL, was conducted with the cooperation of Ms. Glenn, University of Florida, and Mr. David Honnell, AScI. In addition, the cooperation of the SePRO for providing product and analytical services by Ms. Tracy Brenton and Alicia Staddon is greatly appreciated.

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

At the time of publication of this report, Director of WES was Dr. Robert W. Whalin. Commander was COL Robin R. Cababa, EN.

This report should be cited as follows:

Netherland, M. D., Sisneros, D., Fox, A. M., and Haller, W. T. (1998). "Field evaluation of low-dose metering and polymer endothall applications and comparison of fluridone degradation from liquid and slow-release pellet applications," Technical Report A-98-2, U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS.

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# 1 Introduction

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Inconsistent plant control following the use of aquatic herbicides is often attributed to high variability in dispersion rates following application. Recognition of this problem has stimulated research into various application methods that maintain effective herbicide concentrations in the target treatment area over time. Beginning in the early 1970s and progressing through the mid-1980s, research in this area focused on the development of controlled-release (CR) carriers to deliver a defined dose of herbicide for a specified period of time. Early development and field trials using CR carriers in aquatic systems are reviewed by Riggle and Penner (1990). Recent research on CR carriers conducted under the U.S. Army Corps of Engineers Aquatic Plant Control Research Program (APCRP) in the late 1980s and early 1990s is reviewed by Netherland et al. (1994). Conclusions drawn from much of the early work with CR carriers were ambiguous, and research progress on suitable products was hampered by production problems, inconsistent quality control, field application difficulties, performance deficiencies such as buoyancy (off-target movement) or sinking (bound in soft sediments), and inconsistent herbicide-release profiles.

In addition to the problems noted above, early efforts in development of CR technology were also hindered by a lack of understanding of the important role of water exchange and dose-response relationships for individual herbicides and target plant species. Research conducted in the late 1980s through the early 1990s demonstrated the high level of variability in dispersion rates that exists once a chemical is applied to water. The use of the inert tracer dye rhodamine WT was instrumental in proving that residues often move rapidly from a treatment site, even in waters that seem to be quiescent (Fox et al. 1991; Getsinger, Green, and Westerdahl 1990; Sisneros 1992; Fox and Haller 1994; Getsinger, Sisneros, and Turner 1993; Getsinger 1995; and Getsinger et al. 1997). This field application technique work was complimented by other efforts on herbicide concentration and exposure time (CET) relationships that used a laboratory system to predict levels of control achieved following exposures of target plants to various herbicide doses (Van and Conant 1988; Green and Westerdahl 1990; Netherland, Green, and Getsinger 1991; Netherland and Getsinger 1992; Netherland, Getsinger, and Turner 1993; Netherland and Getsinger 1995a,b). These efforts showed that while many factors can affect herbicide performance (e.g., temperature, water quality, plant density, application

rate), the rapid dispersion of herbicide residues is the chief cause of failure to control plants in the field.

The ability to integrate information from the application techniques and CET research efforts was verified by the successful herbicide treatment of Eurasian watermilfoil (*Myriophyllum spicatum* L.) on the Pend Oreille River, Washington (Getsinger et al. 1997). Rhodamine WT (RWT) dye studies were used to estimate water exchange half-lives in selected treatment plots, and based on this information, laboratory CET results were used to select application rates of the herbicide triclopyr (3,5,6-trichloro-2-pyridinyloxyacetic acid) to be used in areas of rapid dispersion and at quiescent sites to provide control of Eurasian watermilfoil. Although the APCRP Application Techniques and CET work units have ended, information generated from these lines of research remains a critical component of the current APCRP Herbicide Delivery Systems (HDS) work unit. Proper development of the HDS work unit requires the use of existing data and the generation of new data to provide information on water exchange characteristics at a particular site or a CET relationship for a selected herbicide and target plant species.

Research conducted in hydraulic flumes using water exchange and CET information demonstrated that the concept of maintaining low doses of herbicide over time via CR matrices (often only 12 to 96 hr) was valid (Netherland et al. 1994; Turner et al. 1995). Yet it was apparent from this work that developing a CR matrix to provide target concentrations under highly variable and changing water exchange conditions found in the field would be a complex task. Alternatively, the utility of a CR matrix or metering pump to deliver defined doses of a herbicide such as endothall (7-oxabicyclo[2.2.1]heptane-2,3-dicarboxylic acid) over short periods of time in linear flow systems (flood control or irrigation canals, rivers) was considered to have good potential if water exchange processes were well defined.

Based on the results obtained to date, emphasis of the HDS work unit has shifted away from exclusive development of CR carriers and has broadened to include several separate but related lines of research. While development of CR technology has not been abandoned, research efforts have focused on accommodating new products with potential for U.S. Environmental Protection Agency (EPA) registration, new treatment strategies for control of plants in high-flow environments, and improved understanding of release properties and residue distribution of EPA-labeled granular formulations.

As the scope of the research has expanded, individual projects are not as closely linked together compared with past efforts that focused on CR matrix development. This report will update three separate projects that have been conducted under the HDS work unit: (a) Metered Application of Low-Dose Endothall for Controlling Sago Pondweed in High-Flow Environments, (b) Evaluation of a New Granular Endothall Formulation for Submersed Plant Control, and (c) Comparison of Fluridone Dissipation Following Treatment with Liquid and Slow-Release Pellets. Each of these research thrusts will include its own background, results, discussion, conclusions, and recommendations. This

broadened research scope continues to complement the overall objectives of the HDS work unit, which are to identify and evaluate herbicide formulations, release characteristics and efficacy of novel carrier or application systems, and formulations to improve handling and applicator safety.

## **2 Metered Application of Low-Dose Endothall for Controlling Sago Pondweed in High-Flow Environments**

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### **Background: Idaho Study 1**

Previous experience with herbicide application has shown that in high-flow environments, traditional slug or surface application techniques may not work, due to rapidly dissipating herbicide concentrations and a concomitant lack of exposure period (Hansen, Oliver, and Otto 1983). Therefore, one objective of the HDS work unit is to identify and evaluate novel application techniques for improved plant control in areas of rapid water exchange. Recent research emphasis has explored the potential of providing low concentrations of herbicide over a sustained period of time to improve plant control. Evaluations conducted in large hydraulic flumes (4 by 110 by 1.2 m) with the herbicides triclopyr and endothall (7-oxabicyclo[2.2.1]heptane-2,3-dicarboxylic acid) incorporated into a gypsum CR matrix or applied with metering pumps showed that target concentrations were maintained while providing excellent plant control using both techniques (Netherland et al 1994; Turner et al. 1995).

Several factors were considered prior to field testing the low-dose extended exposure treatments using CR matrices, including problems with quality control, uncertainties in performance characteristics under highly variable field conditions, and potential difficulties in product application. In addition, support by industrial cooperators is critical at the field-testing stage, and further development of the gypsum CR matrix (or any new CR formulation) will require a substantial investment of resources from the private sector. The costs and time required to develop a CR formulation for the aquatic market (estimated at 3-5 years) discouraged continued field evaluation of the gypsum CR matrix.

In contrast, metering technology has been used in aquatic systems for many years to apply products such as copper, fluridone (1-methyl-3-phenyl-5-[3-(trifluoromethyl)phenyl]-4(1*H*)-pyridinone), and endothall for algae and macrophyte control. While metering technology is not new, the delivery of low target doses of herbicide over an extended time (based on laboratory and mesocosm research) represents a novel approach to treatment of high-flow environments. Canals in the Pacific Northwest were chosen by Bureau of Reclamation (Reclamation) personnel for field testing, as these systems provide a good field evaluation of the low-dose extended exposure period concept. These high-flow canals provide water for agricultural, industrial, municipal, and recreational uses, and they often become infested with aquatic macrophytes, resulting in reduced delivery of water to end users. The submersed species sago pondweed (*Potamogeton pectinatus* L.) is particularly troublesome in many of these canals, and Reclamation personnel are evaluating new application strategies to control this plant. Yeo (1965) provides a good description of the biology and ecology of sago pondweed as well as control measures that have been used in the northern latitudes of the United States.

Due to the high-flow environment in the canals, the use of products such as acrolein (2-propenal) and xylene for control of sago pondweed has been a standard treatment for many years (Anderson 1990). These contact biocides require exceedingly low exposure periods, have no irrigation restrictions, do not persist in the environment, and result in excellent short-term plant control. However, Reclamation water management practices in many areas have changed, resulting in fish and wildlife populations becoming more prevalent in the canals and discharge sites. As issues related to nontarget toxicity increase, alternatives to acrolein and xylene will be needed for continued control of aquatic plants. Researchers at Reclamation and the U.S. Army Engineer Waterways Experiment Station determined that evaluation of the dipotassium salt of endothall (Aquathol K) using the low-dose extended exposure concept was a viable alternative, especially where fish and wildlife were of concern.

The dipotassium salt of endothall is generally nontoxic to fish and wildlife even at the maximum allowable concentration of 5 mg/L (Elf Atochem 1992). However, surface applications of high concentrations of endothall as Aquathol K are often ineffective due to high flows and subsequent short exposure times. The lack of volatility of endothall may allow more canal area to be treated per injection site compared with acrolein and xylene, which tend to lose efficacy a relatively short distance from the application site. In addition, the ability to apply endothall at rates near the acceptable residue level in drinking water (ARLDW) of 0.2 mg/L greatly reduces potential problems with discharge of treated waters into potable water supplies.

Hydraulic flume studies suggested that Aquathol K could be used in a high-flow environment at rates as low as 0.5 mg/L for 72 to 96 hr to provide good control of Eurasian watermilfoil and southern naiad (*Najas guadalupensis* (Sprengel) Magnus) (Turner et al. 1995). A subsequent laboratory study was conducted to determine the dose response of sago pondweed to low rates of endothall applied as Aquathol K. Results indicated that rates between 0.25 and

0.5 mg/L could provide good control of sago pondweed if an adequate exposure period of 48 to 96 hr was maintained (Figure 1). Laboratory CET data were used to determine target treatment rates and exposures for field studies.

In addition to the dipotassium salt of endothall, a dimethylalkylamine salt of endothall (Hydrothol 191) is also used for macrophyte and algae control. It should be noted that operational applications using 0.2 mg/L of Hydrothol 191 for up to 120 hr are currently being used in Arizona waterways to control filamentous algae where the treated water is subsequently discharged into potable water supplies. Although Hydrothol 191 can provide excellent macrophyte control at much shorter exposure periods than Aquathol K, it has a much greater potential for toxicity to fish and wildlife at comparable use rates to the Aquathol K formulation (Elf Atochem 1992). However, reduced exposure requirements, potential for reducing use rates and subsequent nontarget toxicity, and the ability to control algae as well as macrophytes warrant future evaluation of the low-dose/extended exposure concept for Hydrothol 191.

Field studies with Aquathol K were conducted to address the following objectives: (a) determine the feasibility and identify problems associated with applying the low-dose extended exposure treatment strategy to high-flow environments, (b) determine efficacy on sago pondweed, and (c) determine if the ARLDW of 0.2 mg/L endothall would be exceeded in discharge water following the treatment.

## Materials and methods

A field site was chosen at the Minodoka Project on land operated and maintained by the Northside Canal Company at Jerome, ID, in Reclamation's Pacific Northwest Region. The S19 Canal located approximately 40 km west of Twin Falls, ID (Figure 2), is an earthen-lined channel 5 m wide and 1 m deep containing an infestation of sago pondweed. Flow rates in the canal ranged from 336 to 800 L/second (12 to 28 cu ft/second) and consisted of return flow water (wastewater) from agricultural irrigation lands. Treated water entered a 1.25-ha augmentation pond, was diverted through a canal, and was then used for power production prior to entering the Snake River.

Five plant biomass sampling sites were located within the 4.8-km study area to determine efficacy of the herbicide treatment. Each sampling plot was 30 m long, and six random biomass samples were collected in each plot using a 0.25-m<sup>2</sup> frame. An untreated control site was located immediately upstream from the application site. Shoot biomass was collected prior to treatment and at 21- and 42-day posttreatment. Sago pondweed was dominant with small amounts of water plantain (*Alisma* sp. L.) interspersed. Samples were sorted according to species and dried to a constant weight to yield biomass per square meter. Biomass data were subjected to analysis of variance (ANOVA), and differences between mean biomass values of the untreated control site and four treated sample sites were determined by Dunnet's test ( $\alpha = 0.05$ ).



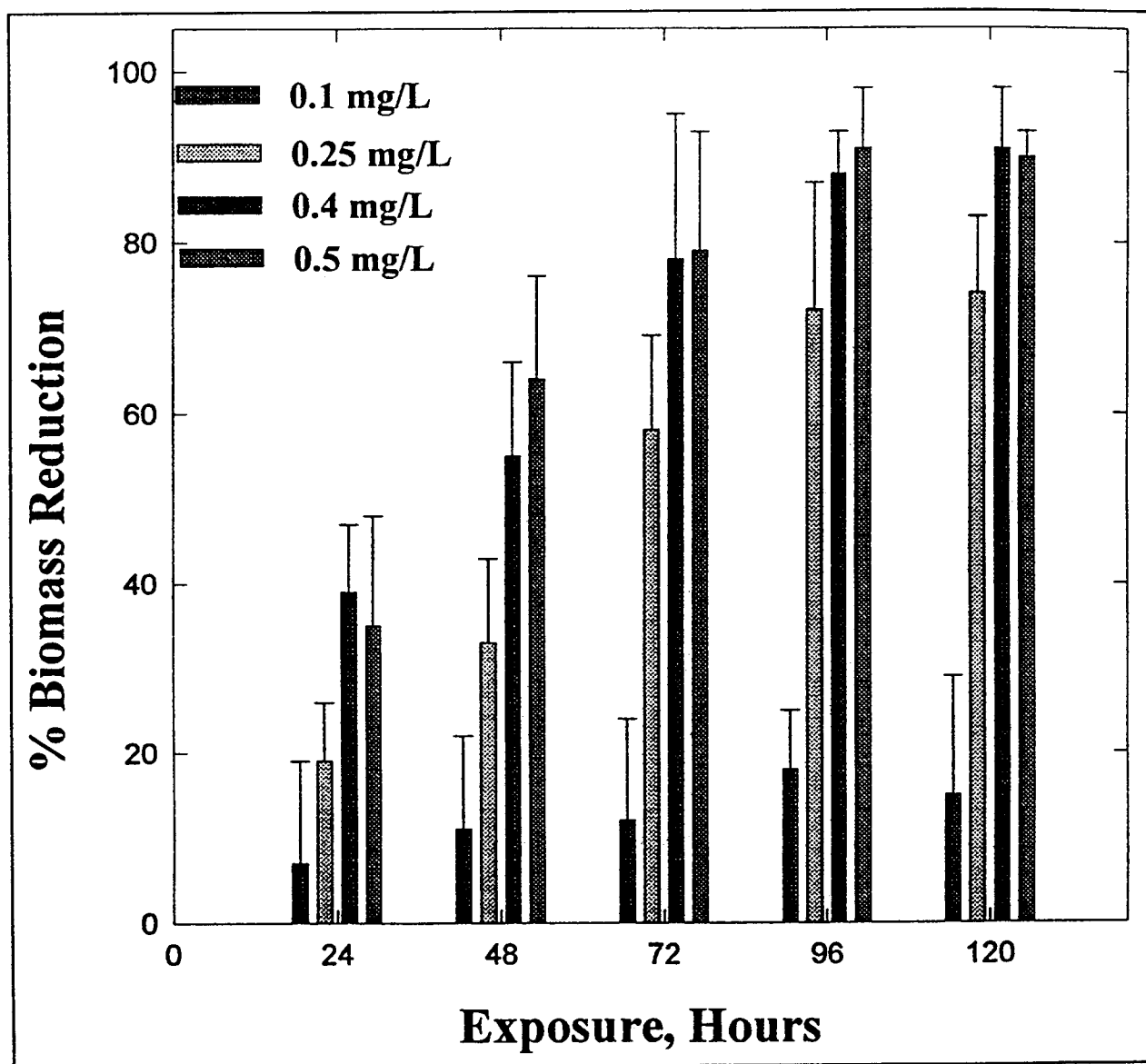


Figure 1. Percent sago pondweed biomass reduction at 35-day posttreatment compared with untreated plants at various endothall concentrations and exposure times in the laboratory (each bar represents the average of three replicate aquaria ( $\pm 1$  SD))

Four water residue sampling sites were established at 0.4, 1.1, 2.2, and 3.6 km from the endothall injection site (Figure 2). In addition, a sampling site was established downstream of the retention pond near the Outlet Lemon's Power Plant to determine if residues were present in the outflow water entering the Snake River. Duplicate water samples were collected from each of the eight sampling sites at 0-, 12-, 24-, 36-, 48-, 60-, 72-, 84-, and 90-hr posttreatment. Water samples were placed on ice and then frozen prior to gas chromatography/mass spectrum (GC-MS) analysis.

