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USE OF HERBICIDE/ADJUVANT FORMULATIONS FOR THE CONTROL OF *MYRIOPHYLLUM SPICATUM* L.

by

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Adjuvants (i.e. polymers, inverting oils, attaclay granules, controlled-release granules) were used in conjunction with the herbicides 2,4-D, diquat, and/or endothall to evaluate their effectiveness for the control of *Myriophyllum spicatum* L. in the Okanogan River and Lake Osoyoos, Wash. The formulations which reduced sparse to moderately dense *M. spicatum* colonies by over 75 percent in Lake Osoyoos were 2,4-D/attaclay granular (Aqua-Kleen) applied at
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20. ABSTRACT (Continued).

20 kg acid equivalent (a.e.) 2,4-D/ha (1.2 mg/ℓ), and 2,4-D/polymer applied at 33 kg a.e. 2,4-D/ha (1.8 mg/ℓ). The 2,4-D applied at a rate of 34 kg a.e. 2,4-D/ha (1.2-1.4 mg/ℓ) and used with both a polymer and an invert reduced dense *M. spicatum* colonies (i.e. >300 g/m^2 dry weight) by less than 40 percent in Lake Osoyoos; the primary reason was rapid growth of the underlying biomass. A diquat/polymer formulation applied at a rate of 27 kg a.e. diquat cation/ha (1.0 mg/ℓ) resulted in a 90-percent reduction of a dense *M. spicatum* colony (i.e. >300 g/m^2 dry weight). Endothall used with and without polymers and inverts did not effectively control (i.e., 0- to 65-percent reduction) moderately dense to dense *M. spicatum* colonies in the lake at application rates varying from 10 to 40 kg a.e. endothall/ha (0.5 to 1.6 mg/ℓ). Regrowth was evident in these plots.

Two controlled-release formulations designed to release low amounts of 2,4-D into the water for an extended time resulted in a 16- to 67-percent reduction of moderately dense to dense *M. spicatum* colonies. The 2,4-D/attaclay granular and the 2,4-D/polymer were applied to dense *M. spicatum* colonies at a rate of 45 kg (a.e.)/ha in flowing water (9 to 15 cms) resulting in a 78- to 80-percent reduction of the treated plants (results of the 2,4-D/attaclay granular may be biased due to interplot contamination). A 2,4-D/invert formulation applied to a dense *M. spicatum* colony in flowing water (i.e. 9 cms) at a rate of 12 kg a.e./ha resulted in a 40-percent reduction in the treated colony.

Persistence of waterborne herbicide residue ranged from 2 hr in flowing water to 2 weeks in nonflowing water. All waterborne herbicide residues were below 0.1 mg/ℓ 24 hr after treatment. Dispersal of the herbicide was evident in all river treatment plots. The invert formulations provided a higher and more sustained herbicide level on the *M. spicatum* plants than the granular or polymer formulations (i.e. 540 μg/ℓ 2,4-D in the invert plot *M. spicatum* plants 32 days posttreatment). Some 2,4-D was detected in the sediment up to 32 days in the 2,4-D/attaclay granular plot while the 2,4-D in the polymer and invert plots persisted for only 8 days. No changes in water quality were observed in response to treatment. However, water velocity was reduced by over 50 percent in the dense *M. spicatum* colonies relative to the velocity outside the colony.

PREFACE

This study was conducted by the US Army Corps of Engineers Aquatic Plant Control Research Program (APCRP). The majority of funds for the effort were provided by the US Army Engineer District, Seattle. Additional funds were provided by the Office, Chief of Engineers (OCE), under Department of Army Appropriation No. 96X3122, Construction General, 902740, through the APCRP at the US Army Engineer Waterways Experiment Station (WES), Vicksburg, Miss. The OCE Technical Monitor was Mr. Dwight L. Quarles. Assistance in conduction of the field work was provided by the Bureau of Reclamation.

The work was initiated in 1979 under the general supervision of Dr. John Harrison, Chief, Environmental Laboratory (EL), and Dr. C. J. Kirby, Jr, Chief, Environmental Resources Division (ERD), and under the direct supervision of Mr. J. K. Stoll, Chief, Environmental Analysis Group, all of WES. Mr. J. L. Decell was Manager, APCRP, at WES.

The principal investigator in 1979-1980 was Mr. A. M. B. Rekas and in 1981-1982, Mr. K. Jack Killgore. This report was written by Mr. Killgore. Dr. Barry S. Payne of ERD and Dr. Howard E. Westerdahl of the Ecosystem Research and Simulation Division aided in implementation of the study and reviewed this report. Project directors at the Seattle District were Mr. R. M. Rawson and Mr. D. R. Bailey. Mr. Ron Pine, Washington State Department of Ecology, provided State coordination.

Commanders and Directors of the WES during the conduct of this study were COL John L. Cannon, CE, COL Nelson P. Conover, CE, and COL Tilford C. Creel, CE. Technical Director was Mr. F. R. Brown.

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USE OF HERBICIDE/ADJUVANT FORMULATIONS FOR THE CONTROL OF *MYRIOPHYLLUM SPICATUM* L.

PART I: INTRODUCTION

Background

1. The Aquatic Plant Control Research Program (APCRP) of the US Army Engineer Waterways Experiment Station (WES) in cooperation with the US Army Engineer District, Seattle, conducted a Large-Scale Operations Management Test (LSOMT) from 1979-1982 to evaluate the concept of prevention as an operational technique for managing *Myriophyllum spicatum* L. (Eurasian watermilfoil) in the Columbia River Drainage System. From 1980-1982, various chemical treatment techniques were evaluated as part of the LSOMT. A consortium of agencies contributed to the 1982 study including: Seattle District; US Army Engineer District, Walla Walla; US Army Engineer District, Portland; US Department of the Interior, Bureau of Reclamation, Engineering and Research Center; Douglas, Chelan, Grant, and Okanogan Washington State Public Utility Districts; Okanogan and Grant Counties, Washington State; and the Grant County Noxious Weed Board.

2. *Myriophyllum spicatum* is a submersed aquatic macrophyte that has become a problem in the Pacific Northwest. In 1970, *M. spicatum* was found in a main stem lake of the Okanogan River (a tributary of the Columbia River) in British Columbia (Aiken, Newroth, and Wile 1979), and, by 1980, it was identified in the Columbia River (Rawson 1982). The establishment and subsequent proliferation of *M. spicatum* has created adverse impacts on the multiple uses of this drainage system (Newroth 1974; Aiken, Newroth, and Wile 1979). Because of the present and potential water-use impacts associated with *M. spicatum*, a comprehensive effort of research and operational program development was undertaken to evaluate herbicide control techniques.

3. The selection of the proper herbicide application technique is essential for obtaining cost-effective control of the aquatic plant as well as ensuring that the herbicide application is compatible to both water uses and other aquatic organisms. The combined use of adjuvants* and herbicides

* An adjuvant is considered an additive chemical that intensifies or modifies a herbicide's effectiveness.

can enhance application techniques. Adjuvants can selectively place the herbicide onto the target aquatic plant, reduce dispersal of the herbicide away from the application site, and possibly allow the applicator to reduce the amount of herbicide required for effective control (Gates 1972; Baker et al. 1975; Bitting 1974; Wortley 1977).

Purpose and Scope

4. The purpose of this study was to determine if selected adjuvants could be used with conventional herbicide formulations to effectively control *M. spicatum* in both lentic (i.e., still water) and lotic (i.e., moving water) habitats of the Columbia River Drainage System. The results of this study will be used by the Seattle District to help develop their aquatic plant management program for this drainage system. It must be recognized that the effectiveness of herbicide/adjuvant formulations will, to a large extent, depend on using proper herbicide/adjuvant ratios and herbicide applicators experienced in adjuvant applications.

Objectives

5. The objectives of this study were:

a. Determine the extent and duration of *M. spicatum* control using various herbicide/adjuvant application rates.

b. Monitor herbicide residue within the areas treated.

Study Approach

6. The initial emphasis of this research in 1980 and 1981 was to evaluate different herbicide/adjuvant formulations in lentic habitats. A proven effective herbicide (i.e., 2,4-D)/adjuvant formulation was chosen based on these studies for application in lotic conditions. Specifically, 2,4-D, diquat, and endothall were applied with a polymer in 1980. Various application rates of endothall were applied with inverting oils in 1981. The results of both studies were subsequently evaluated to design and implement the 1982 study (i.e., the use of 2,4-D/adjuvants in lentic and lotic conditions).