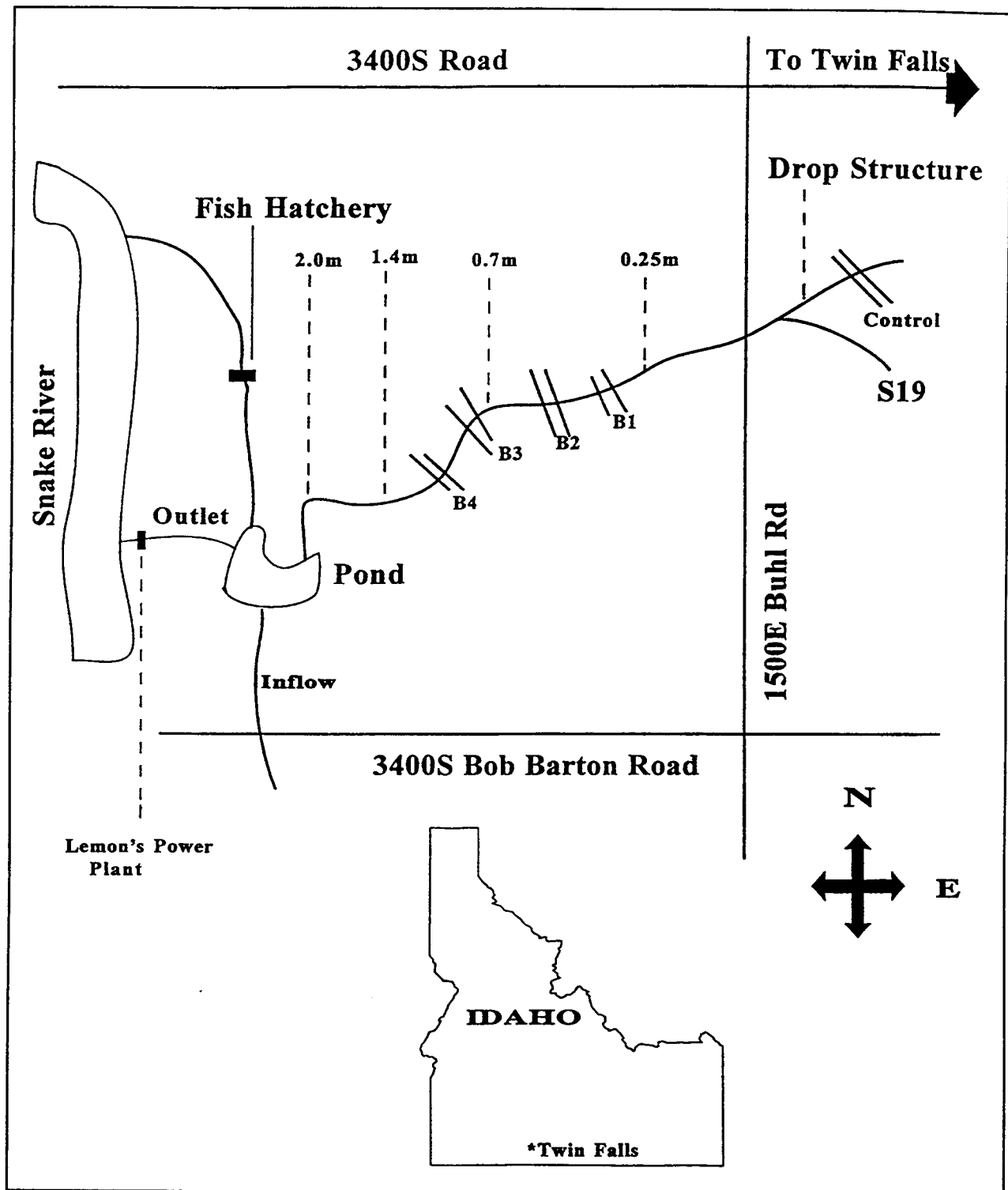


Figure 2. Schematic diagram of the S19 Canal in Idaho depicting locations of endothall water sampling stations and biomass sampling sites

The dipotassium salt of endothall (Aquathol K) was applied on August 14, 1995, at a target application rate of 0.4 mg/L endothall (8 percent of the maximum label rate of 5.0 mg/L) for 96 hr to the S19 Canal, using an electric metering pump manufactured by Liquid Metronics Division of Milton Roy. Metering pumps were initially adjusted to ambient flow rates measured at gauging stations. The inert fluorescent dye RWT was applied concurrently at a rate of 4 µg/L to provide a real-time in situ estimate of endothall concentrations and distribution in the canals. Dye concentrations were measured with a Turner Designs field fluorometer. Previous field work has shown a good short-term correlation between RWT dye and endothall following surface water applications (Fox, Haller, and Shilling 1991).

## Results and discussion

A persistent drought and a significant irrigation demand made it difficult for the North Side Canal Company to maintain constant water flow in the study canal. Consequently, pumps had to be manually recalibrated several times during the study in an effort to maintain target endothall concentrations. Pumps were recalibrated based on measured flow rates and RWT concentrations in the water. Residue data at the 0.4-, 1.1-, 2.2-, and 3.6-km sampling sites showed that endothall concentrations were slightly below the target rate of 0.4 mg/L through 24 hr, just above or at the target rate between 36 and 48 hr, and well below the target rate by 72 hr (Figure 3). Due to logistical and mechanical problems, pumps were shut off at 72 hr, and endothall residues declined to near 0 mg/L by the 90-hr sample. This prevented the full target exposure period of 96 hr from being realized. Residues at the outlet power plant peaked at 0.09 mg/L at 48 hr and never exceeded the ARLDW of 0.2 mg/L.

The unstable flow rates in the canals and the fact that herbicide delivery rates had to be adjusted manually accounted for much of the residue variability observed. A lag time between measuring changing flow rates and RWT values and adjusting the pumps existed. In addition, it was not practical to manually measure flow rates and adjust pumps around the clock. Initial results of this study suggested that this application technique may have limitations in systems subject to substantial fluctuations in flow. Therefore, it was determined that the labor intensiveness as well as problems with the lag time response to fluctuations in flow rates would require improvement in the delivery system prior to any further work.

Sago pondweed in the treated plots generally lost buoyancy within 7 days after treatment (DAT) and remained near the bottom of the canal through 41 DAT. In contrast, plants harvested from the untreated plot grew to the surface and formed a thick canopy. Moreover, while untreated plants remained firmly rooted in the sediment, plants in treated plots were easily removed from the sediments. Although the plants from the treated plots were generally flaccid and discolored compared with untreated plants, the distinction between living biomass with a capacity to recover and plants with no recuperative ability was difficult.

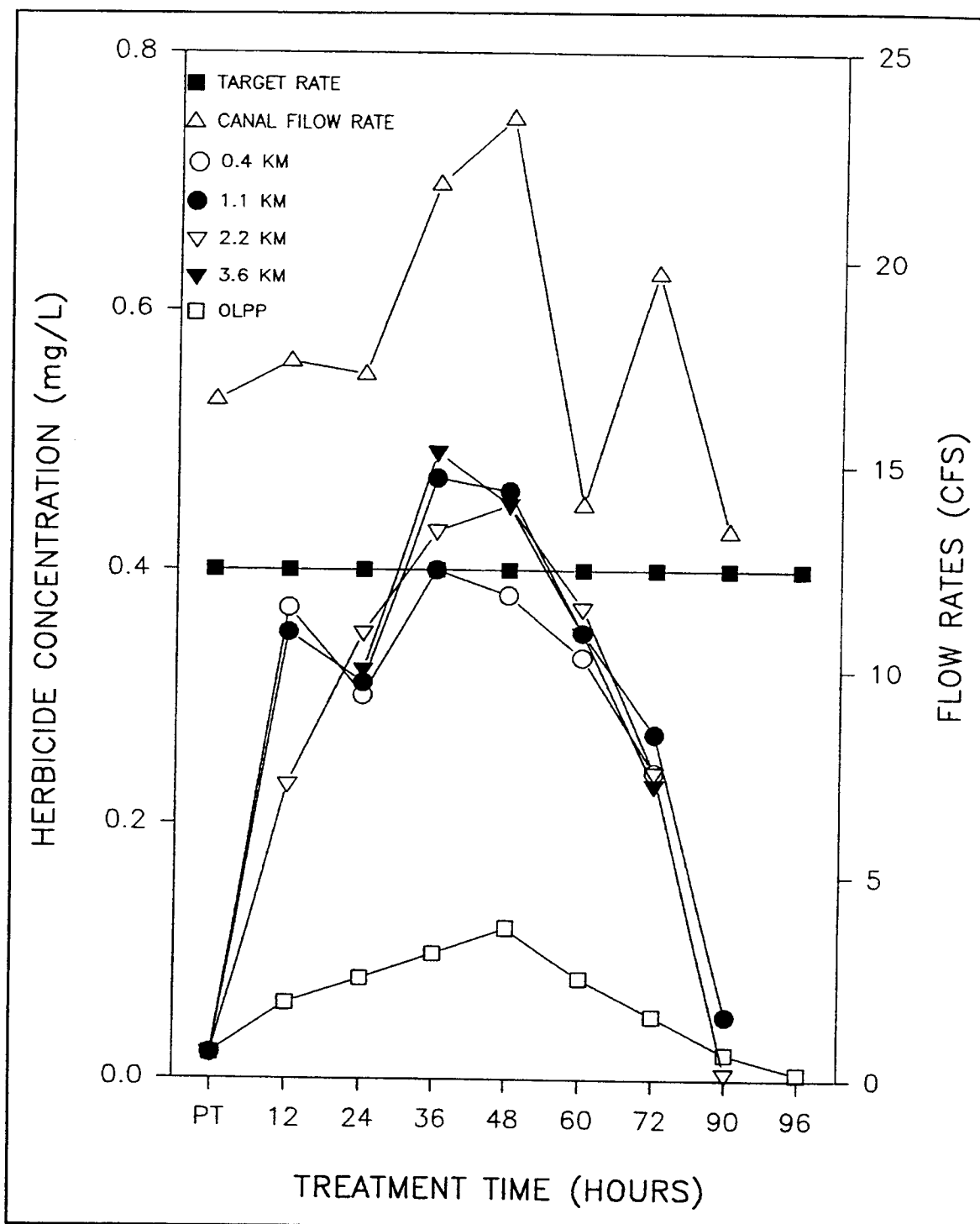


Figure 3. Flow rates measured in the S19 Canal, Idaho, and aqueous endothall residues at an untreated control site and five downstream sampling sites following metered delivery of endothall at a target rate of 0.4 mg/L for 72 hr (each value represents the average of duplicate samples)

Therefore, during the harvests, plant material with any integrity was recognized as biomass.

Results indicated that all plots including the untreated control experienced biomass reductions of 30 to 50 percent by 21 DAT (Figure 4). This reduction was likely tied to cooler weather and shorter days toward the end of the growing season. Despite the reduction in biomass, plants in untreated control sites remained at the water surface and impeded water flow, whereas, treated plants lay prostrate on the bottom of the canal and did not present a significant obstacle to water flow. Although sago pondweed no longer formed a surface canopy in the endothall-treated areas, no significant differences in biomass were noted between the treatment plots at 21 DAT. However, by 41 DAT significant biomass reductions were noted in three of the four treated plots compared with the untreated site (Figure 4). In all treated plots, no evidence of sago pondweed recovery was noted throughout the 41-DAT assessment.

Although biomass differences between treated and untreated plots were not initially dramatic, visual differences between endothall-treated plants laying prostrate on the sediment at 7 DAT and untreated plants forming a thick mat at the water surface were distinct; consequently field project managers were pleased with the results of the treatment.

In addition to reducing the problem of sago pondweed impeding water flow, the following objectives were met: (a) the low-dose extended exposure application technique proved feasible, but will require substantial improvement for operational utility, (b) the ability of the treatments to remove sago pondweed from the water surface within 7 DAT was considered a success by operational personnel, and (c) the ARLDW of endothall (0.2 mg/L) was not exceeded in the discharge water. In addition, endothall was applied at concentrations that were highly unlikely to impact nontarget organisms.

## **Background: Idaho Study 2 and Colorado Study**

Based on results of the first study, additional field trials were conducted to address technical and mechanical problems that were encountered due to variable water flow experienced in the previously described canal evaluation. Fluctuating flows and the lag time associated with manually adjusting metering equipment prevented the target concentration of 0.4 mg/L endothall from being maintained for the desired 96-hr exposure period (Sisneros and Turner 1995). Following problems encountered in the 1995 study, scientists at Reclamation developed an automated delivery system that responds in real-time to changes in water flow by adjusting the amount of chemical applied to the water. This device allows constant aqueous herbicide concentrations to be maintained throughout the exposure period regardless of fluctuating flow rates. The metering system was designed to be used at remote sites, is readily portable, and can be operated using solar power. This delivery system may have many uses for chemical application to aquatic systems and is currently patent-pending.

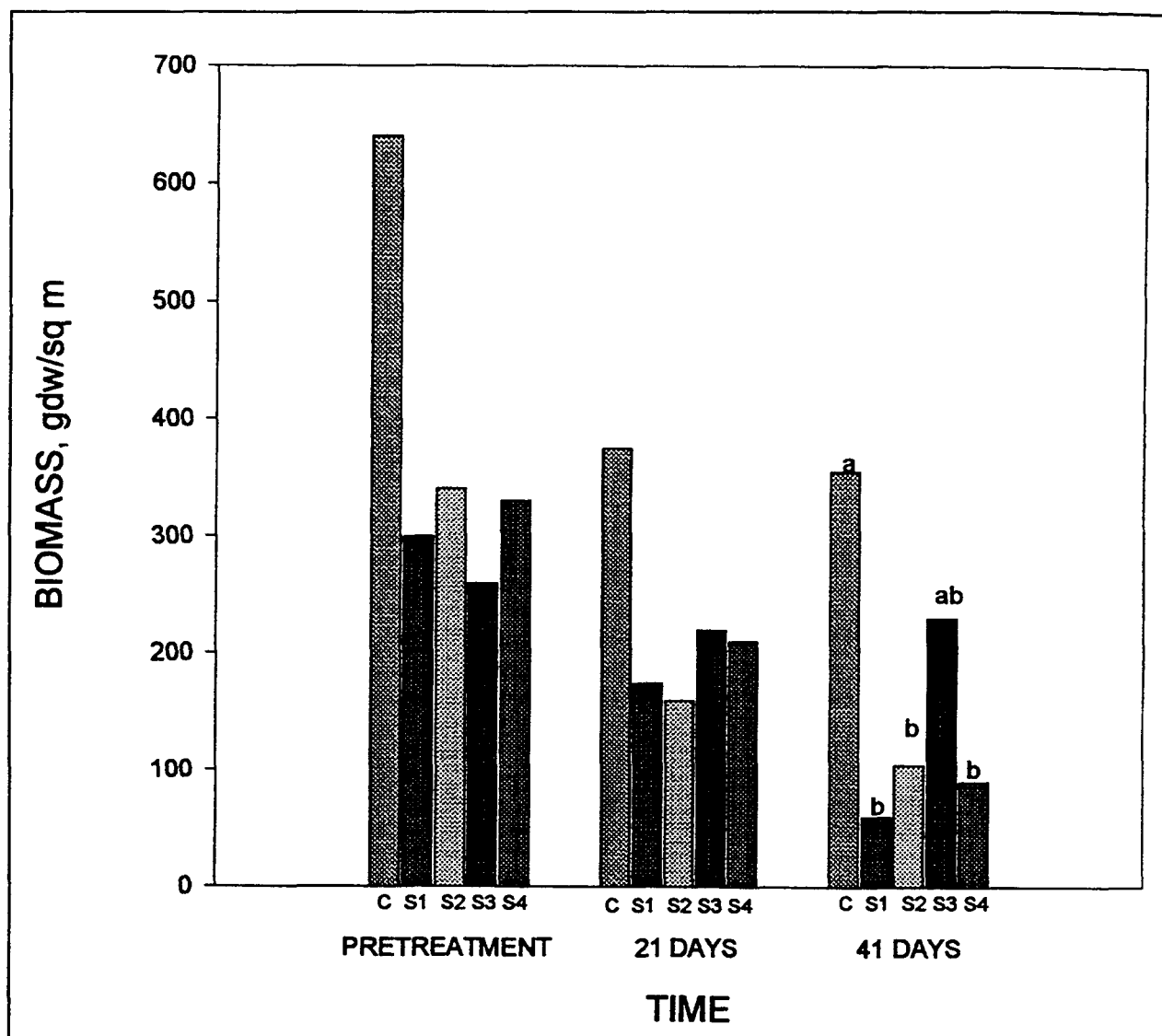


Figure 4. Sago pondweed dry weight biomass in the S19 Canal, Idaho, at 0-, 21-, and 42-day posttreatment at the untreated control site and four downstream sampling sites that were exposed to endothall treatment (each bar represents the average of six biomass samples. Bars with different letters above them represent a significant difference between untreated controls and treatment sites at each sampling time according to Dunnet's test ( $\alpha = 0.05$ ))

During the summer of 1996, low-dose concentration exposure metering studies were conducted in Idaho and Colorado to evaluate the newly developed automated metering system and to validate efficacy of applying low concentrations (8 percent of the maximum allowed label rate of 5.0 mg/L) of the herbicide endothall as Aquathol K to control sago pondweed in high-flow environments.

## Materials and methods

**Idaho site.** The study site was located in Reclamation's Pacific Northwest Region, in the Minodoka Project on the S19 Canal as described for the previous study. At the selected study site, the canal was approximately 4 to 5 m wide and 1 m deep. Flows in the system during the study ranged from 160 to 340 L/sec (5 to 12 cu ft/sec) with linear velocities of approximately 0.5 m/sec. The length of canal treated was approximately 3.2 km with subsequent mixing downstream of the treated section with another canal called the W9, of approximately the same flow. The combined flows from both canals traveled approximately 2 km to a pond of 1.3 ha where additional flows from another canal further diluted any treated water prior to discharge into the Snake River, approximately 2.4 km west of the pond.