PART II: SITE DESCRIPTION

Description of Study Site

7. The study site for this investigation included Lake Osoyoos and the Okanogan River located in Okanogan County, Washington (Figure 1). The

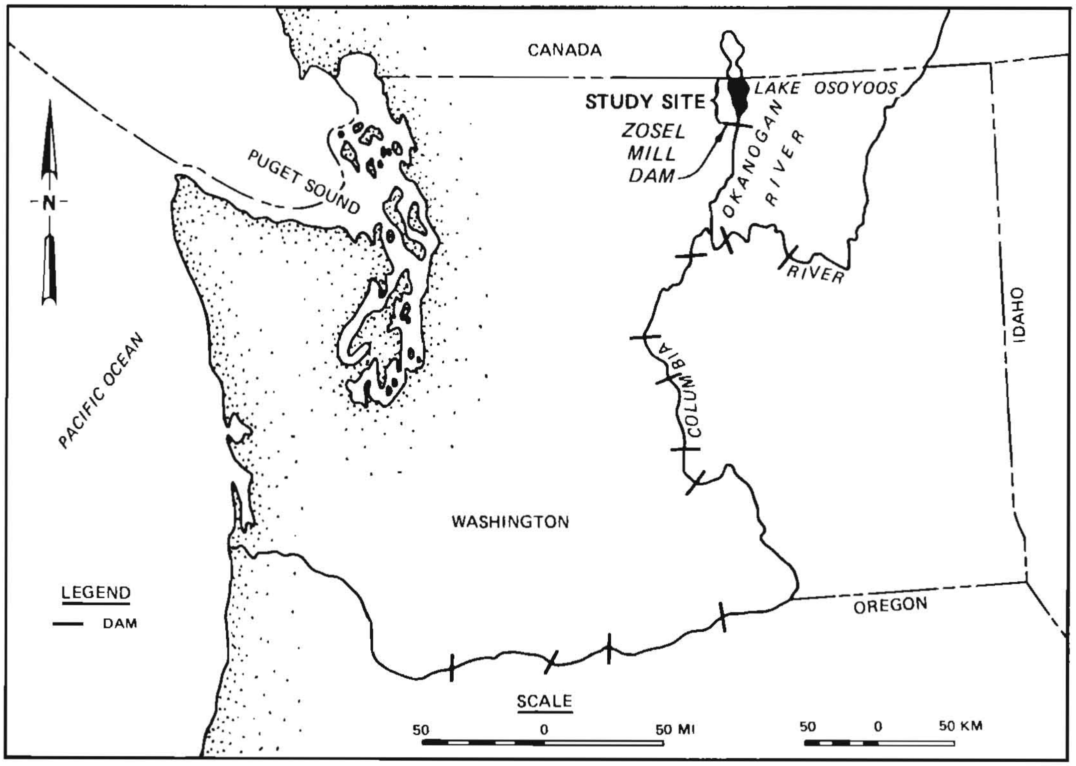

Figure 1. Location of herbicide/adjuvant study site

Okanogan River originates in British Columbia and flows south into Washington where it empties into the Columbia River (313 km). Four lake basins were formed along the Okanogan River during the most recent advance and retreat of glaciers. The southernmost basin contains Lake Osoyoos. Lake Osoyoos is a 2318-ha dimictic lake located on the United States-Canadian border (823 ha in the United States). The mean depth is 14 m and the maximum depth is 64 m. Lake Osoyoos is characterized by a rapid flushing rate (0.7 years) (Pinsent et al. 1974). The Okanogan River study site was approximately 24 ha between Zosel Mill Dam (river mile 77.4) and the downstream end of Lake Osoyoos (river mile 79.0). Daily discharges recorded (October 1942 to present) at the US Geological Survey (USGS) gaging station designated as "Okanogan

River at Oroville, Washington" (river mile 77.3) were: maximum, 106 cms; mean, 19 cms; minimum, -77 cms (reverse flow due to backwater effect). Mean water depth ranged from 1.5 to 2.0 m. Both Lake Osoyoos and the Okanogan River have moderately hard water with little suspended solids. Water quality measurements taken during the study are summarized in Appendix A.

Phenology and Growth Characteristics of *M. spicatum*

8. Colonies of *M. spicatum* in Lake Osoyoos typically occur from the periphery of flat terraces to near shoreline areas in water depths 0.5 to 5 m, and in the deeper (1 to 3 m) main channel in the Okanogan River. *Myriophyllum spicatum* rarely grows in waters less than 0.5 m deep in either Lake Osoyoos or the Okanogan River. This could be due to ice damage of *M. spicatum* during the winter. The effects of ice damage are greatest at the shallow areas (Adams and McCracken 1974). The floral spikes of *M. spicatum* develop during two periods: (a) late June, and (b) late July to early August. Patten (1954) and Newroth (1974) also reported that established *M. spicatum* colonies characteristically have two flowering periods. Autofragmentation occurs after each flowering period, the most extensive being in late August to early September. The stems grow to the water surface by late June and then laterally to form a dense "canopy" in the upper 30 to 50 cm of the water column. Most of the photosynthetic tissue of *M. spicatum* typically becomes localized as a canopy in the top 30 cm of the water column (Adams, Titus, and McCracken 1974). Yellowish to blackish-brown deposits (i.e., marl and periphyton) form on the stems and leaflets of *M. spicatum* in Lake Osoyoos while these deposits are rarely observed in the river. Senescence occurs in late September to early October, resulting in the loss of most of the standing crop. However, *M. spicatum* was observed below ice in Lake Osoyoos.

Aquatic Plant Community

9. *Myriophyllum spicatum* was the dominant aquatic plant species in the study sites. *Scirpus validus, Potamogeton crispus, P. pussillus, P. pectinatus, Elodea canadensis, Certophyllum demersum, Nitella* sp., *Nuphar luteaum*, and *Nymphea odorata* (the latter two found only in the Okanogan River) occurred in the *M. spicatum* colonies. Together these aquatic plants

(excluding *M. spicatum*) comprised approximately 5 percent of the total aquatic plant community in the study sites while some dominated areas adjacent to the study sites. Table 1 shows the areal distribution of submersed aquatic plants (primarily *M. spicatum*) from 1979-1982 relative to the east and west side of Lake Osoyoos and the Okanogan River. The distribution was interpreted from color aerial photography (i.e. S0397 film used with HF3 and HF4 Wratten filters) at a scale of approximately 1:10,000 in 1979 and 1:5,000 from 1980-82. Three years of ground truth data were used in the photointerpretation. The areal determinations were made with an electronic digitizer (see Leonard and Payne 1984).

PART III: METHODS AND MATERIALS

Herbicide Formulations

10. The herbicide formulations, application rates, and application equipment used from 1980-1982 are listed in Table 2. All applications were made from an airboat during the month of July. The liquid formulations were applied below the water surface through 30- to 80-cm-long trailing hoses. A fluorescent dye was added to some liquid herbicide formulations to allow visual tracking of herbicide dispersal.

11. The polymer used was Nalquatic. Nalquatic is a high molecular weight polycarboxylate polymer with selected solvents and surfactants. With the addition of water and herbicide, the polymer is hydrated to a visco-elastic mixture which, on application, physically "shrouds" the herbicide onto the target aquatic plant.

12. Two inverts were used: 403 inverting oil and Spra-mate invert emulsion. The invert formulations consist of an emulsion of water, liquid herbicide, oil (solvent), and emulsifiers. The emulsifiers allow the oil to encapsulate the herbicide/water solution to a controlled size droplet resembling "snowflakes" which will adhere to the target plant upon application. The invert emulsion will not break the water surface tension; thus, it must be applied below the water surface. Spra-mate invert emulsion requires addition of the solvent (usually xylene) while 403 inverting oil is premixed with the solvents (i.e. xylene and d'limonene).

13. The conventional herbicides used were butoxyethenol ester of 2,4-dichlorphenoxyacetic acid (Aqua Kleen or 2,4-D BEE/granular), dimethylamine salt of 2,4-dichlorophenoxyacetic acid (Weedar 64 or 2,4-D DMA), dipotassium salt of endothall (Aquathol K or endothall), and diquat dibromide cation (Ortho diquat or diquat). Two controlled-release formulations (i.e. low concentrations of herbicide released over a prolonged period) were used: 2,4-D acid in Kraft lignin pellets (manufactured by Westvaco, Inc., North Charleston, S. C.) and poly GMA-2,4-D, an acrylic copolymer matrix embedded in clay pellets (manufactured by Wright State University, Dayton, Ohio).

Test Plot Establishment

14. The distribution of the test plots from 1980-1982 are shown in Figures 2, 3, and 4, respectively. Each test plot was permanently established using an electronic positioning system (AGNAV). The AGNAV allows positioning of a boat with field equipment over the same sampling sites during multiple visits to the same water body (Killgore and Payne 1984).

Data Collection Techniques

15. A summary of the types of data collected and the sample frequency from 1980-1982 is shown in Table 3. The instrumentation used in data collection is shown in Table 4. Sample point configurations for the test plots are shown in Figures 2-4.