Five plant biomass sampling sites were located within the treated study area to determine efficacy. Each biomass site was 30.5 m long, with the untreated control biomass sampling site located 200 m above the application point. The five treated biomass sampling sites were established dependent upon plant density (visual observations) between the application site and sampling Site 5, which was a distance of 1.17 km downstream.

Nine random shoot biomass samples were taken from each of the sampling sites using a 0.25-m square frame at 0 and 28 DAT. The major aquatic plant species was sago pondweed, with small amounts of water plantain also present. Plant samples were sorted to species and dried to a constant weight to determine the dry weight/square meter. Shoot biomass data from each sample site were subjected to ANOVA and means compared with the untreated controls using Dunnett's test at  $\alpha = 0.05$ . In addition, t-tests ( $\alpha = 0.05$ ) were used to compare pretreatment biomass with biomass at 28 DAT within each sampling site.

Five water sampling sites were established along the length of the target treatment area to determine endothall residues downstream of the application site. Untreated water samples were taken immediately upstream of the application site. Duplicate water samples were collected from each sampling site at 0-, 7-, 12-, 28-, 36-, 48-, 60-, 72-, and 84-hr posttreatment. Water samples were collected at middepth, placed on ice, and then frozen for storage prior to analysis by GC/MS.

The dipotassium salt of endothall as Aquathol K was applied to the S19 Canal on August 6-10, 1996, using an automated delivery system. Based on laboratory CET information, the application rate of endothall was targeted at 0.4 mg/L for 84 hr.

**Colorado site.** A field site was also selected in Reclamation's Great Plains region on land operated and maintained by the Farmers Independent Ditch Company (FIDCO), Gilcrest, CO. The FIDCO Canal is earthenlined and approximately 3.1 m wide and 0.7 m deep. Flows measured at check structures and gauging stations ranged from 74 to 327 L/sec (2.22 cfs to 9.81 cfs) with linear velocities of 0.5 m/sec. The length of the canal that was treated was

approximately 5.3 km, and this water was subsequently diverted to a 1.3-ha augmentation pond for recharging groundwater during the study.

Five biomass sampling sites (30.5 m long) were located within the 5.3-km area to determine endothall's efficacy on sago pondweed. In addition, one untreated control biomass sampling site was located upstream of the application site. Biomass sampling sites were selected in areas with high densities of plants (visual observations) between the application site and 5.3 km downstream.

Nine random shoot biomass samples were taken from each of the biomass sampling sites using a 0.25-m square frame prior to treatment and at 17 DAT. Biomass samples were then sorted to species and dried at 70 °C for 48 hr to a constant weight. The canal was dominated by sago pondweed. Shoot biomass data from each sample site were subjected to ANOVA and means compared with the untreated controls using Dunnett's test at  $\alpha = 0.05$ . In addition, t-tests ( $\alpha = 0.05$ ) were used to compare pretreatment biomass to biomass at 17 DAT within each sampling site.

Six water sampling sites were established for endothall residues at 0.8, 1.6, 2.4, 4.0, and 5.3 km downstream of the herbicide application site. Water samples from untreated control sites were taken upstream of the herbicide application site. Duplicate water samples were collected from each of the six water sampling sites at pretreatment, 6-, 12-, 24-, 36-, 48-, 55-, 72-, 84-, and 96-hr posttreatment. Water samples were collected from middepth and frozen as soon as possible to reduce herbicide degradation prior to analysis.

Endothall applied as Aquathol K was metered in to the FIDCO Canal from September 23, 1996, to September 28, 1996, at a target rate of 0.4 mg/L for 96 hr using the automated delivery system described above.

## Results and discussion

**Idaho site.** Residue analyses indicated that the projected target rate of 0.4 mg/L (endothall) for 84 hr was closely approximated (Figure 5). Endothall residues for the treatment sites in the canal were approximately 0.30 mg/L or greater and were maintained for 84 continuous hr. Concentrations of approximately 0.40 mg/L were noted at 7-, 24-, and 72-hr posttreatment, while residues of 0.30 or greater were noted at the 12-, 36-, 48-, 60-, and 84-hr posttreatment sampling. Some loss of endothall is to be expected due to degradation, sorption, and uptake, and the maintenance of rates near the target concentration was considered successful. As metering was discontinued, residues in the treatment sites fell to near 0 mg/L by 96-hr posttreatment. Concentration of less than the ARLDW value of 0.2 mg/L were noted in Lemon Power Plant pond water, which eventually flows into the Snake River. Residues in the untreated control were near 0 mg/L throughout the sample period.

Following endothall treatment, sago pondweed and water plantain were significantly reduced by the 28-day posttreatment sampling (Figure 6). Pretreatment biomass ranged from 30 to 150 g dry weight/m<sup>2</sup>, whereas at 28 DAT



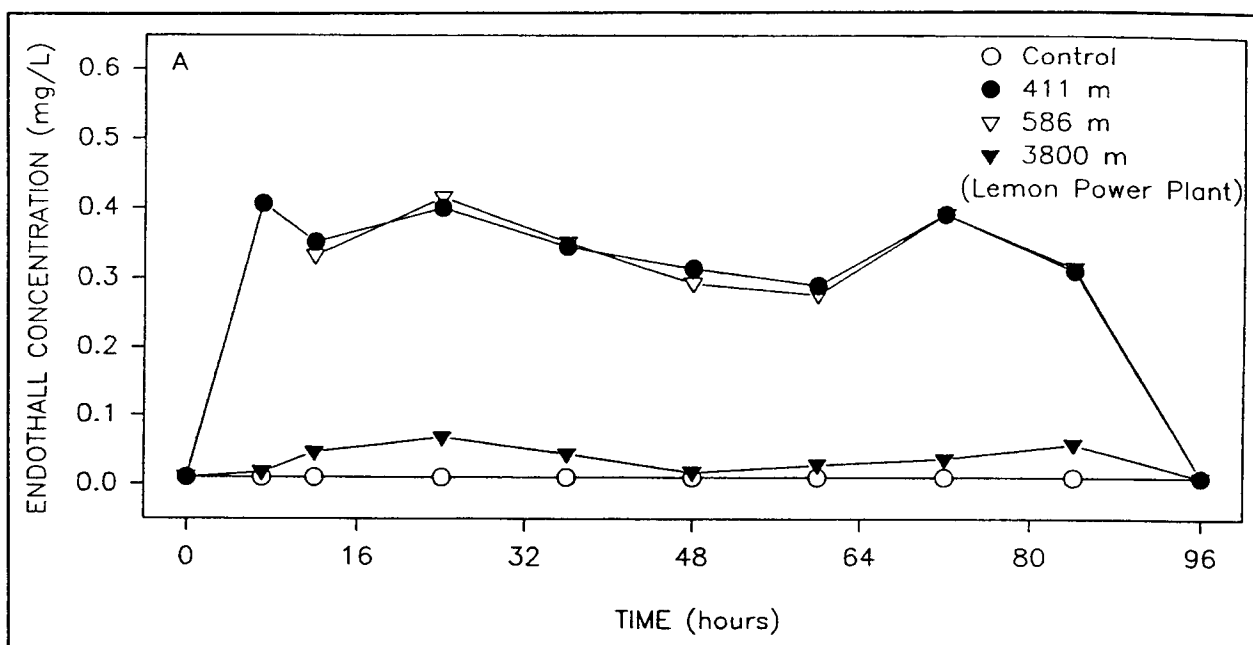


Figure 5. Aqueous endothall residues at four sampling sites in the S19 Canal, Idaho, in August 1996, following a metering treatment targeted to deliver 0.4 mg endothall/L for 84 hr (each value is the mean of duplicate water samples)

biomass ranged from 120 g dry weight/m<sup>2</sup> for the untreated control site to 0 to 2 g dry weight/m<sup>2</sup> for the treated sites. Visual assessments by Northside Canal operational personnel suggested that sago pondweed recovered to about 30 percent of the pretreatment levels during the remainder of the growing season, but was not considered to be at problematic levels.

**Colorado site.** Residue analyses indicated that endothall concentrations ranged from 0.27 to 0.35 mg/L for 55 hr through the entire 5.3-km length of canal (Figure 7). After the 55-hr sampling, residues at all sampling sites dissipated to approximately 0.2 mg/L for the duration of the study (84 hr). Some discrepancies in residue analysis were noted in duplicate water samples, which may account for the variability observed at 36, 48, and 55 hr. While the inability to achieve the target concentration of 0.4 mg/L was not surprising, due to previously described factors, the decrease in residues at 55 hr remains unexplained. Based on the loss of residue after 55 hr, it must be assumed that either flow rates increased without a concomitant increase in delivery rate or the delivery rate slowed without a decrease in flow. Regardless, this represented the initial phase of testing of the new metering pump, and results were generally quite positive. The pump is still in the research phase and will require some development and refinement by Reclamation personnel prior to routine operational use in the field.

Despite lower than targeted endothall concentrations, sago pondweed was significantly reduced by the 17 DAT sampling (Figure 8). Pretreatment biomass ranged from approximately 25 to 137 g dry weight/m<sup>2</sup>, whereas biomass at 17 DAT was 115 g dry weight/m<sup>2</sup> for the untreated control site and ranged from 4 to 20 g dry weight/m<sup>2</sup> for the treatment sites. The late fall treatment and the fact

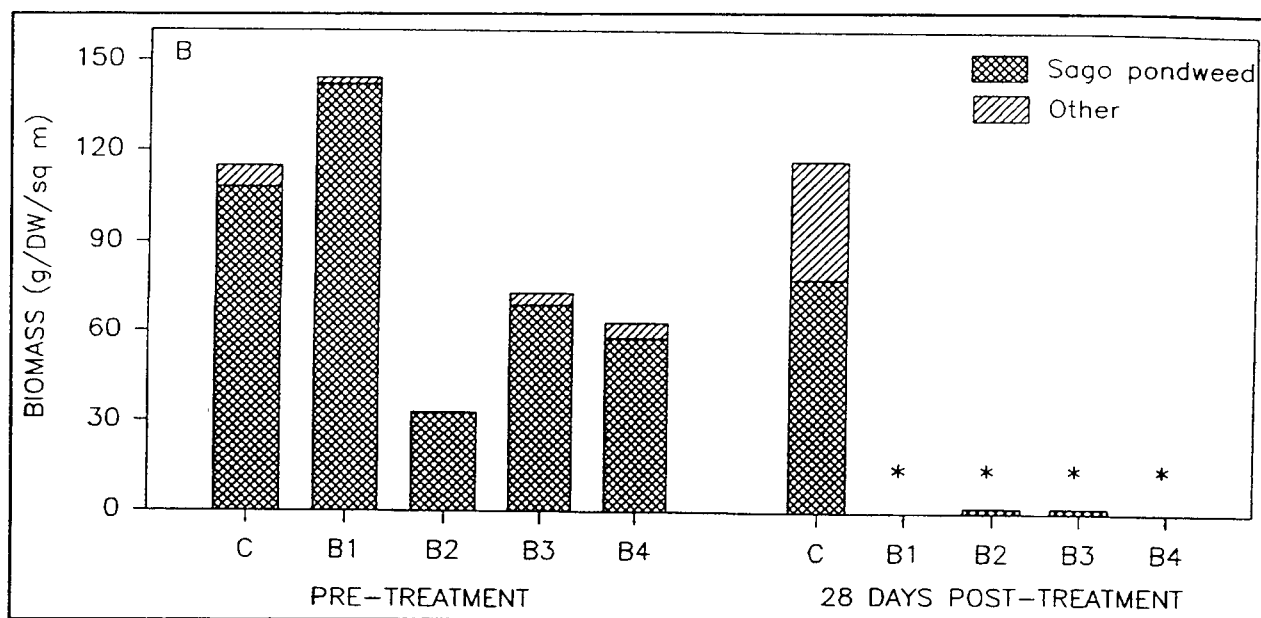


Figure 6. Sago pondweed dry weight biomass in the S19 Canal, Idaho, in August 1996, at 0- and 28-day posttreatment in the untreated control site and four downstream sampling sites exposed to endothall treatment (each bar represents the average of nine biomass samples. Asterisks above the bars indicate that biomass at sampling sites B1-B4 was significantly different from the untreated control site at 28 DAT (Dunnet's test at  $\alpha = 0.05$ ) and was also significantly different from pretreatment biomass values measured at each site (t-test,  $\alpha = 0.05$ )).

that these canals were subsequently dewatered precluded further evaluation of sago pondweed regrowth.

## Discussion

Both the Idaho and Colorado studies demonstrated that delivery of low concentrations of Aquathol K (0.3 to 0.4 mg endothall/L) for 84 continuous hr can effectively control sago pondweed. These results also suggest that it may be possible to further reduce exposure periods and achieve acceptable control of the vegetation. Based on laboratory information from CET relationships, surface applications of higher rates of endothall (3.0 to 5.0 mg/L) along the length of these canals would not have been effective due to the high flow rates and subsequent inadequate exposure periods. In addition, off-target movement of these higher residue levels could cause concern where fish, wildlife, and potable water are issues.

Current irrigation restrictions on the Aquathol K product (7-day restriction at the rates applied) would have to be substantially reduced for the low concentration/extended exposure strategy to find widespread use in Reclamation-managed waters; however, dissipation studies to generate the type of data required for label changes are currently planned by the manufacturer.

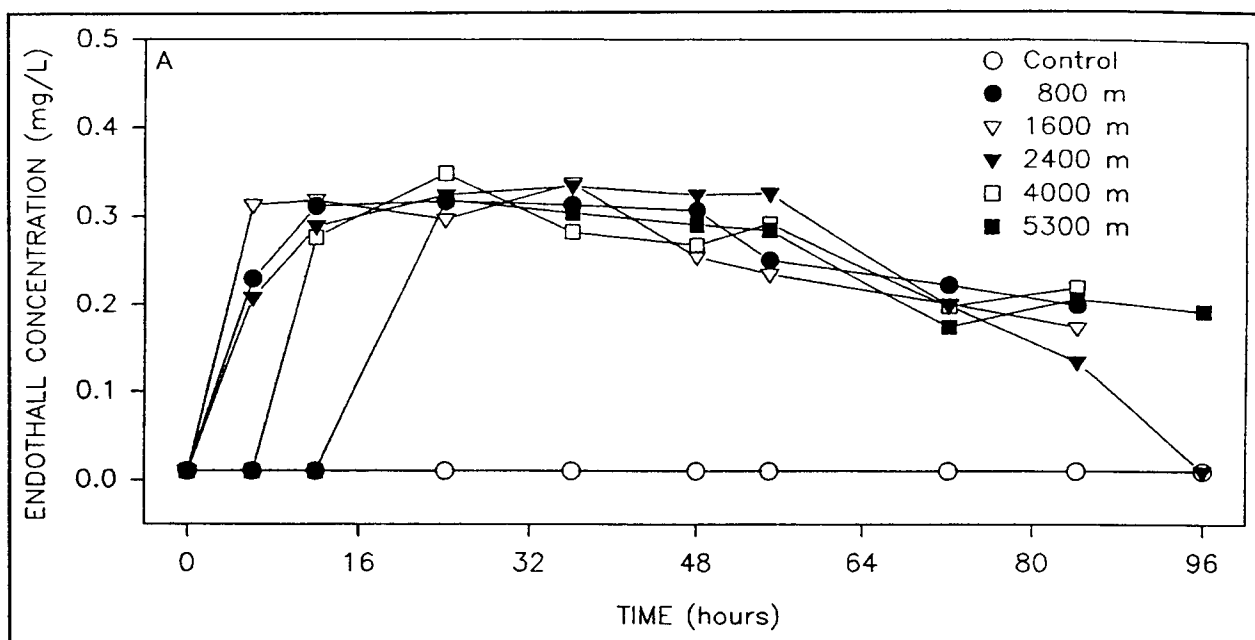


Figure 7. Endothall residues at six sites in the FIDCO Canal in Colorado, September 1996, following metered injection at a target rate of 0.4 mg/L for 84 hr (each value represents the average of duplicate samples)

The delivery pump developed by Reclamation significantly reduced manpower requirements compared with earlier conventional applications and required no maintenance or calibration during the study. Due to the development of this pump, the strategy of delivering low doses of herbicide over time is considered to be quite feasible from an operational standpoint. Although still in the development stage, this pump has excellent potential for precision delivery of herbicides in linear flow systems. The ability to deliver such defined doses should spur future CET studies with several classes of herbicides to determine the minimum rates and exposures necessary for control of a given nuisance species.

While these studies were designed to demonstrate new delivery system techniques and equipment, it should be noted that the biology and life history of the target plants should be taken into account when managing aquatic systems. Although the scope of this study did not address treatment effects on sago pondweed reproduction, proper timing of chemical treatments to interrupt formation of tubers and seeds or to eliminate newly germinating tubers or seedlings should become a goal of operational programs. Determination of optimal treatment times and use rates to reduce propagule banks will negate the need for repeated applications within a growing season and can therefore decrease overall chemical use.

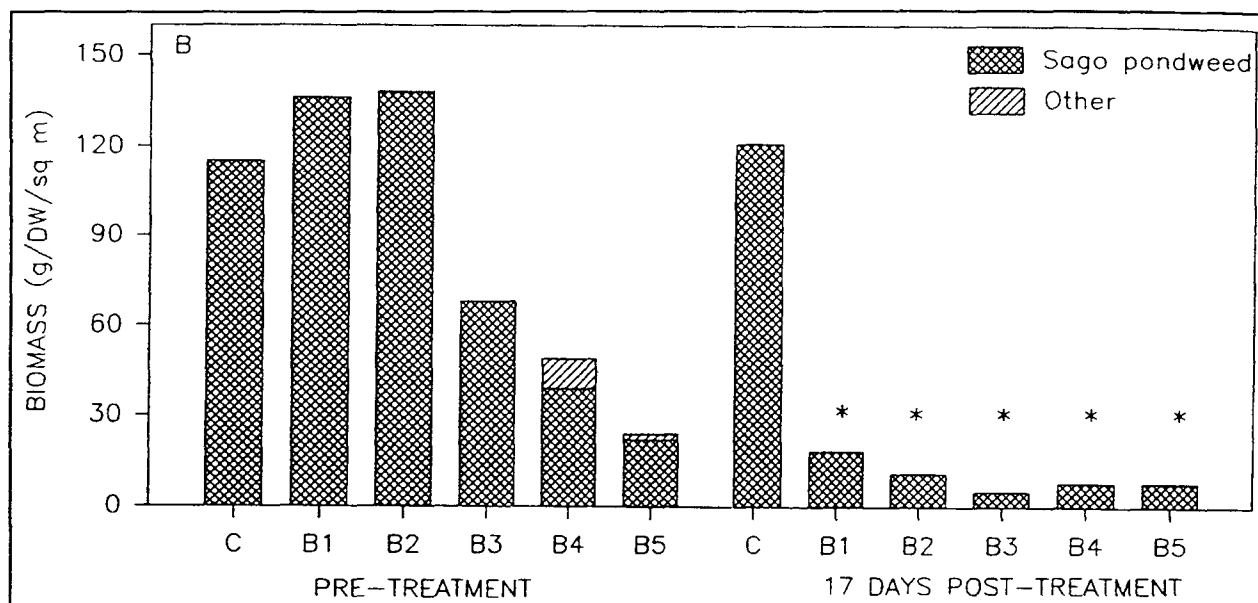


Figure 8. Sago pondweed biomass at pretreatment and 17-day posttreatment collected at six sites from the FIDCO Canal in Colorado in September 1996 (each bar represents the average of nine samples). Asterisks above the bars indicate that biomass at sampling sites B1-B4 was significantly different from the untreated control at 28 DAT (Dunnet's test at  $\alpha = 0.05$ ) and was also significantly different from pretreatment biomass values measured at each site (t-test,  $\alpha = 0.05$ )

## Conclusions and Recommendations

### Conclusions

Based on the field trials conducted with endothall using the low-dose/extended exposure concept, the following conclusions can be drawn:

- The application of endothall at 0.4 mg/L for 72-84 hr was effective in controlling sago pondweed under high-flow conditions (150 to 800 L/sec) in the field.
- The ability to apply low rates of endothall prevented residue levels in discharge waters from exceeding the maximum ARLDW (potable water tolerance) of 0.2 mg/L.
- Utilizing manually adjusted pumps with this treatment strategy has distinct disadvantages in lag-time response and manpower requirements in systems that experience variable flow rates.
- Development of a portable electronic pump with real-time response to variations in flow rates represents a significant improvement over manually adjusted pumps and may provide a tool for applying many

different types of herbicides and application strategies to linear-flow systems.

## **Recommendations**

Based on results of laboratory, mesocosm, and field trials, the following recommendations are made:

- a.* Continue to use laboratory and mesocosm systems to provide guidance for field application rates and exposure times for selected herbicides and nuisance plant species.
- b.* Continue field evaluations in high-flow environments using the low-dose extended exposure concept with aquatic herbicides.
- c.* Work cooperatively with Reclamation personnel to further test and develop delivery metering systems and identify new sites where this technology may be applicable.
- d.* Due to irrigation and potable water restrictions for endothall use, it is suggested that Hydrothol 191 rates of 0.2 mg/L (ARLDW limit) be evaluated at various exposure periods.
- e.* Compare cost-effectiveness of the low-dose extended exposure treatments to conventional applications under a variety of treatment scenarios.

# **3 Mesocosm Evaluation of a New Endothall Granular Formulation for Submersed Plant Control**

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## **Background**

Extensive research has shown that following application, herbicide dispersion and distribution can vary substantially due to changing physical and biological factors such as water exchange characteristics, wind speed and direction, thermal stratification, water quality and temperature, and plant species and density (Fox et al. 1991; Fox and Haller 1994; Getsinger, Sisneros, and Turner 1993; Getsinger 1995). The formulation of an aquatic herbicide (liquid, granular, slow-release granular) can also substantially alter concentration, exposure, and distribution. In addition, the type of carrier used for a granular formulation (clay, lignin, gypsum, polymer), as well as the percent active ingredient (ai) incorporated and release rate from the matrix, can influence both aqueous concentration and exposure (Netherland et al. 1994). Therefore, testing and development of new herbicide formulations are conducted at the laboratory, mesocosm, and field scale to determine methods to optimize application techniques and efficacy as well as to identify potential problems that may occur with operational use of new formulations.

One aspect that must be considered in the development of new herbicide formulations is the impact on the aquatic applicator. Formulations that reduce human exposure by decreasing handling and time required for application are viewed favorably by both applicators and the EPA. A recent example of industry responding to concerns voiced by applicators is demonstrated by the manufacturers of endothall (Elf Atochem North America) pursuing development of a new granular formulation. The current formulation Aquathol (10.1-percent ai dipotassium endothall granule) is generally considered quite efficacious; however, applicators have complained of the high bulk required for treatment (90 percent of the material is inert) and tendency of these granules to form an irritating dust under some environmental conditions. To address these concerns,

a new superabsorbent polymer formulation (SPF) that contains 63-percent dipotassium endothall ai (37-percent inert) and has demonstrated a low potential for dust formation is currently under development.

The SPF is composed of polyacrylate/polyacrylamide polymers (Stockosorb 400 series), which are currently used in agricultural and greenhouse applications to improve soil moisture retention and uniformity (Stockhausen, Inc. 1985). Upon aqueous contact, the granules absorb water and expand (releasing endothall) to form a gel-like material. These polymers are recognized as nontoxic and environmentally safe. Developing nontoxic carriers for herbicide active ingredients is a key objective in the HDS work unit, as it increases environmental compatibility and the likelihood of receiving an aquatic registration by the EPA.

The ability of the new SPF to reduce applicator exposure is based on two primary factors: (a) substantial reduction in the bulk material required to treat a given area, and (b) low potential for the matrix to create dust. For example an endothall treatment targeting 3.0 mg/L in a 2-ha area 2 m in depth would require 1,095 kg of Aquathol (10.1-percent ai), but only 185 kg of the SPF (63-percent ai). Therefore, a conventional granular hopper (91-kg capacity) would only need to be filled twice with the SPF, compared with 10 times for the Aquathol. This can reduce applicator handling and exposure to endothall by decreasing bulk and the time required for application due to decreased reliance upon shore loading or transfer from supply boats.

A series of laboratory and mesocosm studies were undertaken to determine release rates from the SPF, handling and application properties, and efficacy compared with the conventional Aquathol granule. The main advantages of the new formulation are the reduced bulk and potential for decreased applicator exposure; however, due to the decreased number of granules applied to a given area and the unknown release rates of the granules, uncertainty remained as to efficacy of the SPF compared with the conventional Aquathol. Since adequate aquatic plant control is a primary objective of developing a new formulation, efficacy of the new SPF must be comparable with Aquathol to be considered a viable management alternative.

## **Laboratory and Mesocosm Results**

### **Laboratory**

Laboratory studies were conducted with the new SPF to determine release characteristics in the water, physical changes in the matrix, and general handling properties of the product in comparison with the conventional Aquathol formulation (Netherland and Turner 1995). The SPF and Aquathol granules were approximately the same size, and both sank rapidly to the bottom of the 52-L test aquaria following application. The SPF granules expanded (twofold or threefold) within minutes and changed from a hard pellet to a clear gelatinous cube by 15-30 min. In contrast, the Aquathol granules essentially stayed intact. Results in both static water and water that was gently bubbled to provide mixing showed

that both the SPF and the Aquathol granules rapidly released up to 80 percent of the active ingredient endothall within 2 hr and >90 percent by 12 hr (Table 1). Based on the similar release rates, it was evident that efficacy between the products should be comparable.

**Table 1**  
**Laboratory Release of Endothall from the 10.1-Percent**  
**Conventional Granule (Aquathol) and Experimental 63-Percent**  
**SPF Granule Following a 48-Hr Period in Both Static and Mildly**  
**Circulated Water**

Time, hr	Aquathol, % released	SPF, % released <sup>1</sup>	Aqueous Condition
0.1	21	8*	Static
0.5	69	57*	Static
2.0	85	79	Static
6.0	94	87*	Static
12.0	97	95	Static
24.0	97	97	Static
48.0	95	96	Static
0.1	35	15*	Mixed
0.5	85	67*	Mixed
2.0	93	85	Mixed
6.0	98	91	Mixed
12.0	100	98	Mixed
24.0	100	100	Mixed
48.0	100	100	Mixed

<sup>1</sup> Values followed by an asterisk indicate a significant difference (t-test) at the  $\alpha = 0.05$  level between the release of endothall from the Aquathol and SPF.

## Mesocosm

Mesocosm-scale efficacy trials were conducted in outdoor hydraulic flumes (4.5 m wide by 110 m long by 1.2 m deep) located at the Tennessee Valley Authority Brown's Ferry nuclear plant near Athens, AL (Turner et al 1995; Netherland and Turner 1995). Efficacy of the SPF was compared with Aquathol and Aquathol K (liquid formulation containing 40.3-percent ai dipotassium endothall) applied at rates of 1.5 and 3.0 mg/L to the length of the flowing water in the flumes. Pretreatment biomass was collected from plant stands in the upper end (short exposure period) and lower end (longer exposure period) of the flumes, and water residue sampling stations were also established at these sites. Results indicated that efficacy between the two granular formulations was similar, whereas the liquid application was not as effective in the upper end of the flume where the herbicide was rapidly diluted (Figure 9).



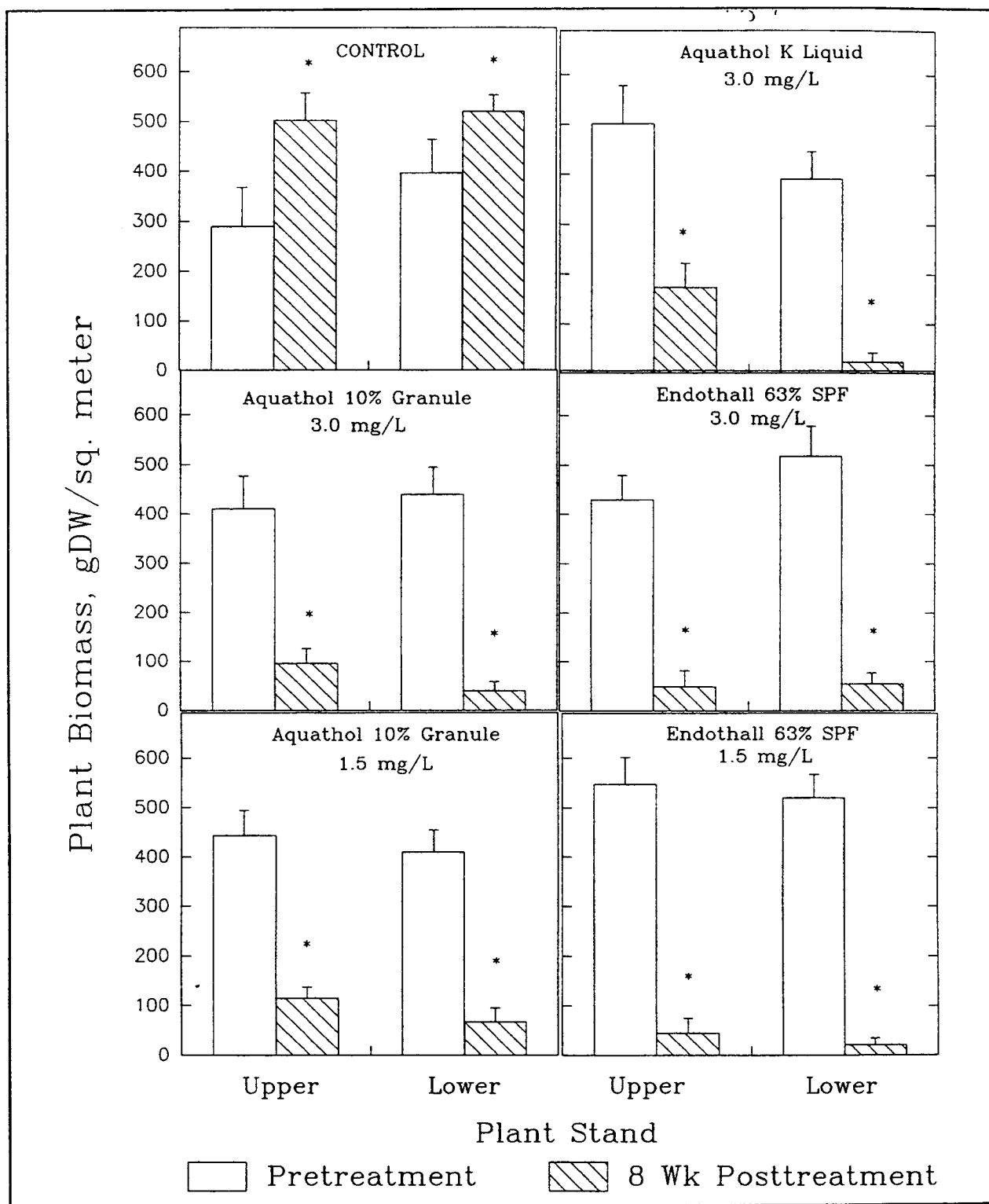


Figure 9. Aquatic plant biomass (Eurasian watermilfoil, American pondweed, and southern naiad) at 0- and 8-week posttreatment following application of a 40.3-percent endothall liquid and 10.1- and 63-percent endothall granules (each bar represents the average of five biomass samples ( $\pm 1$  SD), and asterisks above bars indicated a significant difference between pretreatment and 8-week posttreatment biomass according to a t-test ( $\alpha = 0.05$ ))

Endothall concentrations were near target levels by 1- to 3-hr posttreatment, indicating that SPF and Aquathol rapidly release endothall from the matrix (Table 2). Residues were substantially reduced between 12- and 24-hr posttreatment in all treatments. The linear flow pattern in the flumes resulted in longer exposure periods in the lower plant stands.

**Table 2**  
**Aqueous Endothall Concentrations Sampled over a 48-Hr Period from Middepth Following Treatment of Hydraulic Flumes with a Liquid (Aquathol K), Granular (Aquathol), and Polymer (SPF) Formulation**

Target rate, mg/L	Hours Posttreatment							
	1	3	6	12	18	24	36	48
SPF 1.5	1.5	1.6	1.2	0.9	0	0	0	0
SPF 3.0	3.3	3.4	2.0	1.3	0.5	0	0	0
Aquathol 3.0	7.6	3.1	1.6	0.8	0.2	0	0	0
Aquathol K 3.0	5.8	3.5	1.1	0.2	0.3	0	0	0

Sampling at depths of 25 and 100 cm indicated that the distribution of endothall following the SPF treatments was not uniform through the water column (Table 3). Surface readings were consistently higher through 12-hr posttreatment. Between 12 and 24 hr, the readings were much more comparable. These results were somewhat surprising, as granules generally tend to concentrate the compound at lower depths (Getsinger 1995). It should be noted that the flumes were nearly 100 percent covered with thick mats of Eurasian watermilfoil, Southern naiad, and filamentous algae (*Hydrodictyon* spp. and *Cladophora* spp.), which represented a worst-case treatment situation and likely resulted in the granules becoming entangled in the upper portions of the mats. In addition, the thick vegetation enhanced formation of a daily thermocline with water temperatures ranging from 35 °C in the top 25 cm to 23 °C near the sediment. This example illustrates the difficulty of achieving even distribution of a herbicide if plants are allowed to form a surface canopy. Tying up the majority of herbicide in the upper portions of the water column can be particularly problematic with contact herbicides such as endothall because the lower portions of the plant may not be affected although they represent an excellent source of posttreatment regenerative potential. In contrast, it is thought that delivering the herbicide to the bottom waters can increase efficacy, especially with plants like Eurasian watermilfoil and hydrilla (*Hydrilla verticillata* L.f. Royle), which often recover from rootcrowns following treatment.

Although the flumes were much smaller than typical field-treated sites, conventional application equipment was used to compare handling characteristics and ease of application of the SPF and Aquathol formulation. The SPF at a target rate of 3.0 mg/L required only 2.7 kg of material, while the Aquathol required

**Table 3**  
**Distribution of Aqueous Endothall Concentrations in Hydraulic Flumes Following Treatment of Dense Vegetation with SPF and Aquathol Granular Formulation (samples were collected 25 cm below the water surface and at 25 cm above the sediment)**

Target rate, mg/L	Depth, cm	1	3	6	12	18	24	36
SPF 1.5	25	3.3	1.0	0.9	0.3	0.1	0	0
	100	0.5	1.0	0.7	0.3	0.2	0	0
SPF 3.0	25	3.7	5.7	4.2	0.7	0.2	0	0
	100	1.2	1.4	1.7	1.2	0.4	0	0
Aquathol 3.0	25	4.5	3.7	3.8	3.1	0.5	0	0
	100	0.4	0.9	1.1	0.7	0.2	0	0

24 kg of material. Even at this small scale, a marked difference was noted in the amount of effort required to apply the two formulations. Applicators did note an irritating dust generated by the Aquathol granules, whereas no significant production of dust was noted when applying the SPF granules.