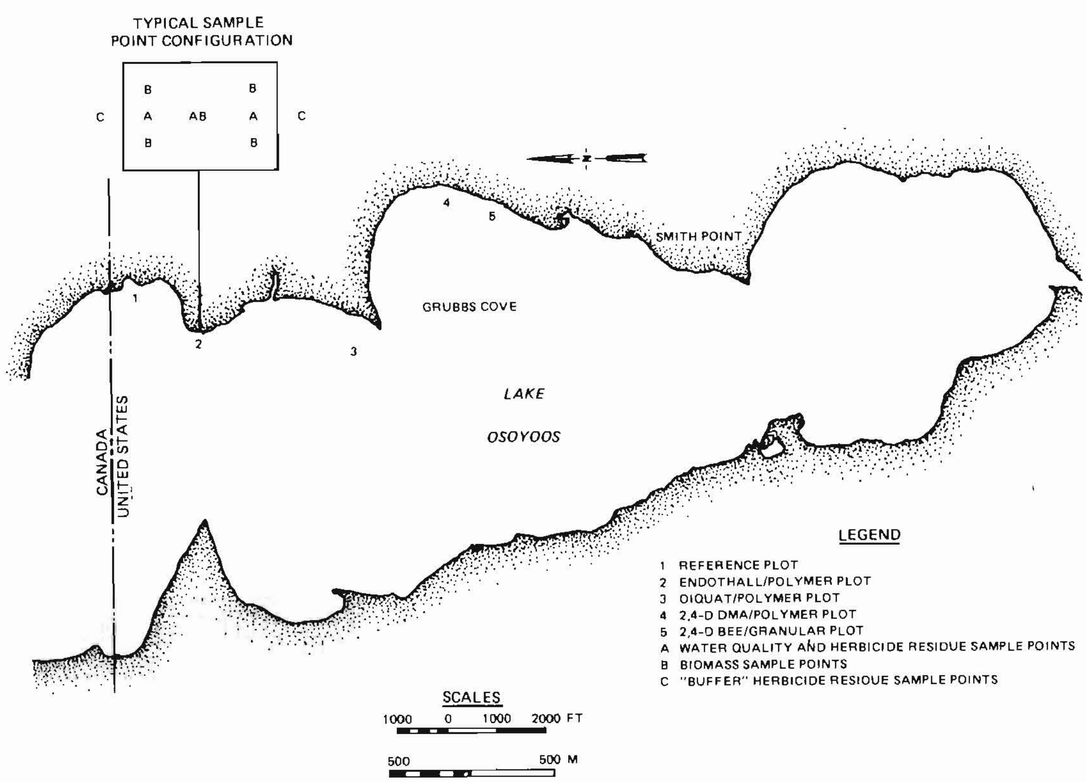

Figure 2. Plot distribution and sample configuration for 1980

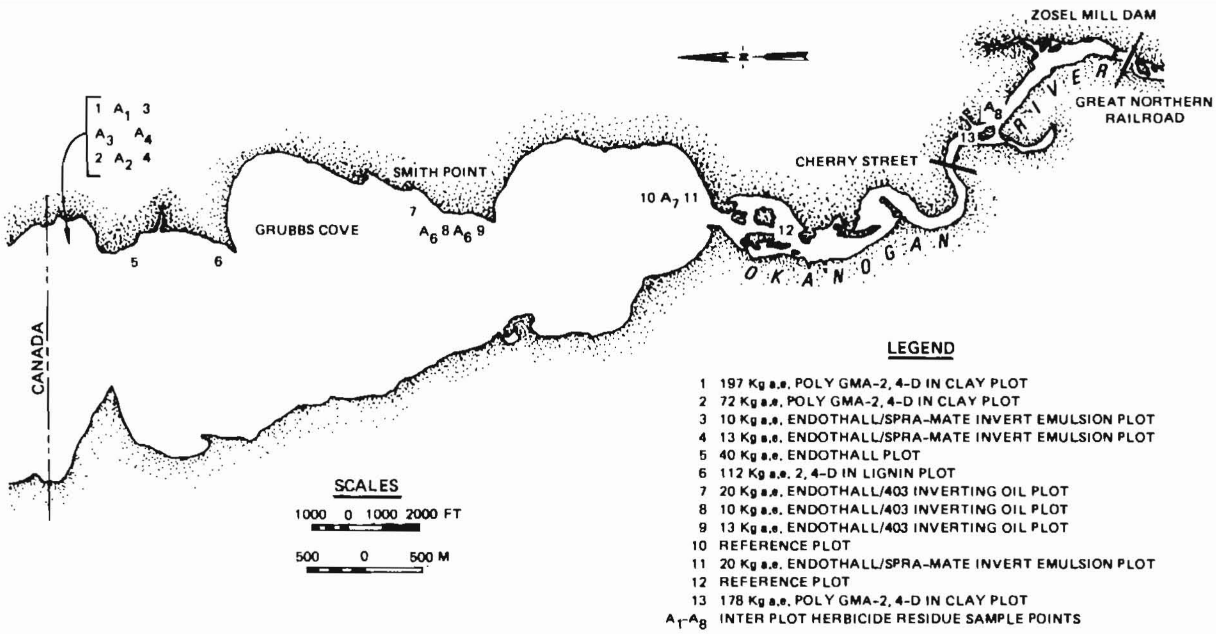

Figure 3. Plot distribution and sample configuration for 1981

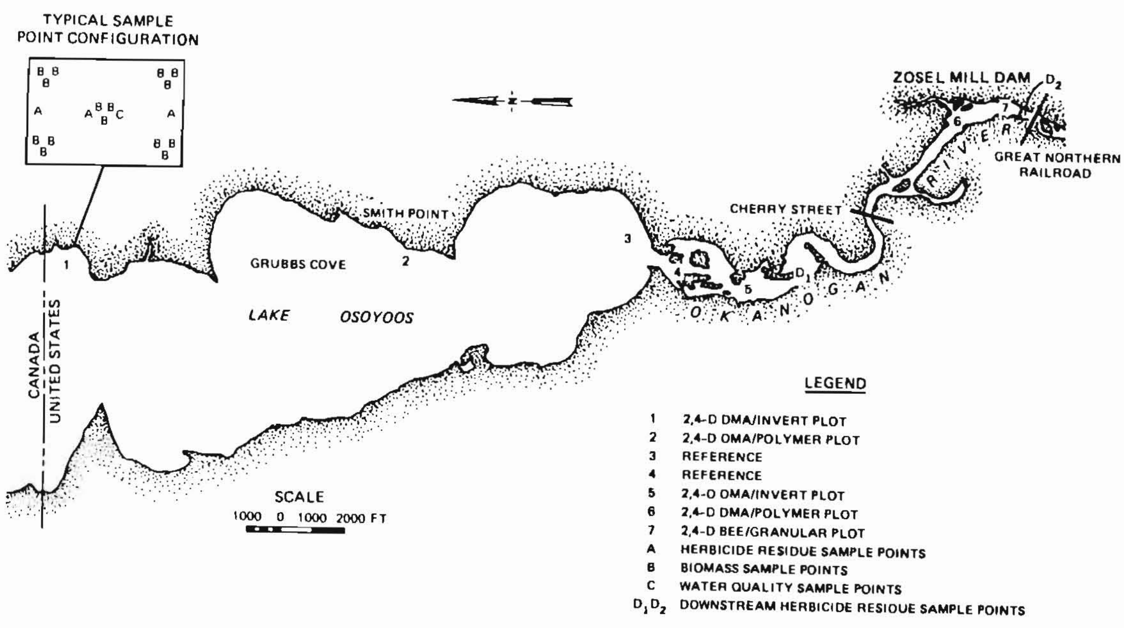

Figure 4. Plot distribution and sample configuration for 1982

16. Treatment effectiveness was quantified by measuring changes in plant biomass and/or height over time. Biomass samples were collected with a hydraulically operated sampler at five permanent points within each plot in 1980. In 1982, each plot was divided into five quadrants and three biomass samples were obtained in each quadrant (a total of 15 samples per plot). Biomass samples were placed in plastic bags and sent to a laboratory for weight measurements. Biomass samples were not taken in 1981.

17. The AGNAV and a Raytheon DE-719 fathometer were used to measure plant height in 1981 and 1982 (Killgore and Payne 1984). Straight-line transects were positioned over the plot and the *M. spicatum* colonies using the AGNAV. While traveling over each transect, event marks were put on the fathometer paper at equal intervals. By using the AGNAV positioning system, fathometer tracings were taken over the same transects during multiple visits to the same plots.

18. Herbicide retention and dispersal were monitored from samples collected within and outside the treatment areas at selected time intervals. In 1980, herbicide residue measurements were collected at the surface, 1 m below the surface, and 0.5 m above the bottom sediment at each sampling point. In 1981 and 1982, each waterborne herbicide residue sample was a composite taken at 0.2 and 0.8 of the depth at each sampling point. "Buffer zones" or herbicide dispersal sampling points were established outside of each plot to monitor herbicide dispersal and/or cross-contamination during all 3 years (see Figures 2-4). Water circulation was evaluated in the Okanogan River in 1982 prior to herbicide application using the fluorescent dye Rhodamine WT. The purpose was to determine 2,4-D dispersal stations and sample frequency which would optimize the probability of detecting maximum 2,4-D concentrations.

19. Herbicide water samples were stored in acid-washed glass bottles with teflon-lined caps and acidified to pH 2 by adding H_2SO_4. Sediment samples were also stored in acid-washed glass bottles with teflon-lined caps. Aboveground *M. spicatum* plants were placed in plastic bags. All samples collected for herbicide analysis were packed in ice and sent to the laboratory. Herbicide analysis techniques and recovery rates are shown in Appendix B.

Data Analysis

20. The mean and standard deviation were determined for the herbicide

residue, water quality, and biomass values for each plot for each sampling date. The fathometer tracings in each plot were interpreted for plant height and water depth at each event mark and a mean was obtained for each sampling date. In 1980, wet weight, dry weight, and ash-free dry weight were determined for all biomass samples. Least squares linear regressions of both wet-to-dry weight and wet-to-ash-free dry weight were calculated from the 1980 biomass values (Appendix C). The regression equations were subsequently used to determine dry and ash-free dry weights of *M. spicatum* biomass samples taken in 1982.

PART IV: RESULTS

1980

Biomass

21. Mean biomass values sampled throughout the study are shown in Table 5. The diquat/polymer plot had the highest pretreatment biomass density (i.e. 331.1 g/m^2 dry weight) followed by the reference plot, 2,4-D DMA/polymer plot, endothall/polymer plot, and the 2,4-D BEE/granular plot. Changes in *M. spicatum* ash-free dry weight density for all plots 28 and 84 days after treatment are shown in Figure 5. The biomass decreased in all plots. However,

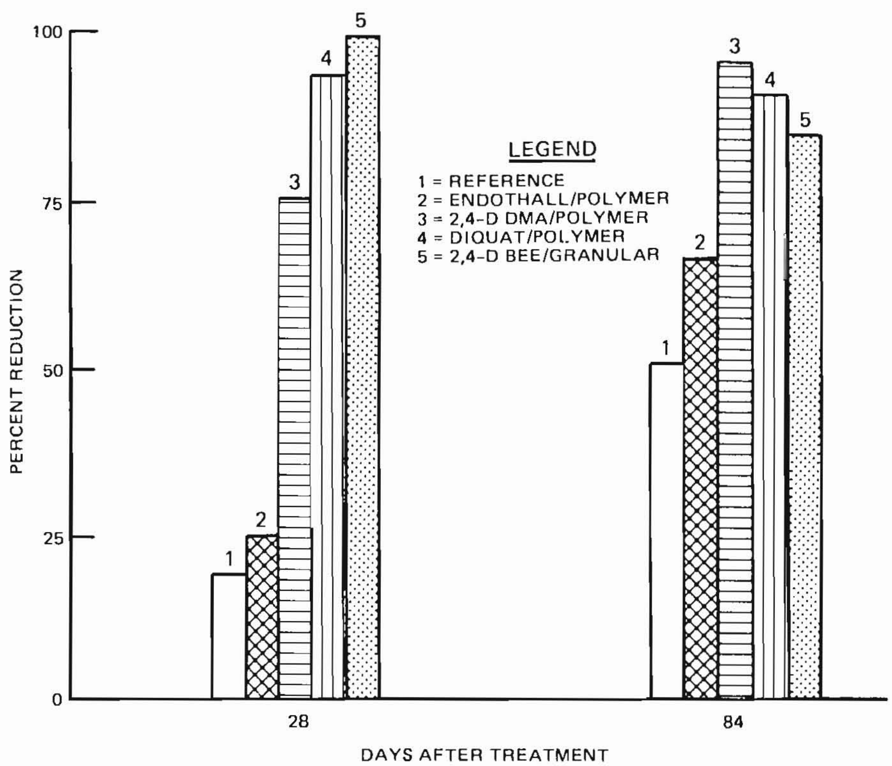

Figure 5. Percent change in *M. spicatum* ash-free dry weight 28 and 84 days after treatment for the 1980 treatment plots

biomass in the reference plot decreased only 18 percent 28 days after treatment while the endothall/polymer plot decreased 25 percent, the 2,4-D DMA/polymer plot decreased 75 percent, the diquat/polymer plot decreased 94 percent, and the 2,4-D BEE/granular plot decreased 91 percent. By 84 days after treatment (i.e. mid-October), the reference plot biomass decreased 50 percent (due to natural senescence during this time of year), endothall/polymer decreased 65 percent, 2,4-D DMA/polymer decreased 96 percent, diquat/polymer decreased 91 percent, and 2,4-D/granular decreased 86 percent. Substantial regrowth after treatment was evident only in the endothall/polymer plot.

Herbicide residue in the water

22. The mean waterborne herbicide residue values both within and outside of the treatment plots are shown in Table 6. Herbicide concentrations were detected in the plots only during the day of application; except for the diquat/polymer plot where the highest mean herbicide concentration was detected 14 days after treatment (i.e. 20 μg/ℓ). The highest mean herbicide concentration found in the 2,4-D BEE/granular was 20.8 μg/ℓ, 166.1 μg/ℓ in the 2,4-D DMA/polymer plot, and 40.0 μg/ℓ in the endothall/polymer plot. The buffer zone samples showed 2 to 173 times less herbicide concentration than samples within the plots. Herbicide was detected in the buffer zones 14 days after treatment in the diquat/polymer plot, 28 days after treatment in the 2,4-D DMA/polymer plot, and only during the day of treatment in endothall/polymer and 2,4-D BEE/granular plots. A mean endothall concentration of 2.1 μg/ℓ was detected in the reference plot the day of treatment.

1981

Plant height

23. The mean *M. spicatum* height and the mean water depth before and 28 and 56 days after treatment for all plots are shown in Table 7. Water depth gradually decreased during the study by 0.5 to 1.0 m. Figures 6 and 7 show the percent change in *M. spicatum* plant height 28 and 56 days after treatment for the endothall/invert and controlled-release plots, respectively. The height in both the lake and river reference plots increased 7 and 28 percent, respectively, 28 days after treatment; 56 days after treatment the plant height in the lake reference plot decreased 15.4 percent relative to the pretreatment value while the river reference plant height was 16.7 percent higher than the pretreatment plant height (Figure 7). *Myriophyllum spicatum* plant heights for all treatment plots decreased 28 days after treatment except for the endothall/403 invert plot applied at 10 kg a.e. endothall/ha which remained the same. In order of decreasing plant height 56 days after treatment the 2,4-D in lignin plot showed the greatest decrease (66.7 percent) followed by 13 kg a.e. endothall/403 (47.1 percent), 20 kg a.e. endothall/Spra-mate (41.7 percent), 20 kg a.e. endothall/403 (41.2 percent), 178 kg a.e. poly