Results from laboratory and mesocosm studies suggested that the SPF and conventional Aquathol produced similar release rates, aqueous concentrations, and efficacy. The major difference between the materials was noted in the bulk amount required for application and the potential for limiting dust production. Field testing of the SPF product was recommended to provide information on any potential problems with its handling and application or quality control problems with the bulk material.

# **4 Evaluation of Endothall SPF and Aquathol in a Central Florida Lake**

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## **Background**

Based on results of laboratory and mesocosm studies, field testing of the endothall SPF product was recommended to provide information on any potential problems with its handling and application or quality control problems with the bulk material. While this information is difficult to quantify and is somewhat subject to the opinion of the individual applicator, it is nonetheless quite important as early detection of potential problems can lead to modifications that prevent handling difficulties or fouling of application equipment. Residue and efficacy data should also be collected to determine if notable differences between the SPF and Aquathol exist. It should also be noted that direct field efficacy comparisons can be somewhat arbitrary, as conditions within treatment plots (water exchange, plant density, depth) can vary greatly, resulting in different distribution patterns and exposure periods.

## **Methods**

### **Site selection and methods**

Lake Weohyakapka (Lake Weo) is a 3,048-ha (7,532-acre) lake near the city of Lake Wales (Polk County) in central Florida. A substantial infestation of hydrilla (60- to 80-percent coverage) exists throughout the shallow lake. Four square treatment plots of 1.6 ha (4 acres) were established along the shoreline in the southwest cove of the lake (Figure 10). A control plot receiving no herbicide treatment was established on the west shoreline of the lake. Ten depth measurements were taken in Plots 1 to 4, and average depths of 1.8, 1.8, 1.8, and 2.7 m were recorded. Assignment of treatments to the plots and the amount of product applied are shown in Table 4.

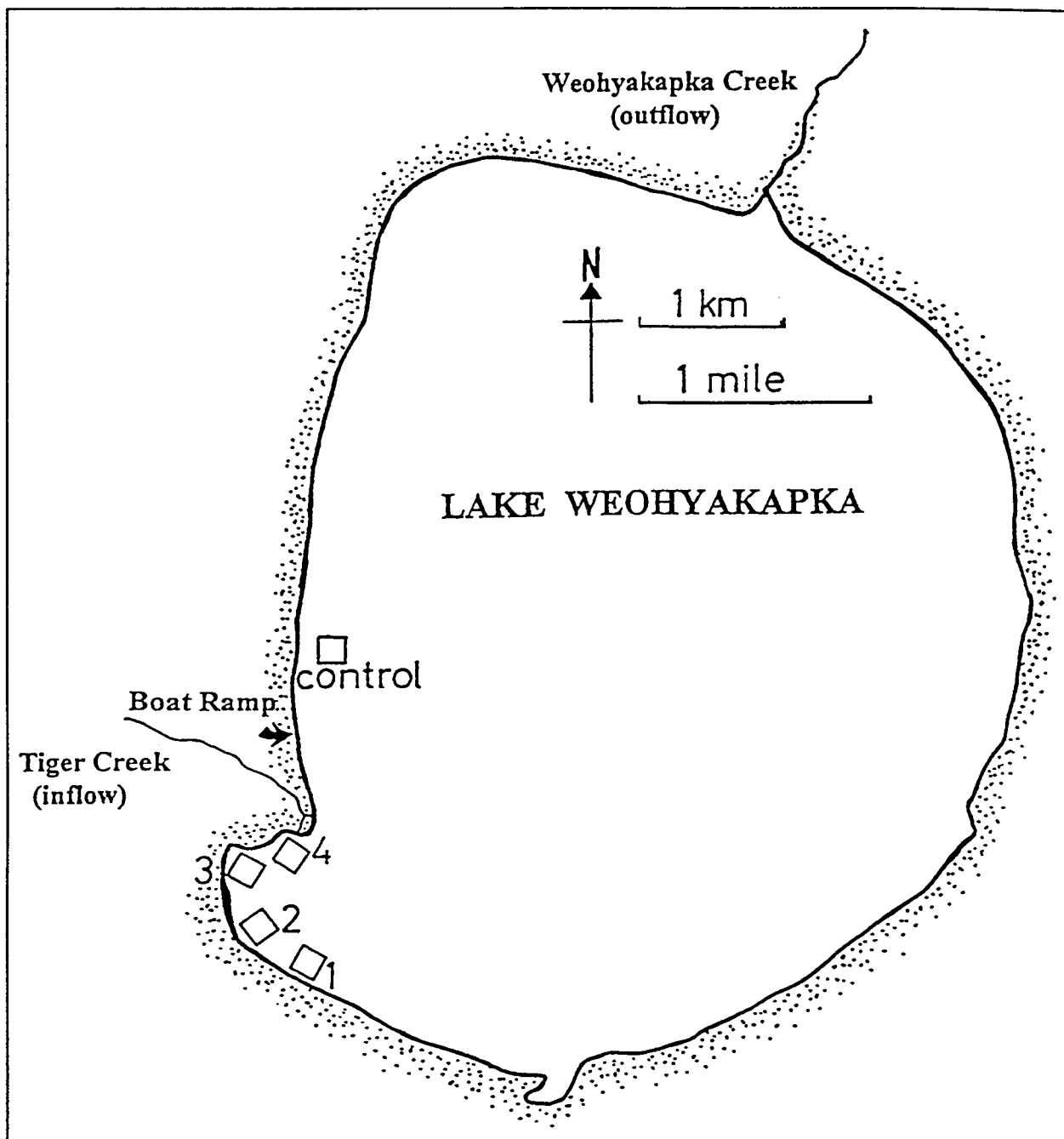


Figure 10. Schematic map of 3,048-ha Lake Weohyakapka, Florida, showing locations of 1.6-ha treatment plots (plots not drawn to scale)

**Table 4**

**Amount of Aquathol (10.1-percent ai) and SPF (63-percent ai) Endothall Formulation Applied to 1.6-Ha Treatment Plots on Lake Weohypapka, Florida**

Plot	Formulation	Rate, mg/L	Acre-Feet Treated	lb a.i.	Total lb of Formulation
1	Aquathol	2.0	24	129.6	1,283
3	Aquathol	3.0	24	194.4	1,925
2	SPF	3.0	24	194.4	299
4	SPF	2.0	36	194.4	299

The SPF and Aquathol formulation were applied between 7:20 and 9:45 a.m. on 21 March 1995. A Gran-blo granule blower was used in Plots 1 and 4 to apply SPF and Aquathol at 2 mg/L endothall, and cyclone spreaders were used in Plots 2 and 3 to apply SPF and Aquathol at 3 mg/L endothall.

#### **Endothall distribution and correlation of residues to rhodamine WT**

The liquid dye RWT was applied to Plots 2 and 4 (1,500 and 2,000 ml, respectively) concurrent to the granule applications to achieve an initial concentration of 12 µg/L. Water samples for endothall residue analysis were collected from three stations in each plot from 0.25 m below the water surface and 0.25 m above the bottom. Residue samples were collected from the plots at 0, 3, 6, 11, and 24 hr and 2, 3, 5, and 7 DAT. RWT concentrations were measured using a Turner Designs Field Fluorometer in Plots 2 and 4 under the same sampling regime as for endothall residues, but only up to 2 days after treatment.

#### **Efficacy**

Prior to treatment, hydrilla was just below the water surface in all plots, and a fathometer was used to record the percentage infestation of a cross section of the water column. Fathometer transects were run across both diagonals of each plot at 0, 4, 9, 17, and 27 weeks after treatment. The area of the weed-infested portions of the tracings was calculated, and a percent water column infested with hydrilla was determined.

## Results

### Residue distribution and correlation

**Plots 1 and 3 (conventional granules).** In Plots 1 and 3 (to which the 10-percent granules were applied without dye), endothall residue concentrations were higher at the bottom of the water column through the first 12-hr posttreatment (Table 5).

<b>Table 5</b> <b>Endothall Concentrations Averaged for All Three Sampling Stations at Each Sampling Time in Plots 1 and 3 (these plots were treated with Aquathol granules at target rates of 2.0 and 3.0 mg endothall/L, respectively)</b>			
Time, hr	All Avg	Top Avg	Bottom Avg
<b>Plot 1</b>			
3	0.271	0.09	0.45
6	0.407	0.05	0.76
12	0.073	0.00	0.15
24	0.053	0.11	0.00
48	0.000	0.00	0.00
<b>Plot 3</b>			
3	1.345	0.628	2.061
6	0.740	0.603	0.877
12	0.888	0.759	1.016
24	0.450	0.568	0.332
48	0.120	0.110	0.129
72	0.245	0.000	0.490
120	0.085	0.101	0.068
168	0.021	0.043	0.000

**Plot 2 (SPF).** Dye concentrations in Plot 2 were much greater at the top of the water column than at the bottom throughout the 48-hr sampling period. Conversely, endothall residues were higher in the bottom samples than in the top, at least for the first 12 hr (Table 6). Thus, the linear correlation between the dye and endothall concentrations in Plot 2 was very poor ( $r^2 = 0.0031$ ).

The limited vertical mixing of the dye was attributed to the short hoses (0.6 m long) with which it was applied and the temperature stratification of up to 5.3 °C difference top to bottom that established in each plot (Table 7). The higher

**Table 6**  
**Endothall and Dye Concentrations (temperature corrected)**  
**Averaged for Three Sample Stations at Each Sampling Time in**  
**Plots 2 and 4 (these plots were treated with the SPF formulation at**  
**target rates of 3.0 and 2.0 mg endothall/L, respectively)**

Time, hr	All Avg	Top Avg	Bottom Avg
<b>Plot 2</b>			
<b>Corrected Dye</b>			
3.25	6.313	12.54	0.08
6.33	3.292	6.58	0.00
11.4	2.578	5.15	0.01
24.3	0.573	1.13	0.02
48	0.115	0.23	0.00
<b>Endothall</b>			
3.25	0.383	0.34	0.42
6.33	0.703	0.20	1.21
11.4	0.322	0.18	0.47
24.3	0.203	0.27	0.13
48	0.000	0.00	0.00
<b>Plot 4</b>			
<b>Corrected Dye</b>			
3	17.325	34.520	0.130
6	5.900	11.793	0.006
10.5	0.697	1.393	0.000
23.5	0.507	0.923	0.092
47.2	0.490	0.814	0.166
<b>Endothall</b>			
3	1.200	1.819	0.580
6	0.324	0.558	0.090
10.5	0.000	0.000	0.000
23.5	0.000	0.000	0.000
47.2	0.000	0.000	0.000



**Table 7**  
**Dye Concentration and Temperature Profiles Throughout the**  
**Water Column at Selected Sampling Stations in Plots 2 and 4 at**  
**6-Hr Posttreatment**

Plot	Depth, m	Temperature, °C	Corrected Dye, ppb
2 Station C	Surface	24.8	5.25
	0.25	24.7	5.56
	0.50	24.0	13.87
	0.75	22.0	10.90
	1.00	21.4	2.11
	1.25	21.1	0.10
	1.50	20.9	0.02
	1.75	20.9	0
4 Station A	Surface	24.9	29.68
	0.25	25.4	26.14
	0.50	24.2	12.71
	0.75	22.7	4.27
	1.00	21.6	1.40
	1.25	20.4	0.12
	1.50	19.8	0
	1.72	19.7	0
	2.00	19.6	0
	2.25	19.6	0
	2.50	19.7	0

endothall concentrations near the bottom in Plot 2 suggest that the polymer granules penetrated the thermocline and were releasing more herbicide near the bottom of the water column (Table 6). The water volume in Plot 2 was 100-percent infested with hydrilla at the time of treatment, which encouraged thermal stratification. It was suspected that the thick plant canopy might trap endothall polymer granules near the surface, but the passage of the airboat over the hydrilla mat may have assisted in the vertical dispersal of the granules.

**Plot 4 (SPF).** The vertical stratification of dye was similar in Plot 4 to that in Plot 2; however, unlike Plot 2, endothall residue concentrations were higher at the top of the water column than at the bottom (Table 6). Whether the endothall polymer granules were more limited in penetrating the water column in Plot 4

because of a thicker hydrilla canopy or because of a greater initial thermal stratification (this plot was treated 1.25 hr later) was not determined.

A consequence of the different endothall residue distributions in Plot 4 was that there was a significant correlation between the dye and endothall concentrations ( $r^2 = 0.773$ ,  $n = 30$ ) mostly due to samples from the top of the water column ( $r^2 = 0.848$ ,  $n = 15$ ), as there was not a significant correlation in the bottom samples ( $r^2 = 0.097$ ,  $n = 15$ ). Significance of dye/endothall correlation in Plot 4 was dependant on the earliest samples (3 and 6 hr after treatment), so there was limited value in later comparisons.

The lateral distribution of endothall in each plot was somewhat uneven by the first sampling time, with concentrations varying between 0, 0.52, and 0.74 mg/L at the bottom of the water column in Plot 2 and between 0.95, 1.88, and 2.63 mg/L at the top in Plot 4. Such lateral variations were typical at each sampling time. This high variability in lateral and vertical distribution of residues should be acknowledged as a limitation of the dissipation data. It is possible that applying fewer granules with a more concentrated active ingredient may be responsible for some of the lateral variation in residues.

## Residue dissipation

**Plots 1 and 3 (conventional granules).** There was a significant exponential reduction in endothall concentrations in Plot 1, with a half-life of 9.1 hr ( $r^2 = 0.95$ ). Endothall was not detected in this plot after 24 hr (Table 5). However, the regression predicted an initial endothall concentration of only 0.35 mg/L.

Plot 3 was unique in having detectable concentrations of endothall at all sampling times (Table 5). Concentrations from 120 and 168 hr after treatment were between 0.13 and 0.22 mg/L. Based on the first five sampling times only (as with Plots 1 and 2), a significant exponential reduction in endothall concentrations resulted in a half-life estimate of 13.9 hr and an initial concentration of 1.4 mg/L ( $r^2 = 0.98$ ). If data from all eight sampling times were included, the regression remained significant, the half-life increased to 27.3 hr, and the initial concentration would have been 0.91 mg/L ( $r^2 = 0.95$ ).

**Plot 2 (SPF).** A slight delay in total release of endothall from the polymer in Plot 2 was suggested, but overall dissipation showed an exponential decrease in concentration (Figure 11). The half-life for endothall from all samples was 7.9 hr ( $r^2 = 0.92$ ), which was the same as that of the dye ( $r^2 = 0.98$ ).

Based on the y intercept of the exponential dissipation equations, the initial average residue concentrations in Plot 2 were 0.91 mg/L of endothall and 6.5  $\mu$ g/L of dye. These values were considerably lower than the objective concentrations of 3 mg/L and 10 to 12  $\mu$ g/L for endothall and dye, respectively.

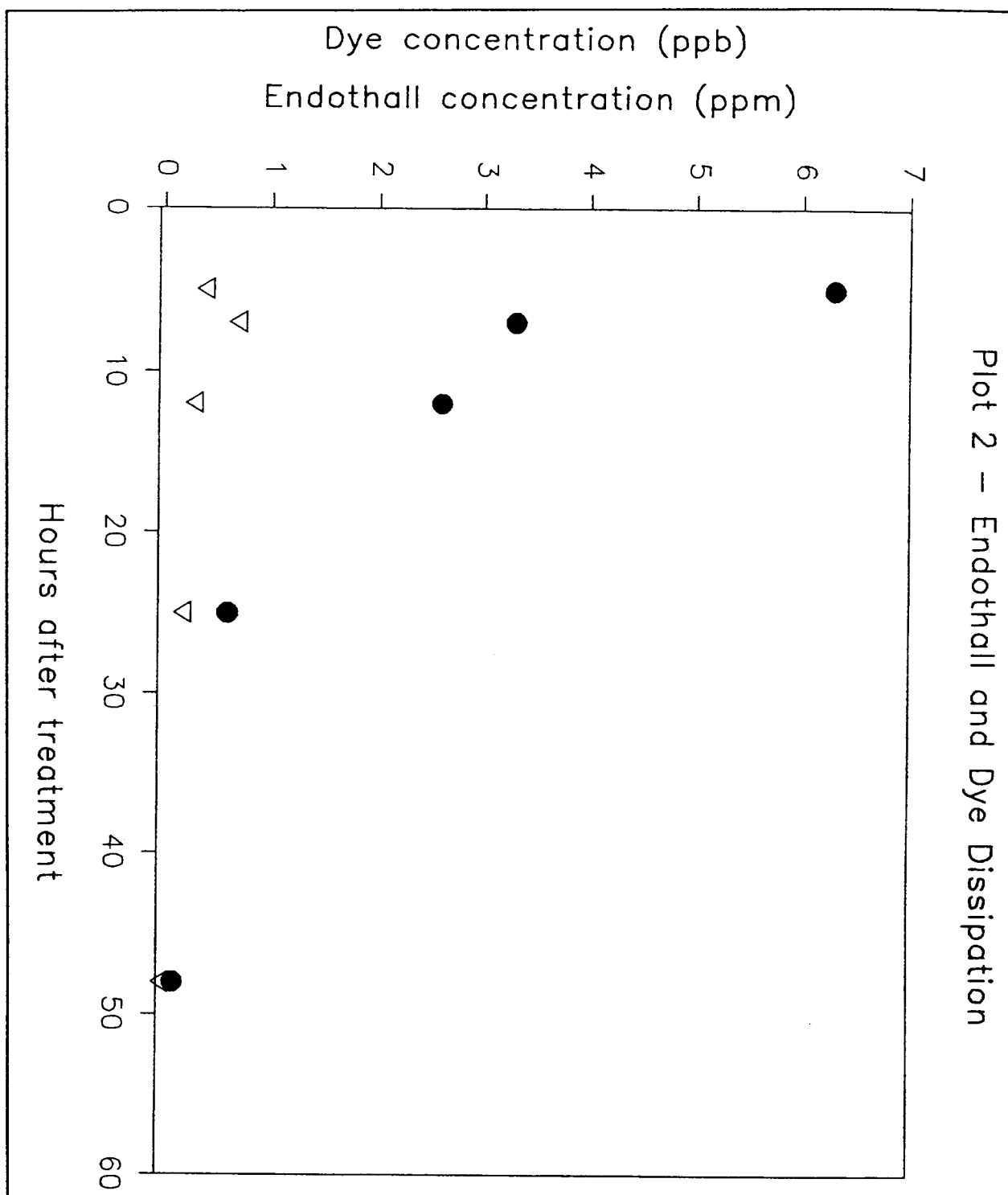


Figure 11. Endothall and rhodamine dye dissipation from Plot 2 following target application rates of 3.0 mg/L endothall as the SPF and 12  $\mu\text{g/L}$  liquid dye

The half-life for endothall was calculated using data from only the first five sampling times. Endothall was not detectable in Plot 2 at 48 or 72 hr after treatment. For regression calculations, a value of 0.01 mg/L is used for nondetectable concentrations. It should be noted that 5 out of 6 samples collected 120 hr after treatment had detectable endothall concentrations between 0.13 and 0.22 mg/L, and two of six samples collected 168-hr posttreatment had endothall concentrations between 0.24 and 0.62 mg/L. Previous data from the studies conducted in the Idaho canals suggest that exposure of 72 to 84 hr to endothall at 0.2 to 0.4 mg/L was quite effective at controlling sago pondweed. Therefore, it is possible hydrilla control was related to long-term exposure to low concentrations of endothall being released from the granules.