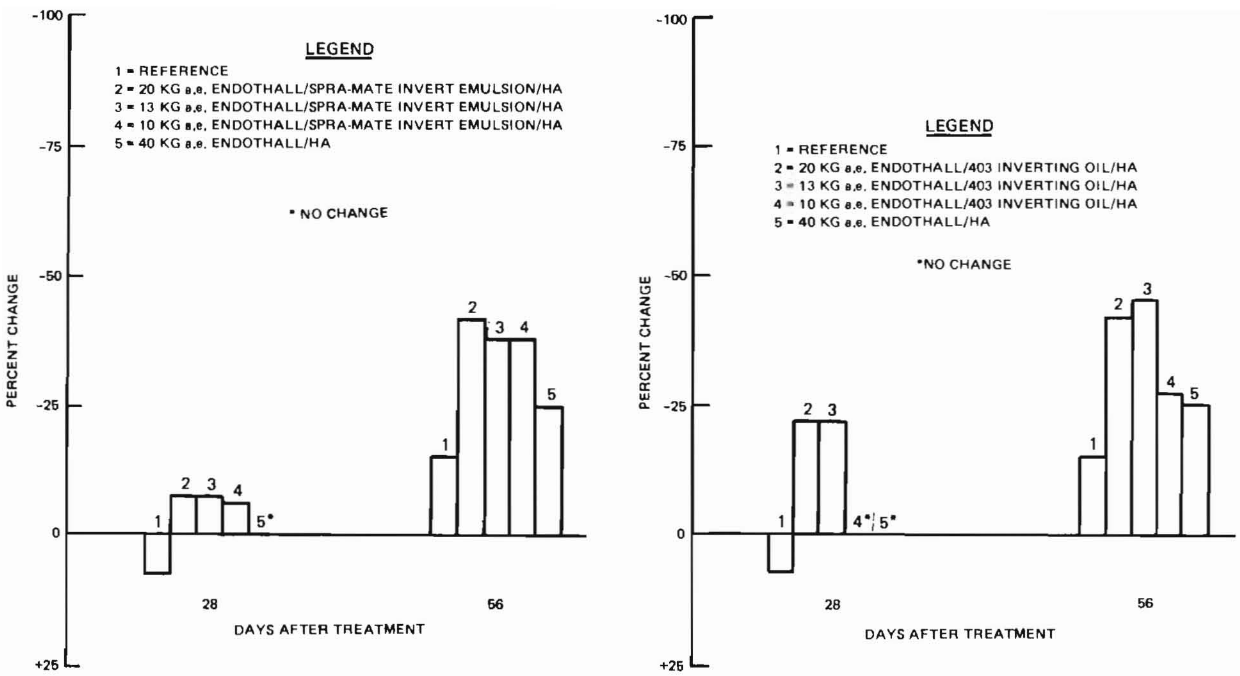

Figure 6. Percent change in *M. spicatum* plant height 28 and 56 days after treatment for the 1981 endothall/invert plots

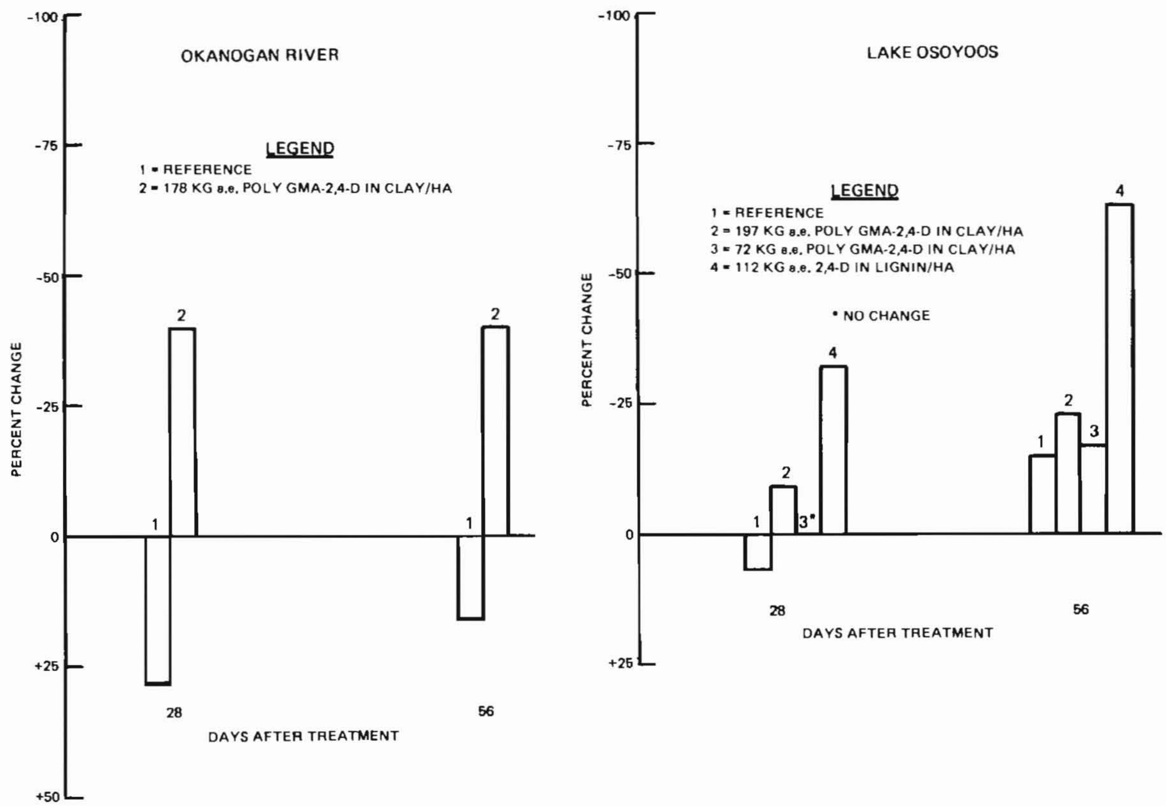

Figure 7. Percent change in *M. spicatum* plant height 28 and 56 days after treatment for the 1981 controlled-release plots

GMA-2,4-D in clay (40 percent), 13 kg a.e. endothall/Spra-mate (37.5 percent), 10 kg a.e. endothall/Spra-mate (37.5 percent), 10 kg a.e. endothall/403 (26.7 percent), 197 kg a.e. poly GMA-2,4-D in clay (22.7 percent), 40 kg a.e. endothall (20 percent), and the 72 kg a.e. poly GMA 2,4-D in clay (15.8 percent) (Figures 6 and 7).

24. Qualitatively, the treated *M. spicatum* canopies lost their buoyancy and slumped over 1 to 3 weeks after treatment in all treatment plots. However, underlying viable *M. spicatum* plants were continuously growing through the canopy. Thus, growth below the canopy was evidently responsible for the relatively minimal reduction in plant height.

Herbicide residue in water

25. Measurements of herbicide residue in the water for the controlled release and endothall/invert plots are summarized in Tables 8 and 9, respectively. The controlled-release formulations exhibited an initial high release (25.9 μg/ℓ for the high rate poly GMA and 24.3 μg/ℓ for the lignin formulations) and gradually decreased to undetectable levels 64 days after treatment. The maximum mean herbicide concentration in the water for the endothall and endothall/invert plots was 317.7 μg/ℓ, detected in the endothall/403 inverting oil plot applied at 13 kg a.e. endothall/ha. Endothall was not detected in the endothall/invert plots applied at low rates (i.e. 10 kg a.e. endothall/ha) and endothall was not detected 2 days after treatment in any of the treatment plots.

26. Table 10 shows herbicide concentrations outside the treatment plots. Low endothall and 2,4-D concentrations (i.e., less than 0.1 mg/ℓ) were detected between the lake treatment plots the first 2 days after treatment. Downstream of the controlled-release river plot 2,4-D was detected in one sample 32 days after treatment. Endothall and 2,4-D were not detected in the reference plots.

Herbicide residue in the sediment

27. The mean 2,4-D concentrations found in the sediment for the controlled release plots are shown in Table 11. Concentrations of 2,4-D were detected in the poly GMA-2,4-D lake plot applied at 197 kg a.e. 2,4-D/ha 32 and 64 days after treatment (182.2 and 157.5 μg/ℓ, respectively). The poly GMA-2,4-D lake plot applied at a lower rate was not sampled. The lignin 2,4-D plot in the lake and the poly GMA-2,4-D plot in the river showed virtually no 2,4-D residue. No residue was found in the reference plots.

Biomass

28. Mean *M. spicatum* biomass values in wet weight are shown in Table 12. Dry and ash-free dry weight, as calculated from the equations presented in Appendix C, are also shown in Table 12. Changes in ash-free dry weight of *M. spicatum* 32 and 64 days after treatment are shown in Figure 8.* The

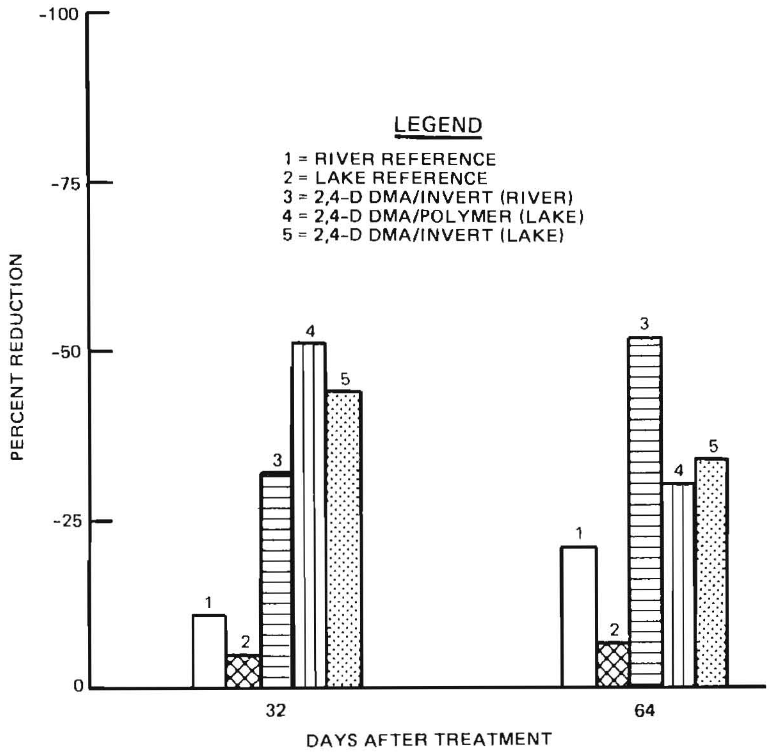

Figure 8. Percent change in *M. spicatum* ash-free dry weight 32 and 64 days after treatment for the 1982 treatment plots

biomass decreased in all plots. However, the river reference plot decreased only 22 percent 64 days after treatment, while the biomass in the 2,4-D DMA/invert river plot decreased 52 percent. The lake reference plot biomass decreased 6 percent 64 days after treatment while the biomass in the 2,4-D DMA/polymer and 2,4-D DMA/invert lake plots decreased 30 and 38 percent, respectively. These data indicate regrowth of *M. spicatum*, probably due to

* The 2,4-D BEE/granular and 2,4-D DMA/polymer river plots were not sampled because the biomass sampler could not be navigated to these areas.

growth from existing root stocks or from fragments floating into the plot from adjacent areas.

Plant height

29. The mean *M. spicatum* heights and water depths for the test plots during the 1982 study are shown in Table 13. Changes in plant height 32 and 64 days after treatment are graphically shown in Figure 9. The river

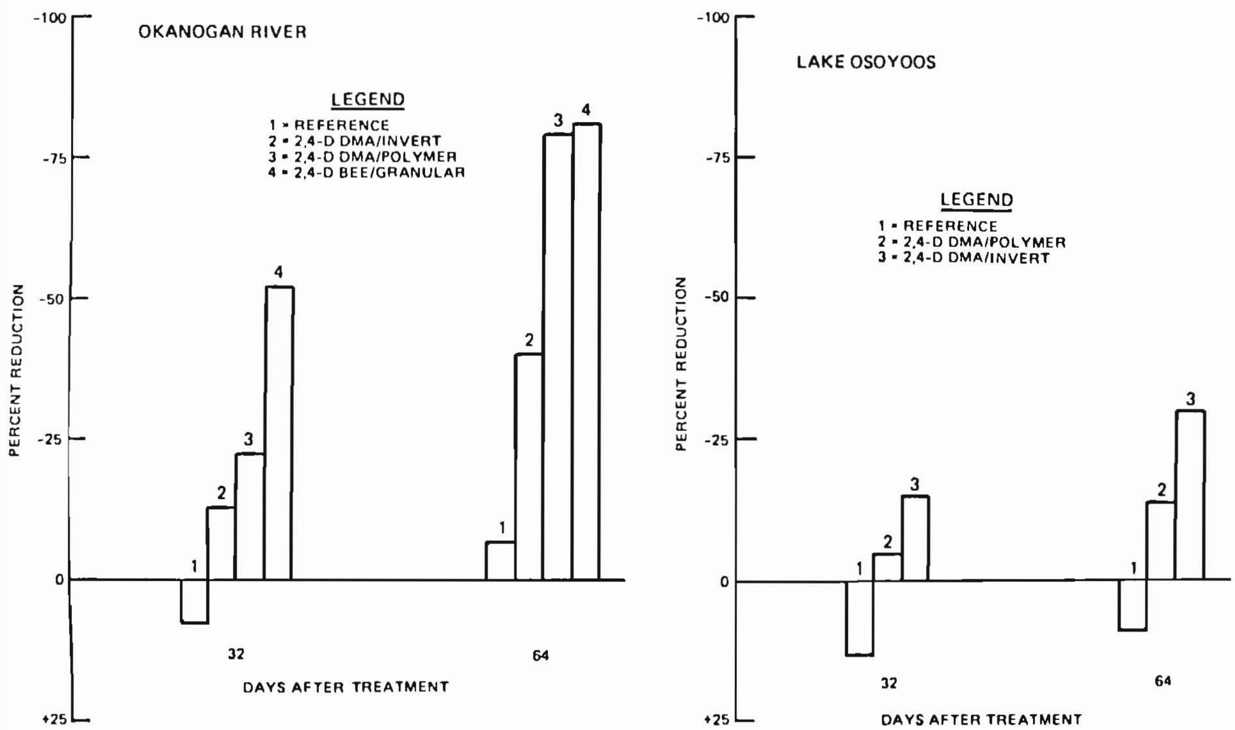

Figure 9. Percent change in *M. spicatum* height 32 and 64 days after treatment for the 1982 treatment plots

reference plot showed a slight increase in plant height 32 days after treatment but decreased 3 percent 64 days after treatment. The three treatment plots in the river continued to decrease throughout the sampling period. Necrotic plants would break off and move downstream. By 64 days after treatment, the 2,4-D BEE/granular plot showed an 80-percent reduction in plant height, followed by a 78-percent reduction in the 2,4-D DMA/polymer plot and a 40-percent reduction in the 2,4-D DMA/invert plot. Based on a review of the herbicide residue data, the degree of reduction of plant height in the 2,4-D BEE/granular plot was partly attributed to subsequent addition of 2,4-D from the upstream polymer plot. Also, the 2,4-D DMA/invert river plot was originally scheduled to receive the same application rates as the other two river treatment plots. However, the 2,4-D DMA/invert river plot was applied at a lower rate due to an application equipment malfunction, and thus the plant height (and biomass) reduction were not as pronounced as the other two river treatment plots applied at maximum application rates. The water depth remained the same throughout the study.

30. The lake reference plot plant height increased 32 and 64 days after treatment compared to the pretreatment value. The 2,4-D DMA/polymer and 2,4-D DMA/invert lake plots showed a decrease in plant height of 14 and 30 percent, respectively, 64 days after treatment. Growth from underlying *M. spicatum* plants did not allow a significant reduction in plant height. The water depth in the lake remained the same throughout the study.

Herbicide residue in water

31. The mean 2,4-D concentrations in the water are shown in Table 14 and Figure 10. The majority of 2,4-D residues in the river were found 0 to 2 hr after treatment and up to 24 hr after treatment in the lake. Herbicide travel time in the river was similar to the travel time of the dye (i.e., peak concentration of the dye and herbicide was detected within 120 min after application). The maximum mean concentration detected in the river was 24.5 μg/ℓ (2,4-D DMA/invert river plot) and 573.3 μg/ℓ in the lake (2,4-D DMA/polymer lake plot). Except for the 2,4-D BEE/granular river plot, 2,4-D persisted in the river plots for only 4 hr after treatment. The 2,4-D BEE/granular river plot was contaminated from the application of the upstream polymer plot (890 μg/ℓ of 2,4-D acid was detected between the 2,4-D DMA/polymer and 2,4-D BEE/granular river plots 30 min after the 2,4-D DMA/polymer application) and 2,4-D was subsequently detected in the water 3 days after the 2,4-D BEE/granular application. No 2,4-D was found in the lake or river reference plots.

32. Higher residue levels were found in the downstream dispersal sampling stations than inside the 2,4-D BEE/granular or 2,4-D DMA/polymer river plots. Conversely, higher residue levels were found inside the 2,4-D DMA/invert river plot than in the downstream dispersal sampling station, partly a function of the lower application rate. Also, lower 2,4-D residue levels were found outside of both the 2,4-D DMA/polymer and 2,4-D DMA/invert lake plots than within the plots.

Herbicide residue in *M. spicatum*

33. Mean herbicide values in *M. spicatum* plants are shown in Table 15 and Figure 11. The 2,4-D levels were higher in the plants than in the