**Plot 4 (SPF).** Endothall rapidly dissipated from Plot 4 with no detectable residues by 12 hr ( Figure 12). Using only three sampling times, the exponential regression of endothall dissipation was not significant ( $r^2 = 0.090$ ). (This regression would have indicated a half-life of 1.1 hr and an initial endothall concentration of 10.7 mg/L). As observed in Plot 2, endothall was detected in three of six samples from Plot 4, at concentrations of 0.155 and 0.139 mg/L, 120 hr after treatment.

## Efficacy

Due to the variability in initial endothall concentrations, distribution, and dissipation rates, comparisons of efficacy between the two formulations are quite speculative. Prior to treatment, hydrilla was just below the water surface and occupied from 62 to 100 percent of the water column (Table 8). Following treatment, water levels in Lake Weo rose 0.25 m, and a significant algae bloom developed lakewide. Hydrilla control occurred (50- to 70-percent control) in Plots 1, 2, and 4 by 4 weeks after treatment (WAT) and held through 9 WAT (Table 9). With the exception of Plot 4, hydrilla began to recover at 9 weeks although treatment areas were still visually distinguishable from surrounding untreated zones up to 27 WAT. Plot 3 (10.1-percent granule at 3.0 mg/L) experienced virtually no hydrilla control throughout the study, whereas Plot 4 (SPF at 2.0 mg/L) still had excellent control of hydrilla at 27 WAT.

## Discussion

The lack of strong correlations between the dye and endothall concentrations is not surprising, given the different application methods and formulations of the two materials, as well as the apparent effects of thermal stratification. Why the vertical distribution of endothall was different in Plot 4 (more endothall at top than at bottom of water column) is not clear, but the positive correlation with dye in this plot was very brief (less than 12 hr).

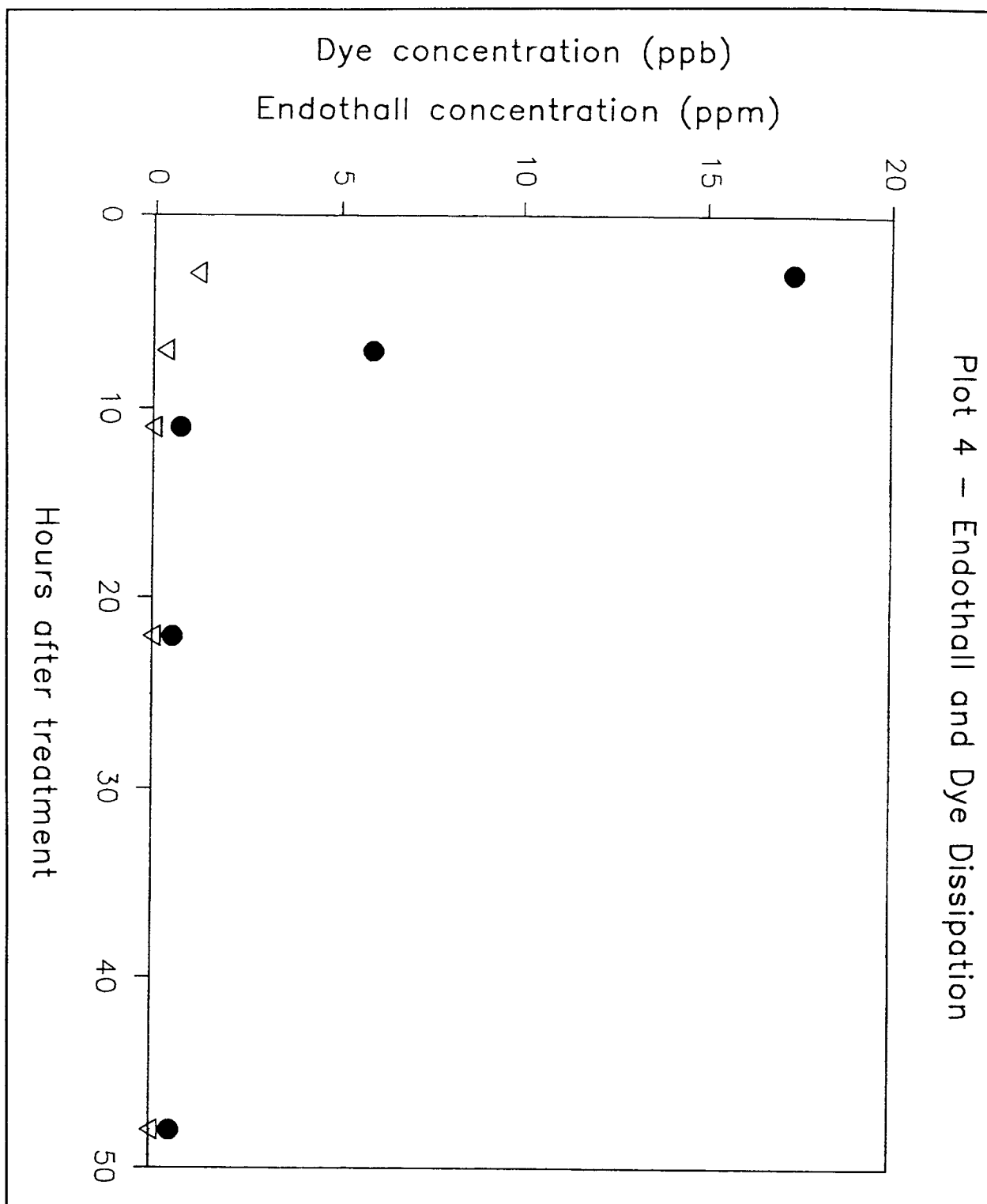


Figure 12. Endothall and rhodamine dye dissipation from Plot 4 following target application rates of 2.0 mg/L endothall as the SPF and 12  $\mu$ g/L liquid dye

**Table 8**  
**Percent Water Column Infested with Hydrilla Determined from**  
**Fathometer Transects Across the 1.6-Ha Endothall-Treated Plots**  
**in Lake Weohyapapka, Florida**

Plot		Weeks After Treatment				
		0	4	9	17	27
1	A	62	23	28	26	76
2 ppm 10%	B	77	25	37	36	70
	Ave	70	24	32	31	73
2	A	100	51	42	70	93
3 ppm 65%	B	100	47	33	76	98
	Ave	100	49	37	73	96
3	A	94	70	71	100	100
3 ppm 10%	B	97	78	81	93	100
	Ave	96	74	77	97	100
4	A	95	49	20	18	17
2 ppm 65%	B	94	38	25	18	15
	Ave	95	44	22	18	16
	Check Plot	100	100	100	100	100

Note: Time 0 = Day of treatment (March 21, 1995). A = SW-NE Transect. B = SE-NW Transect.

**Table 9**  
**Percent Reduction (control) of Hydrilla Biomass in Aquathol- and**  
**SPF-Treated Plots in Lake Weohyapapka, Florida, Corrected for**  
**Water Depth (m mean sea level) and Initial Plot Infestation (data**  
**are based on the percent reduction in volume of water infested)**

Plot	Weeks After Treatment, depth				
	0 (18.8)	4 (18.7)	9 (18.6)	17 (18.8)	27 (18.9)
1	0	69	77	76	32
2 ppm 10%, Aquathol					
2	0	54	62	27	5
3 ppm 65%, SPF					
3	0	26	34	8	0
3 ppm 10%, Aquathol					
4	0	56	60	90	96
2 ppm 65%, SPF					
Check Plot	0	0	0	0	0

Note: 0 = Treatment Date, March 21, 1995. 4 = April 19, 1995. 9 = June 1, 1995. 17 = July 28, 1995. 27 = Sept. 28, 1995.

Dye half-lives were not very long in either plot, probably due to wind-generated water movement. When applied in comparable formulations, endothall tends to have a shorter half-life than RWT because it is more rapidly degraded and removed from the aquatic environment (Fox, Haller, and Shilling 1991). However, in Plot 2, because the endothall was applied in the supersorbent formulation, there was a slight delay (up to 6 hr) in the release of the endothall, which resulted in similar half-lives for the dye and endothall.

It remains unclear as to why the endothall residues apparently dissipated so much faster from Plot 4 than from Plot 2, or in comparison to the dye. Such divergent behavior of endothall residues between the two polymer plots is difficult to explain, especially considering that the dye did not seem to disperse from Plot 4 much faster than from Plot 2. Examination of the polymer application methods (which were somewhat complicated by lumps of granules sticking in the spreaders) and subsequent polymer release rates would likely yield useful information. It is also possible that the water-sampling protocol may have been insufficient to adequately characterize the distribution and subsequent dispersion of aqueous endothall concentrations in Plot 4. The excellent level of hydrilla control achieved in this plot further suggests that active endothall residues were present and missed by sampling.

Endothall dissipation from Plot 1 fit into the general rate for the bay (similar to Plot 1 dye and endothall, Plot 4 dye). Endothall dissipation from Plot 3 was slower both in terms of half-lives and the detection of residues at all sampling times. The position of this plot in the back of the cove (furthest from the open lake) might have contributed to the reduced dissipation rate.

Although the plots were separated by several hundred meters, dye was detected downwind of Plots 2 and 4, indicating that contamination between plots could have occurred. Based on the constant presence of endothall in Plot 3, this plot could have contaminated the others. However, it should be noted that the general lack of hydrilla control outside of the plot treatment boundaries and the significant dilution that would occur between plots better supports continued release of endothall from the granular matrices versus interplot contamination.

The low initial endothall concentrations predicted by the dissipation regression equations (0.35, 0.91, and 1.4 mg/L instead of the intended 2, 3, and 3 mg/L for Plots 2, 3, and 4, respectively) is not uncommon, particularly following the use of granular formulations. The release of endothall from the granules or polymer is delayed, and therefore the exponential model of dissipation (which is based on an assumption of a constant rate of water dilution or chemical decomposition) may no longer be entirely valid. The exponential regression may be significant and the half-life reflective of the average rate of dissipation, but the y-intercept will not accurately predict the initial concentration of endothall if there is release over a period longer than the interval between sampling times. Data on the release rate of endothall from the granules and polymer (accounting for concurrent degradation) would be needed to establish whether this was an important issue here.

Despite the relatively short half-life predictions and lower than expected initial endothall concentrations, the efficacy of both polymer and granules in Plots 1, 2, and 4 was considered to be good (62- to 96-percent reduction from initial biomass). Information on herbicide distribution following granular treatments is limited compared with liquid applications, and it is possible that sampling protocols designed for liquid applications may not accurately reflect the vertical distribution of endothall following a granular application. This could lead to incorrect estimations of endothall concentrations and predicted half-lives.

The limited treatment effect in Plot 3 (34-percent hydrilla reduction) remains anomalous, as this plot had the longest measured endothall half-life and persistence of detectable residues, the highest initial endothall concentration, and one of the most even vertical distributions of endothall in the water column. Again, it may be possible that granules applied in Plots 1, 2, and 4 continued to release endothall near the bottom sediments that was not being detected by the water-sampling techniques employed. The long-term and excellent hydrilla control (50 to 60 percent at 27 weeks) observed in Plot 4 (63-percent SPF granule at 2.0 mg/L) is in stark contrast to the other plots and remains somewhat anomalous. The greater depth of Plot 4 (0.9 m > in depth than Plots 1, 2, and 3) is suspected to have contributed to a lack of hydrilla regrowth. Untreated hydrilla in other portions of the lake at depths of 3 to 3.66 m (10 to 12 ft) was also noted to have declined in conjunction with increased water depth.

Based on the results collected to date, Elf Atochem has submitted the new SPF formulation to the EPA for registration. Recent studies in the spring of 1997 suggest that the formulation has been improved to prevent some of the clumping of the material that was observed during this study. As the process of EPA registration of a new formulation moves forward, plans are underway for 1998 field testing of the SPF at several locations.

## **Conclusions and Recommendations**

### **Conclusions**

Based on results of this field study, the following conclusions can be made:

- a. Due to the variation in residue distribution and anomalous results between plots, direct comparisons of efficacy between the SPF and Aquathol formulations are difficult based on these data.
- b. The SPF provided good hydrilla control from 9 to 27 WAT.
- c. There was no strong evidence that the SPF was any less or more effective than the currently registered granular Aquathol formulation.



- d.* Field application of the SPF resulted in the use of six times less bulk compared with the conventional Aquathol granular formulation.
- e.* Although some clumping of the SPF formulation was noted, the product presented only minor application problems and did not result in the formation of any irritating dust.

## **Recommendations**

Based on results of this field study, the following recommendations can be made:

- a.* Improved methods are needed for sampling and determining aqueous residues following granular herbicide applications.
- b.* Continued field trials with the improved SPF are suggested in order to identify potential application problems and recommend treatment rates prior to EPA registration of this product.
- c.* New formulations with potential for increased efficacy, enhanced environmental compatibility, or improved safety for applicators should be identified.

## 5 Comparison of Fluridone Degradation in Ponds Following Treatment with Liquid and Slow-Release Pellet Formulations

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### Background

In comparison to most aquatic herbicides that require contact times ranging from a few hours to a few days, the herbicide fluridone is unique in that it requires exposure periods of several weeks to provide effective submersed plant control (Netherland, Getsinger, and Turner 1993; Netherland and Getsinger 1995a). Although an extended exposure period is required, fluridone has been shown to maintain herbicidal activity on some aquatic species at concentrations approaching 1 µg/L (Netherland and Getsinger 1995b; Fox, Haller, and Shilling 1996). The activity of fluridone on key target species such as Eurasian watermilfoil and hydrilla (*Hydrilla verticillata* L.f. Royle) at exceedingly low concentrations is also seen as the basis for the selective potential of fluridone with many desirable native species (Netherland, Getsinger, and Skogerboe 1997).

Techniques that allow for extending fluridone exposure in large flowing environments by staggering the applications to apply low doses (8-15 µg/L) of the herbicide over time have been developed for hydrilla control (Fox, Haller, and Shilling 1994a,b). However, use of fluridone for both large and small spot treatments in lakes and reservoirs continues to provide inconsistent control due to variable dispersion of residues from the treatment site.

Most of the early residue work with granular formulations of fluridone were conducted with a clay pellet formulation (Sonar 5P) that is no longer available and was not specifically designed as a controlled-release matrix (West et al. 1983). Currently, the only aquatic herbicide formulation on the market with claims of controlled-release properties is the Sonar slow-release pellet (SRP). In comparison to data collected for the liquid aqueous suspension (AS) and 5P

formulation, there is limited information on fluridone residues following SRP treatments. While off-target dispersion of residues is the major factor affecting efficacy of fluridone treatments, the SRP formulation can be affected by many variables (e.g., initial concentration achieved and the rate and duration of release of fluridone from the pellets, sediment type, and percent organic matter) that do not affect the liquid; therefore, results obtained with SRP treatments are often not easily explained. In the case of the SRP, both successes and failures with spot treatments have rarely been documented by collecting aqueous fluridone residues, and the release rates from the SRP formulation following both low- and high-rate applications to treatment blocks of varying size are not well characterized or understood.

While maximum aqueous concentrations of fluridone are reached soon after the application of the liquid formulation, a significant delay of 1 to 3 weeks is noted with the SRP formulation as the fluridone slowly dissociates from the clay matrix. Due to the extended release of fluridone from the SRP formulation, maximum aqueous residues are much lower than would be predicted if all the fluridone in the pellet dissolved instantly to achieve the theoretical application rate. This difference in initial aqueous concentrations and subsequent exposure periods likely accounts for field observations that suggest that treatment with the SRP may result in differential selective potential compared with the liquid AS formulation. Determination of fluridone residues following SRP treatments under various field conditions will help with improved recommendations for spot treatment of nuisance vegetation and improved species-selective control.