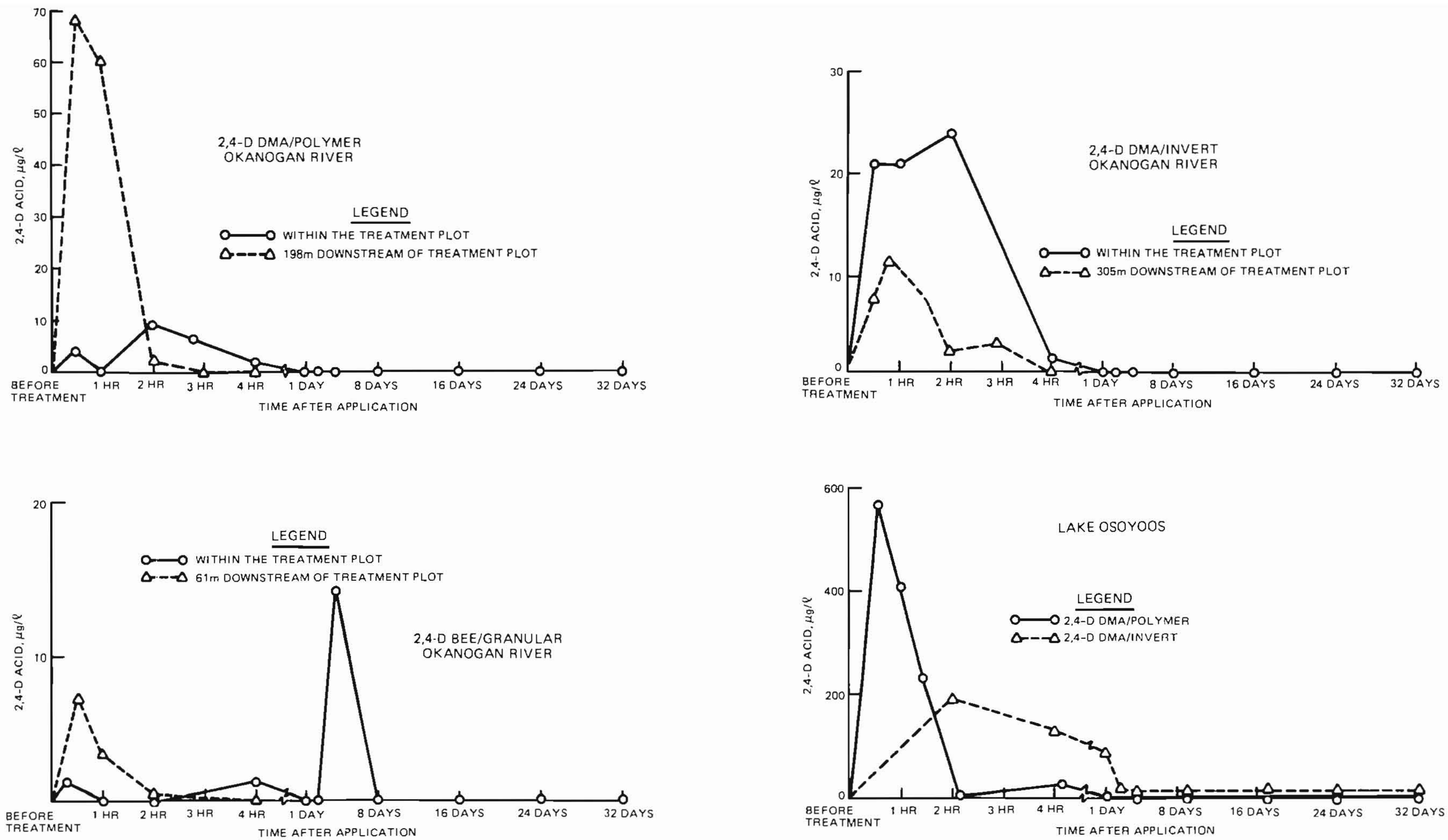

Figure 10. Mean herbicide concentrations in the water for the 1982 river and lake treatment plots

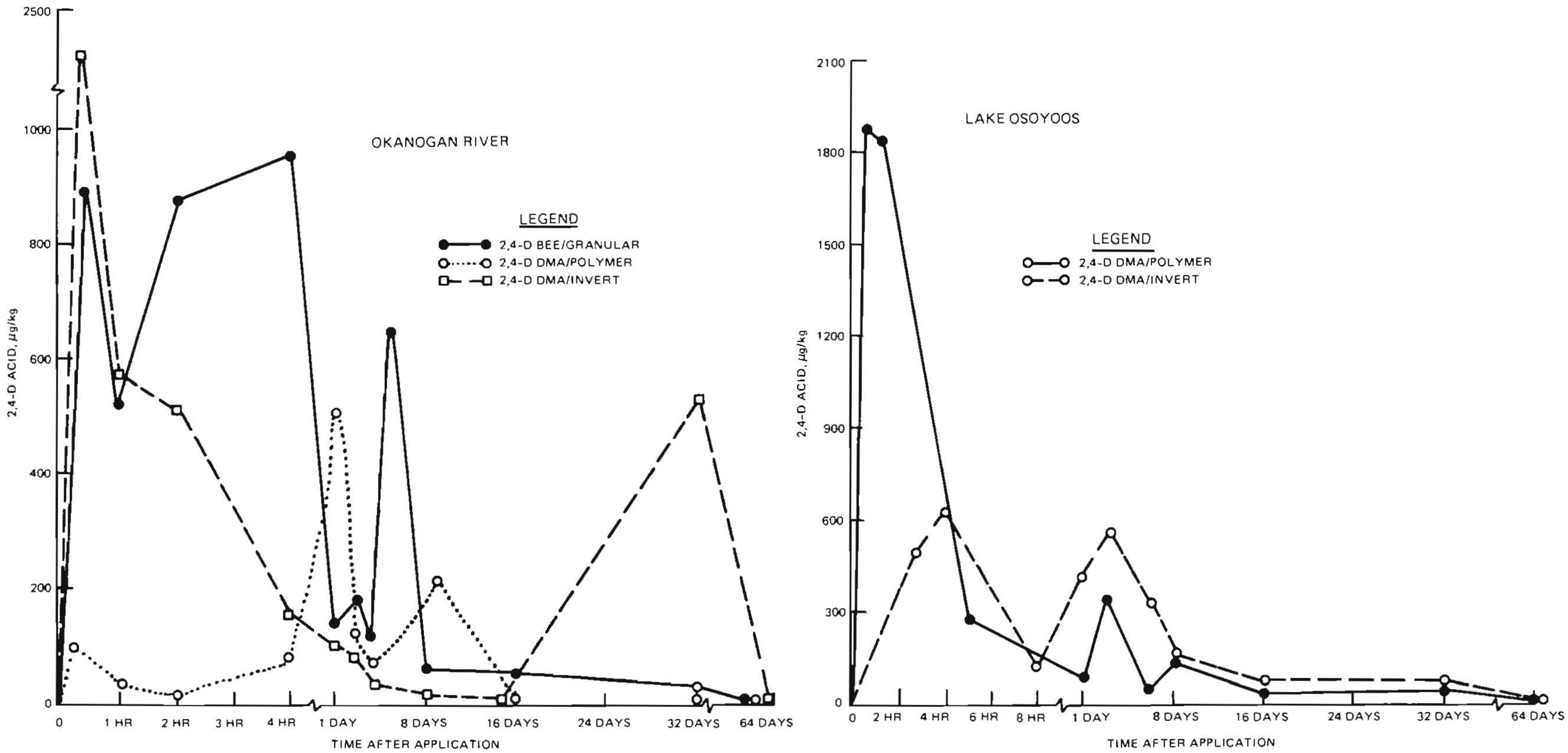

Figure 11. Mean herbicide residue in *M. spicatum* for the 1982 treatment plots

surrounding water. The maximum mean concentration was 2160.2 μg/ℓ in the 2,4-D DMA/invert river plot 30 min after treatment. The 2,4-D levels remained detectable in all plots 32 days after treatment, except for the 2,4-D DMA/polymer river plot where 2,4-D residues were not detected 8 days after treatment. These data also reflect interplot contamination between the 2,4-D BEE/granular and 2,4-D DMA/polymer river plots as a second distinctive peak. Low levels of 2,4-D (i.e. 88 μg/ℓ) were detected in the river reference plot 1 week after treatment.

Herbicide residue in sediment

34. Mean 2,4-D residue values in the sediment are shown in Table 16 and Figure 12. The mean pH in the sediment was 7.3 ± 0.3, n = 137. (Lim and Lozoway (1977) point out that the 2,4-D herbicidal action is favored by acidic pH whereas alkaline pH may inhibit its effect.) Higher residues were found in the 2,4-D BEE/granular river plot (maximum of 4333.3 μg/ℓ) than in the other treatment plots and were detected before interplot contamination. Sediment herbicide residue persisted 32 days after treatment in the 2,4-D BEE/granular river plot, 8 days in the 2,4-D DMA/polymer river plot, 2 hr in the 2,4-D DMA/invert river plot, 24 hr in the 2,4-D DMA/invert lake plot, and nondetectable in the 2,4-D DMA/polymer lake plot. Low levels of 2,4-D (i.e. 81 μg/ℓ) were detected in the river reference plot 1 week after treatment.

Water velocities

35. Mean water velocity values within and outside of the river test plots are graphically presented in Figure 13. Empirical numbers used in the water velocity calculation are shown in Table 17. Average stream velocity and flow rates were proportional to the density of *M. spicatum* colonies. As density decreased in response to treatment, stream velocity increased. Average stream velocities measured outside the colonies were approximately twice that of the velocities measured within the colonies. According to the USGS gaging station below Zosel Mill Dam, there was a slight variation in the flow rates ranging from 37 to 56 cms during the first 2 weeks after treatment. However, there was negligible change in the water level within the study site (above Zosel Mill Dam).