While the release of fluridone from the SRP matrix has been described in the laboratory under short-term agitation (400 hr) (Mossler et al. 1993; Netherland et al. 1994), long-term release remains poorly understood, especially in the static conditions likely to be observed under a plant canopy. In cases where block treatments have failed with the SRP, it is unknown whether the application rate and subsequent release rates from the SRP were too low to allow a threshold concentration to be achieved or if threshold concentrations were achieved but not maintained for an adequate exposure due to dispersion from the treatment area. Moreover, species selectivity observed following SRP treatments remains largely unexplained. A variety of factors including sediment type, temperature, application rate, and water flow likely contribute to variability observed in release rates. However, very little research exists to demonstrate these impacts. One factor that has limited research in this area has been costs and time associated with analyzing large numbers of water samples for fluridone.

Recently an enzyme-linked immunoassay (ELISA) called the FasTEST that detects fluridone down to a level of 1 µg/L has been developed by the SePRO Corporation. This assay allows for rapid and accurate screening of large numbers of samples. High performance liquid chromatography (HPLC) has been the standard technique to analyze water for fluridone content. While the HPLC method is generally considered quite accurate, the turn-around time for sample analysis is generally 2-4 weeks depending on the number of samples to be analyzed. In addition, most laboratories required a minimum number of samples due to the preparatory time required. Development of the ELISA technique now

allows for rapid return of results (24–48 hr) and can help aquatic plant managers determine if the levels of fluridone in the water are sufficient or if additional application is needed. Although the ELISA technique is considered to be accurate, field confirmation using the standard HPLC method will increase confidence in the results.

Sonar SRP release rates under a variety of field conditions need to be quantified to allow for use-rate recommendations that provide consistent target plant control under a variety of field conditions. Therefore, a pond study was initiated to compare the rate of fluridone dissipation in small static ponds infested with hydrilla following liquid (Sonar AS) and SRP applications. Water samples were analyzed using both the FasTEST and HPLC methods to compare results. Determination of the level of hydrilla control provided by the AS and SRP treatments, potential for plant recovery, and treatment impacts on subsequent tuber sprouting were also intensively monitored.

## **Materials and Methods**

On June 20, 1996, three ponds approximately 0.2 ha in size and 1.2 m in depth were selected for fluridone treatment at rates of 0 µg/L, 150 µg/L using the AS formulation, and 150 µg/L using the SRP formulation. Pond dimensions were measured and water depths estimated by sampling 20 sites and taking the average to determine the volume of water to be treated. Sonar AS and SRP were applied evenly to the water surface at rates calculated to achieve 150 µg/L. Sediment temperature and DO (at the sediment/water interface) were monitored with Hydrolabs at 6-hr intervals throughout the study.

### **Aqueous fluridone residues**

Water samples were collected in 250-ml amber bottles at 2 and 6 hr and 1, 2, 4, 6, 9, 12, 15, 18, 21, 24, 27, 33, 39, 48, 57, 76, 97, 115, 135, 148, 161, 167, 198, and 224 days. Three replicate samples were collected from middepth in each pond. Residue data from the AS and SRP treatment were subjected to linear regression using the highest aqueous concentration measured as the initial point. While this was 1 DAT for the AS treatment, the maximal concentration for the SRP was not achieved until 15 DAT.

### **Comparison of HPLC and FasTEST analyses**

Following sample collection in the 250-ml bottles, 50 ml of water was transferred to 60-ml amber bottles to allow for comparison between two analytical procedures. Bottles were stored frozen prior to shipment to SePRO, Carmel, IN, for FasTEST analysis or to the Lewisville Aquatic Ecosystem

Research Facility, Lewisville, TX, for HPLC analysis. The analytical procedures were compared using regression analysis.

### **Treatment efficacy and recovery potential**

Pretreatment hydrilla biomass was collected from nine random sites in treated and untreated ponds using 0.5-m<sup>2</sup> frames, and the condition of the plants and percent coverage was noted each time water samples were collected. Shoot biomass was separated according to species, dried to a constant weight (65 °C for 48 hr), and a dry weight was recorded. Posttreatment biomass samples were collected from nine random sites in each pond at 33, 57, 97, 115, 167, and 224 DAT. Data were analyzed by ANOVA, and means at each sample date were compared with untreated control ponds using a Dunnet's test at the 0.05 level of significance.

Forty apical shoots of hydrilla were removed from the fluridone-treated ponds and an untreated control pond at 33-, 57-, 97-, and 115-day posttreatment and transferred to 20, 2-L sediment-filled pots to assess potential for plants to recover. Ten pots containing two apical shoots were then placed in 1,000-L concrete vaults in untreated water and allowed a 35-day recovery period. Biomass production of hydrilla from all three treatments was analyzed by ANOVA and means compared using Fisher's Least Significant Difference test at  $\alpha = 0.005$ . The remaining 10 pots were harvested on May 5, 1997, to quantify tuber production following various fluridone concentrations and exposure times.

## **Results and Discussion**

### **Aqueous residues**

Fluridone concentrations from the two treatments are shown in Figure 13. Values represent the average of three replicate samples from each pond. Regression of data from the AS treatment (1 DAT thru 223 DAT) resulted in a calculated fluridone half-life of 26.6 days ( $r^2 = 0.94$ ). Regression of data from the SRP treatments (the peak at 15 DAT was used as the initial concentration) resulted in calculated fluridone half-lives of 138.6 days ( $r^2 = 0.77$ ). Although a true half-life for the SRP formulation cannot be calculated because the fluridone concentration measured represented both dissipation from the water and continued release from the granule, the calculated value represents a good estimate of concentration of fluridone present in the water column at any given time. The marked differences in calculated half-lives between the AS and SRP suggest that the SRP formulation was continuing to release fluridone from the pellet long into the treatment. Moreover, the relative lack in degradation between 100 and 225 days suggests that release rates were essentially equal to degradation rates during this time. Langeland and Warner (1986) also observed distinct differences in maximal fluridone residues and subsequent dissipation following pond applications of Sonar AS and a clay formulation (Sonar 5P).

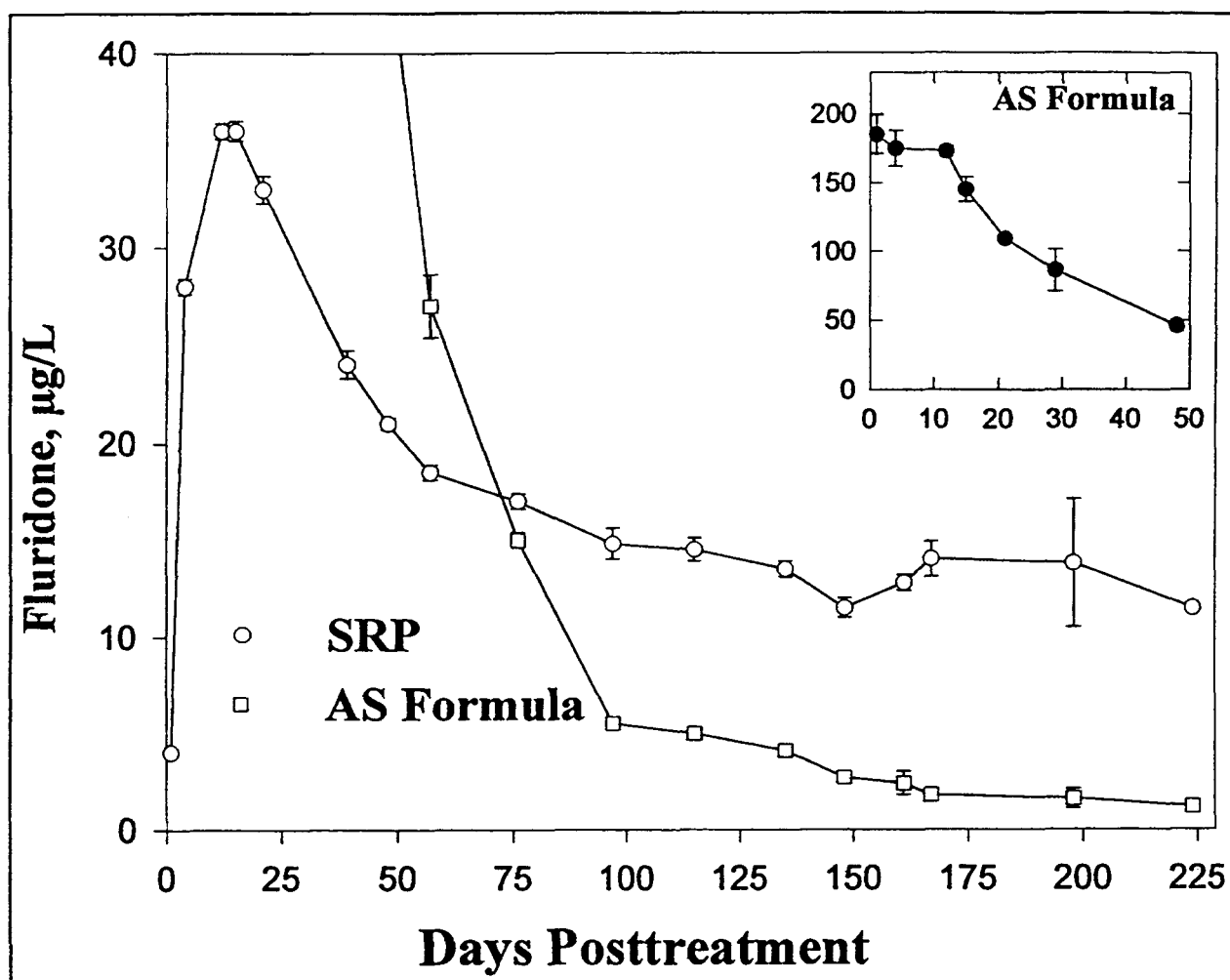


Figure 13. Fluridone degradation over time following treatment of ponds at a target rate of 150 µg/L with the SRP formulation and the liquid AS formulation (each point represents the mean of three samples ( $\pm 1$  SE). Half-lives were calculated using the time at which the peak aqueous concentration was observed as the initial data point)

Temperatures at the sediment/water interface remained quite stable from June through September due to the continued presence of a vegetative canopy, dropped slightly from October through January, and increased again in February (Table 10). In contrast, daily surface temperatures often fluctuated between 28 and 43 °C during June through September. Dissolved oxygen levels at the sediment/water interface never exceeded 0.5 mg/L from June through October, but did increase up to 1 mg/L in some areas during the winter months. Due to the fact that sediment temperatures did not change appreciably during the first 4 months of treatment, initial effects of temperature on release from the SRP pellets was not discernable. However, it should be noted that in more temperate areas of the country, sediment temperatures ranging from 5 to 10 °C would be quite common and may impact release from the SRP formulation following early spring and fall treatments.

**Table 10**  
**Monthly Sediment Temperatures (minimum, maximum, and mean)**  
**Measured Following Fluridone SRP Treatment of Ponds**

Month	Sediment Temperature, °C		
	Min	Max	Mean
June	21	25	23
July	22	26	23
August	21	27	22
September	19	23	21
October	18	23	21
November	17	21	19
December	16	20	19
January	14	19	18
February	15	22	20
March	17	22	21

### Comparison of HPLC and FastEST analyses

Residue analyses showed good agreement between the HPLC and FastEST analytical procedures at both high and low fluridone concentrations throughout the study (Figure 14). However, the HPLC analyses consistently predicted slightly higher concentrations compared with the FastEST, suggesting a bias between the procedures. This trend requires confirmations using standard quality assurance/quality control procedures. Due to the high level of fluridone activity at exceedingly low use rates, accurate and precise reporting of aqueous residues is critical. However, based on the slight differences noted between the analytical procedures in this study, the FastEST should provide a viable management tool allowing fast and accurate prediction of fluridone.

### Treatment efficacy and recovery potential

Although initial fluridone concentrations were markedly different between AS and SRP treatments, effects on the vegetation were similar. Hydrilla biomass in the treatment ponds decreased slightly 57 days into the treatment (Figure 15). By 87 DAT, several stems were still noticeable, but biomass had decreased by approximately 75 percent compared with pretreatment levels. At 115 days into the treatment, hydrilla biomass was reduced >90 percent. In both the AS- and SRP-treated ponds, the macro algae *Chara* spp. proliferated by 87 DAT ( $55 \pm 21$  g dry weight/m<sup>2</sup>). By the end of October (120 DAT), hydrilla was barely visible in the treated ponds, and *Chara* spp. continued to grow ( $135 \pm 33$  g dry weight/m<sup>2</sup>) and form a thick “carpet” at the bottom of the treated ponds.

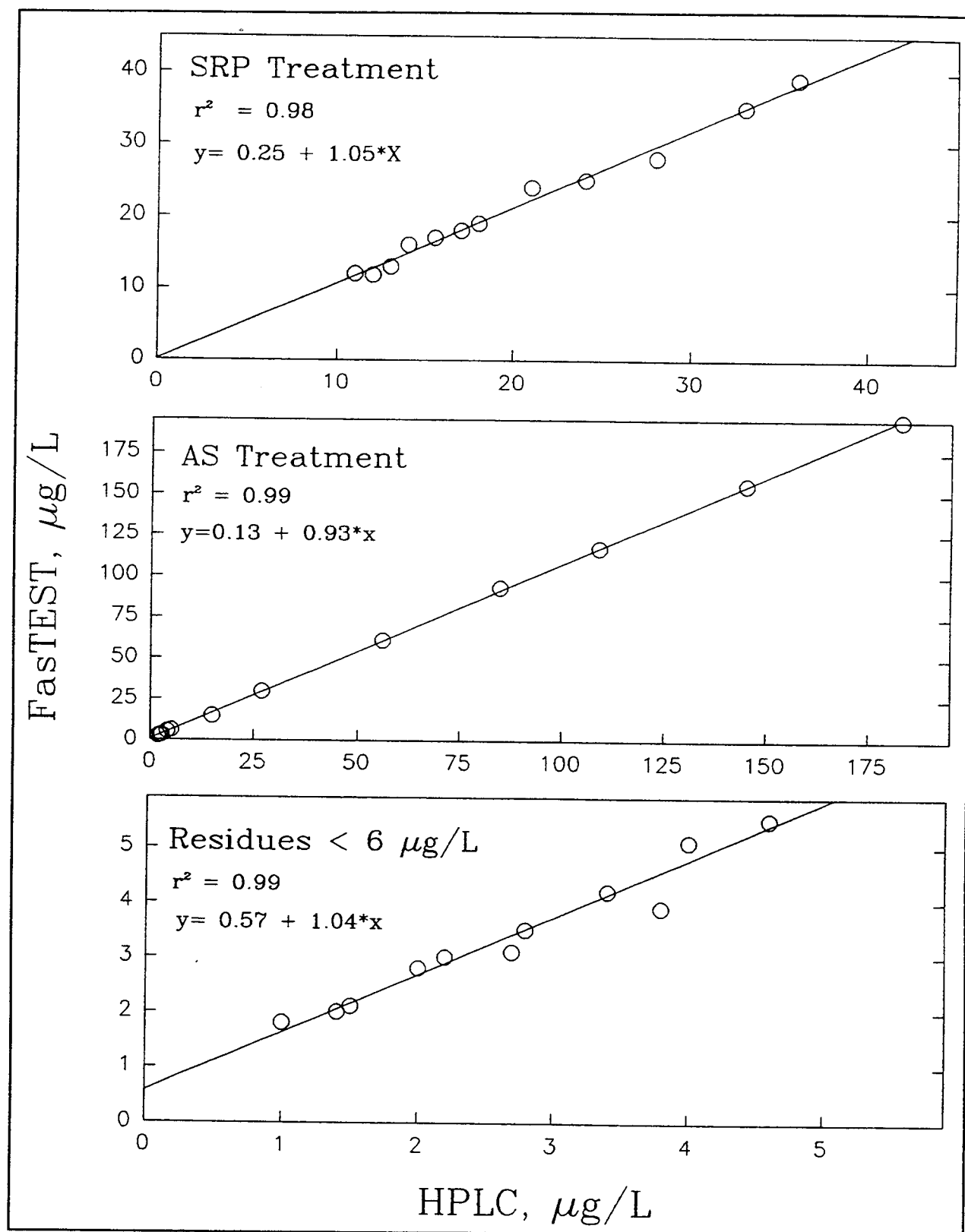


Figure 14. Relationship between fluridone concentrations analyzed using HPLC and the FastEST method



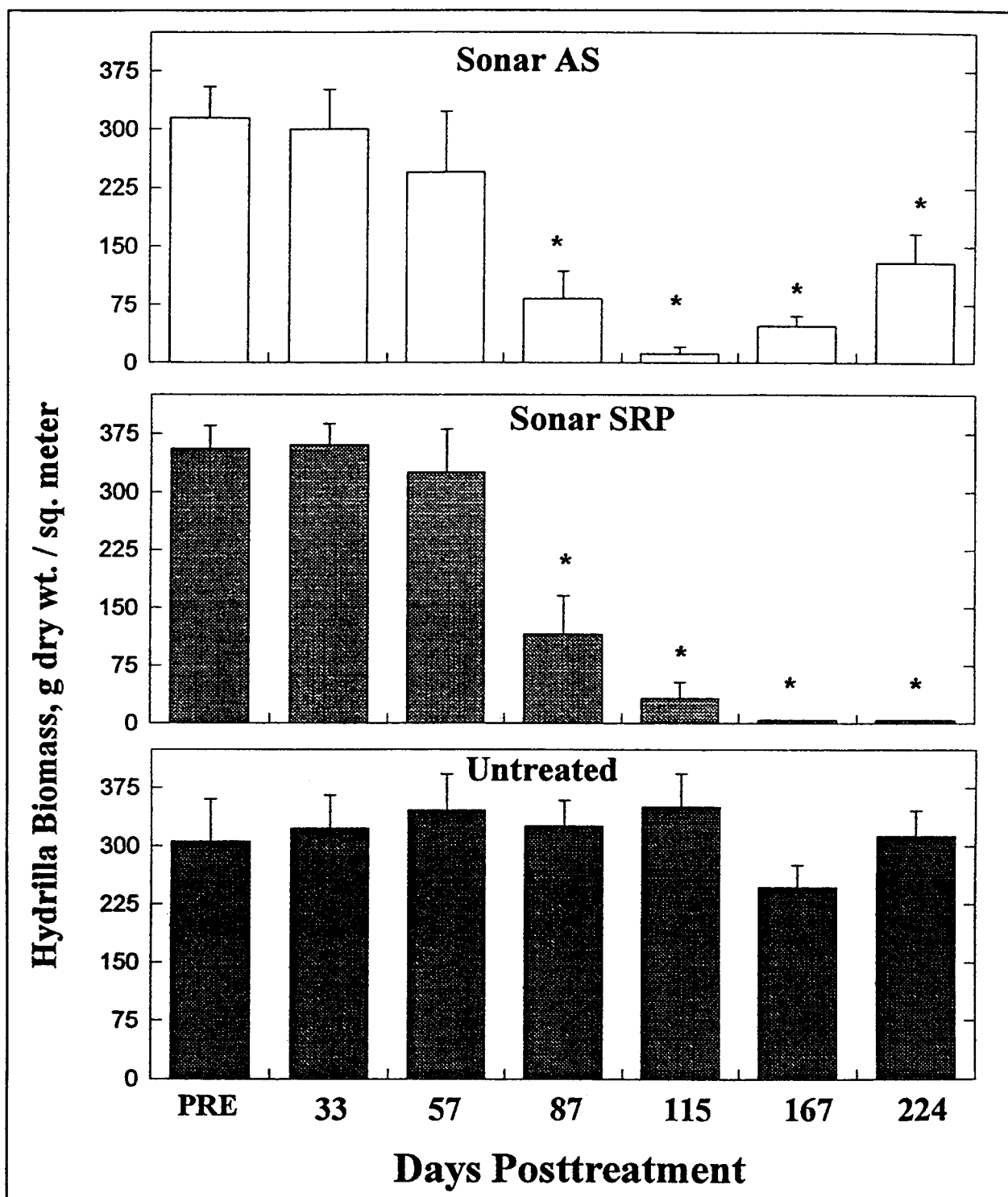


Figure 15. Dry weight biomass of hydrilla in fluridone-treated and untreated ponds through 224-day posttreatment (each bar represents the average of nine samples. Bars with an asterisk above indicate a significant difference between untreated and fluridone-treated ponds according to Dunnet's Test ( $\alpha = 0.05$ ))

Removal of apical tips from fluridone-treated water resulted in excellent regrowth potential of hydrilla at 35 days of recovery following all sampling dates (Figure 16). In addition, tuber production from the transferred tips measured in May of 1997 showed no differences in the number of tubers produced between fluridone-treated and untreated shoots (Table 11). These results support laboratory data from Netherland and Getsinger (1992, 1995a), which suggest that removal of hydrilla from fluridone exposure can result in rapid recovery of the plants. These data also support the hypothesis that an extended exposure period to fluridone is the key to achieving control of hydrilla.

During tuber sampling in early December (167 days into the treatment), some recovery of hydrilla in the AS ponds was observed. Numerous individual shoots of apparently healthy hydrilla were noted throughout the pond growing under the substantial vegetative mat formed by *Chara* spp. No hydrilla shoots were observed to have grown above the *Chara* spp. mats through December and January (in contrast to nearby copper- and endothall-treated ponds), suggesting that growth was inhibited by the continued presence of low levels of fluridone. No fluridone symptoms were noted on plants growing under the *Chara* spp. mat; however, Anderson (1981) has suggested that plants exposed to fluridone under low light intensities (such as under the cover of a vegetative canopy) may not exhibit significant fluridone injury until a sufficient light intensity is reached.

In February 1997 (224 days into the treatment), recovery of hydrilla from sprouted tubers resulted in a substantial biomass increase in the AS-treated pond, whereas, no viable hydrilla was found in the SRP-treated ponds. Continued high aqueous fluridone concentrations (11 µg/L) suggest that fluridone was inhibiting the development of newly sprouted tubers in the SRP ponds, while the lower concentrations in the AS ponds (1.2 µg/L) were not impacting sprouting tubers. The SRP pond results agree with those of Arnold (1979), who found that hydrilla from germinated tubers was controlled 80 to 100 percent for 7 to 11 months following a single fluridone application of 200 µg/L. Schmitz et al. (1987) found detectable concentrations of fluridone in the hydrosol up to 86-week posttreatment following a whole-lake treatment and have suggested that low fluridone residues in the hydrosol may also prevent reestablishment from tubers.

Rates of tuber sprouting following fluridone treatment remained very low and were similar to untreated ponds for up to 8 months following treatment (Figure 14). An increase in sprouted tubers was noted for all ponds during November. Some of these sprouted tubers survived in the AS ponds, but no survival was detected in the SRP-treated ponds. These results suggest that fluridone treatment does not increase tuber sprouting.

Mossler et al. (1993) have demonstrated that the substrate type (sand, clay, organic) may significantly affect aqueous concentrations of fluridone following release from SRP pellet. These laboratory studies involved agitation of treated beakers, and thus the release of fluridone still needs to be documented under variable sediment and flow conditions experienced in the field. The close proximity of the SRP and hydrosol could result in enhanced binding of fluridone with a subsequent decrease in aqueous concentrations. These observations have

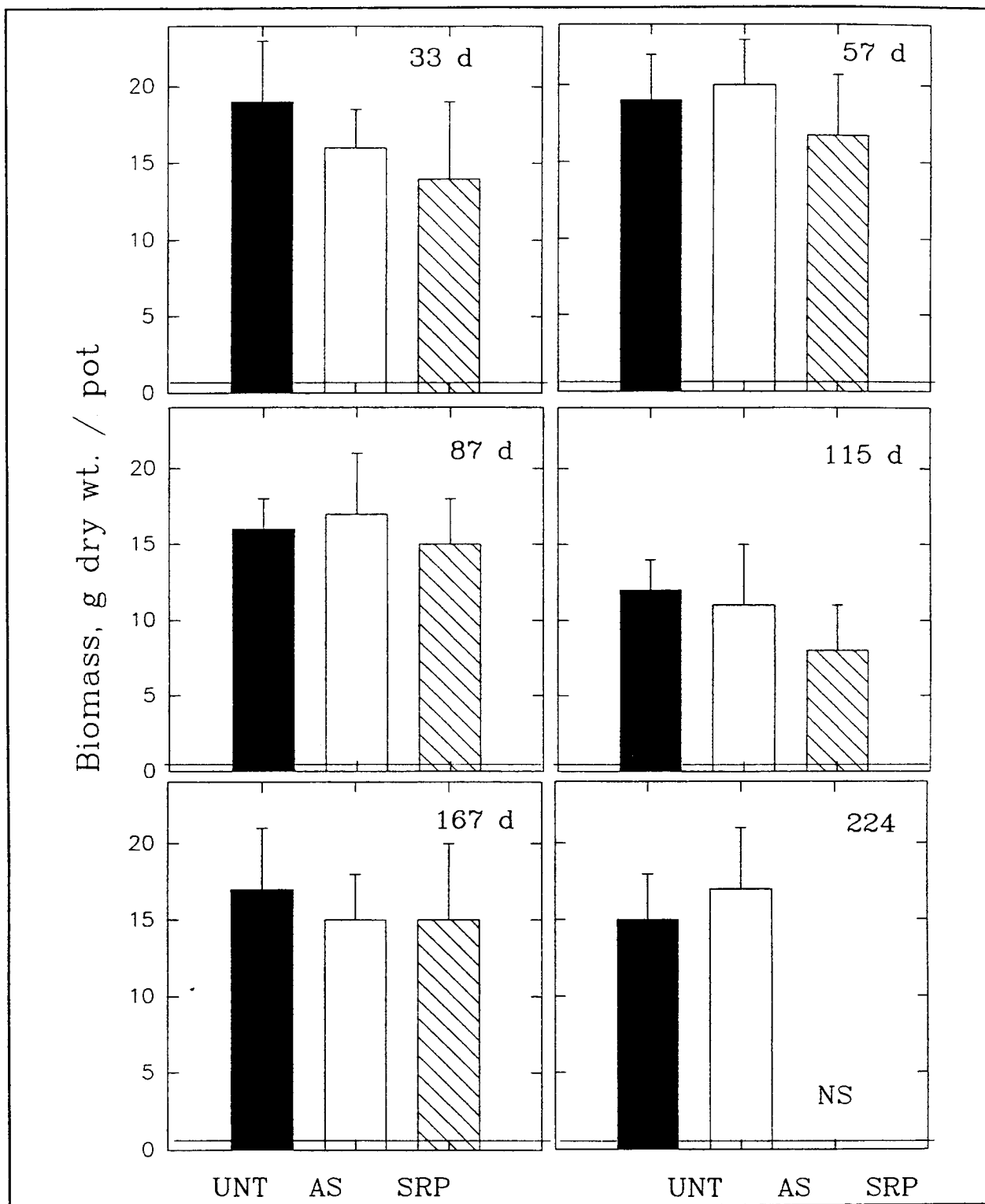


Figure 16. Dry weight biomass of hydrilla following removal of apical shoots at 33-, 57-, 87-, 115-, 167-, and 224-day posttreatment from fluridone-treated and untreated ponds (these shoots were transferred to 2-L sediment-filled pots, placed in untreated water, and given a 35-day recovery period. Dashed lines represent the initial shoot biomass placed in each growout tank. Each bar represents the mean of 10 pots ( $\pm 1$  SD))

**Table 11**  
**Hydrilla Tuber Production ( $\pm 1$  SD) Measured in May 1997 by Plants That Had Been Removed from Fluridone-Treated (150  $\mu\text{g/L}$  on June 20, 1996) and Untreated Ponds at 33-, 57-, 87-, 115-, and 167-Day Posttreatment and Replanted in 2-L Containers in Untreated Mesocosm Tanks**

Treatment	Exposure Days				
	33	57	87	115	167
	Number of Tubers Produced				
Untreated	56 (15)	75 (18)	49 (13)	47 (11)	40 (8)
Sonar AS	55 (12)	57 (14)	55 (11)	51 (15)	43 (12)
Sonar SRP	45 (10)	64 (10)	43 (12)	53 (9)	37 (7)

not been validated in the field following use of the SRP formulation. The ponds used in this study had relatively high fractions of sand and clay and were characterized by a relatively low percent organic matter content (3 to 5 percent). Provided the SRP pellets do not sink deeply into highly organic sediments, it is feasible that the slow continuous release of fluridone from these pellets could provide effective efficacy. Diffusive flux of fluridone in sediment interstitial water could also be a source of long-term fluridone residues in aquatic systems.

## Conclusions and Recommendations

### Conclusions

Based on the work conducted to date with the SRP formulation, the following conclusions are made:

- a.* Due to the controlled-release properties of the SRP formulation, initial treatment residues were significantly reduced compared with liquid treatments.
- b.* The controlled release properties of the SRP formulation also resulted in a greater than fivefold increase in the estimated fluridone half-life compared with the liquid AS treatments.
- c.* Good agreement between the HPLC and FastEST analytical procedures for fluridone analyses suggests the FastEST will provide a rapid and accurate tool for determining aqueous fluridone residues.

- d.* Initial hydrilla control was similar between the two treatments despite the fact that residues from the liquid application exceeded 100 µg/L for up to 33 days, whereas the maximum residues measured following the SRP treatment were below 40 µg/L
- e.* The long-term exposure (224 days) to low rates of fluridone following the SRP treatment provided complete hydrilla control and prevented germinating tubers from becoming established, whereas the reduction of aqueous fluridone residues below 1 µg/L at 167 days following the liquid AS treatment allowed rapid reestablishment of hydrilla from germinating tubers.
- f.* Hydrilla removed from fluridone-treated water and transferred to untreated water grew at the same rate as hydrilla collected from untreated water regardless of the fluridone rate or length of time the plants were exposed.
- g.* Rates of tuber sprouting in both fluridone-treated ponds remained very low and were similar to untreated ponds and ponds treated with other contact herbicides for up to 8 months, indicating that no stimulation of tuber sprouting occurred.

## Recommendations

Based on the work conducted to date with SRP formulation, the following recommendations are made:

- a.* Due to the widespread and increasing use of the fluridone SRP formulation as a treatment option, it is important to expand this research to understand how the SRP performs under various temperatures and sediment types in the field.
- b.* The SRP and liquid AS formulations should be compared for their selective potential to determine optimal application methods and rates that provide selectivity with these products.
- c.* Improved sampling techniques at the sediment/water interface and in sediment interstitial water are required to improve the understanding of fluridone distribution in the water column and in the sediment pore water after an SRP application. This is also true of other granular herbicide formulations.
- d.* Rigorous residue sampling of treatment areas following large and small partial treatments with the SRP will help explain both successes and failures with the SRP formulation and will help with improved recommendations for application rates and the size of the treatment area.

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# REPORT DOCUMENTATION PAGE

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<b>1. AGENCY USE ONLY (Leave blank)</b>		<b>2. REPORT DATE</b> July 1998	<b>3. REPORT TYPE AND DATES COVERED</b> Final report	
<b>4. TITLE AND SUBTITLE</b> Field Evaluation of Low-Dose Metering and Polymer Endothall Applications and Comparison of Fluridone Degradation from Liquid and Slow-Release Pellet Applications			<b>5. FUNDING NUMBERS</b>	
<b>6. AUTHOR(S)</b> Michael D. Netherland, David Sisneros, Alison M. Fox, William T. Haller				
<b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b> U.S. Army Engineer Waterways Experiment Station, 3909 Halls Ferry Road, Vicksburg, MS 39180-6199; U.S. Bureau of Reclamation, Denver Federal Center, 6th and Kipling, Building 56, Denver, CO 80225; University of Florida, Center for Aquatic Plants, 7922 NW 71st Street, Gainesville, FL 32653			<b>8. PERFORMING ORGANIZATION REPORT NUMBER</b> Technical Report A-98-2	
<b>9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)</b> U.S. Army Corps of Engineers Washington, DC 20314-1000			<b>10. SPONSORING/MONITORING AGENCY REPORT NUMBER</b>	
<b>11. SUPPLEMENTARY NOTES</b> Available from National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161.				
<b>12a. DISTRIBUTION/AVAILABILITY STATEMENT</b> Approved for public release; distribution is unlimited.			<b>12b. DISTRIBUTION CODE</b>	
<b>13. ABSTRACT (Maximum 200 words)</b> Field studies were conducted to evaluate three herbicide delivery system techniques. Metering pumps were used to apply low rates of endothall (0.4 mg/L) over a 72- to 96-hr period for control of sago pondweed in western irrigation canals. Treatments were evaluated for efficacy and feasibility to use under a variety of flow conditions. Treatments effectively controlled sago pondweed, and the development of a prototype metering pump greatly improved the feasibility of conducting treatments in remote settings where flow rates often vary greatly within a 24-hr period. In addition to metering technology, a new granular supersorbent polymer formulation of endothall that contains 61 percent active ingredient was evaluated in Lake Weohyapapka, Florida. These evaluations were conducted to compare efficacy and applicator handling properties versus the conventional clay formulation (10.1-percent active ingredient). The new formulation required 85 percent less bulk material than the conventional clay and presented no problems with dust creation. Although no differences in efficacy on hydrilla were noted between the products, reduced applicator exposure through decreased product handling and the time required for herbicide application were all seen as significant benefits of the new formulation. Lastly, the slow-release pellet (SRP) of the herbicide fluridone was applied to research ponds near Gainesville, FL, to improve the understanding of the release properties of this product. Following application of rates calculated to achieve 150 µg/L, the liquid aqueous suspension (AS) and SRP showed distinct differences in residues and dissipation. Initial concentrations following SRP application were reduced fivefold compared with the AS, whereas, the half-life of the SRP was estimated to be fivefold greater than that of the AS. Maintaining low residues for an extended period of time provided a full year of hydrilla control with the SRP, whereas the loss of threshold residue levels due to increased degradation rates of the AS allowed recovery of the hydrilla from tubers within 1 year posttreatment.				
<b>14. SUBJECT TERMS</b> Aquatic herbicide Chemical control Endothall			<b>15. NUMBER OF PAGES</b> 64	
			<b>16. PRICE CODE</b>	
<b>17. SECURITY CLASSIFICATION OF REPORT</b> UNCLASSIFIED	<b>18. SECURITY CLASSIFICATION OF THIS PAGE</b> UNCLASSIFIED	<b>19. SECURITY CLASSIFICATION OF ABSTRACT</b>	<b>20. LIMITATION OF ABSTRACT</b>	

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