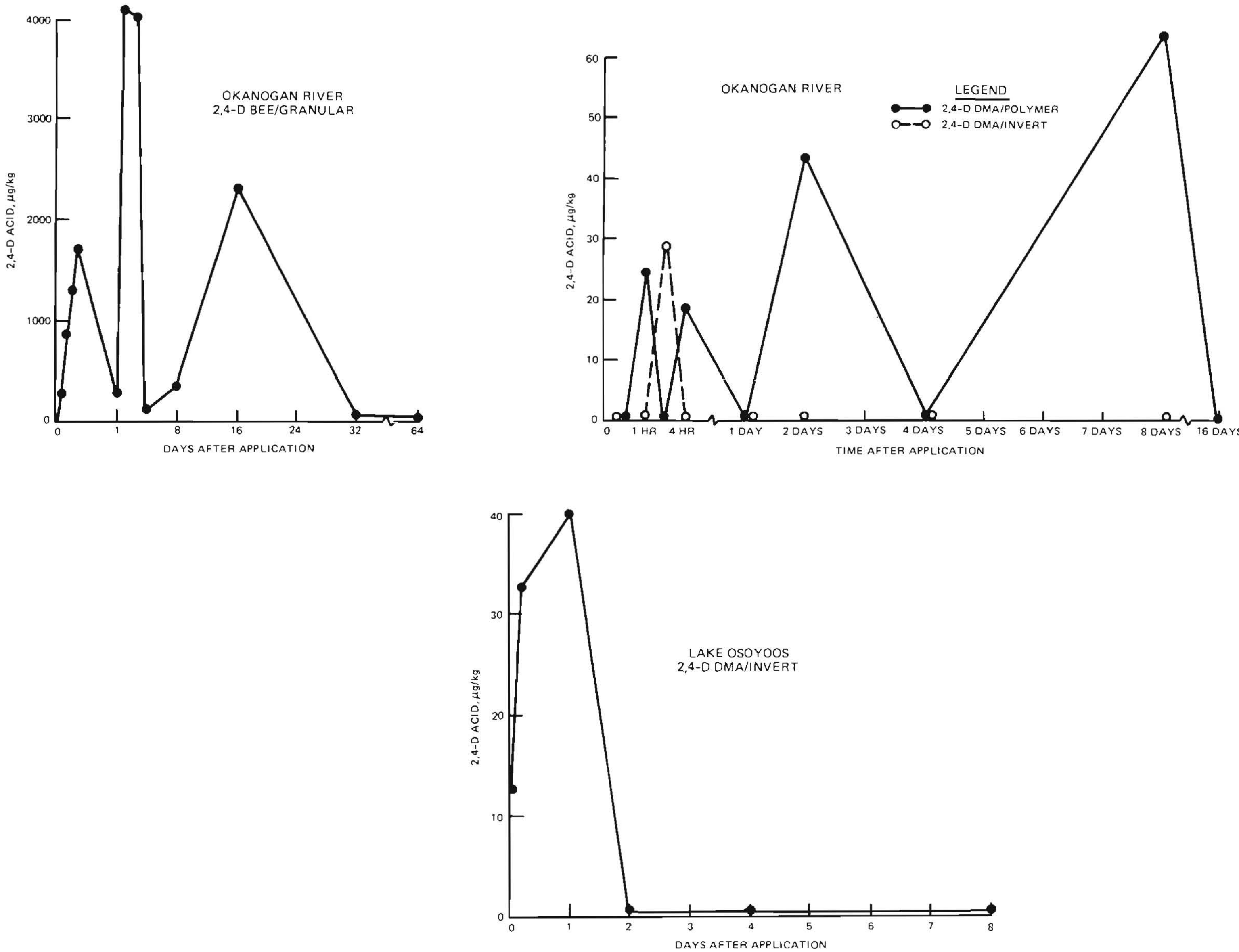

Figure 12. Mean herbicide residue in the sediment for the 1982 treatment plots

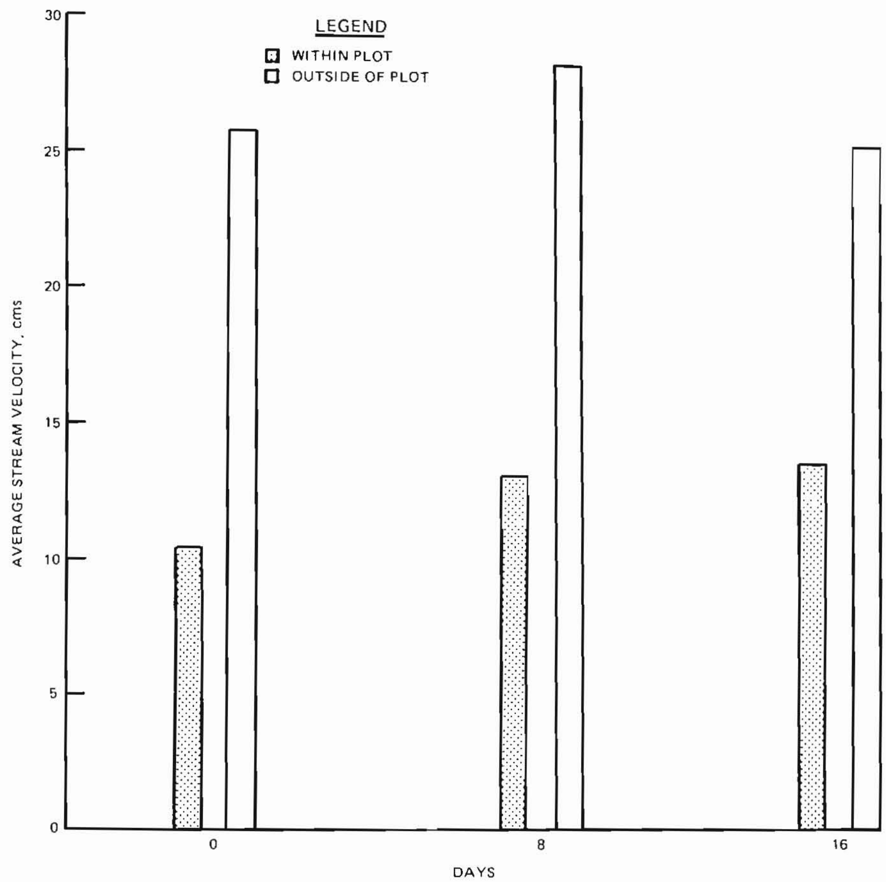

Figure 13. Mean water velocity within and outside of the 1982 river plots 0 to 16 days after treatment

PART V: DISCUSSION

36. When liquid formulations of a herbicide are applied without adjuvants, the applicator is, in effect, treating the entire water column. Conversely, when adjuvants are used in the treatment, the applicator can selectively place the herbicide onto the target plant colony, and thus treat only the standing biomass. Adjuvants serve to maintain toxic concentrations of the herbicides in close proximity to the target plant, reduce dispersal, and increase the efficiency and longevity of the herbicide by protecting it from environmental degradation (Gates 1972). It is still unclear if adjuvants can decrease treatment costs in moving waters since the primary justification would be a decrease in the herbicide required to effect control when using adjuvants. The available quantitative data are not yet conclusive to support this assumption. It is clear, however, that moving water, caused by either wind-induced or gravity currents, has been the primary dissipator of a herbicide from the treatment area (Smith, Hall, and Stanley 1967; Bergquist 1970; Wojtalik, Hall, and Hill 1970; Frank 1972; Foret and Barry 1979; Bowmer 1979; Goodard 1980; Eaton, Murphy, and Hyde 1981). The use of adjuvants may be justified in still water habitats to maintain a toxic herbicide concentration in vicinity of the plants longer than without their use.

Still Water Applications

2,4-D

37. This study showed that sparse (i.e., 65 g/m^2 dry weight) to moderately dense (i.e., 155 g/m^2 dry weight) *M. spicatum* colonies can be controlled with 2,4-D BEE/granular and 2,4-D DMA/polymer formulations applied at less than label-recommended application rates (i.e., 20 to 33 kg a.e./ha or 1.2 to 1.8 mg/ℓ). Conversely, dense *M. spicatum* colonies (i.e., greater than 300 g/m^2 dry weight) were not controlled with 2,4-D DMA/polymer or 2,4-D DMA/invert formulations applied at 34 kg a.e./ha (i.e., 1.2-1.4 mg/ℓ). A 2,4-D application rate of 34 kg a.e./ha is in the range considered as an effective rate for controlling *M. spicatum* (Smith, Hall, and Stanley 1967; Elliston and Steward 1972; Steward and Nelson 1972; Getsinger, Davis, and Brinson 1982), but in this study a sufficient amount of the herbicide/adjuvant formulation did not penetrate through the dense canopy to prevent growth of underlying

plants. This is exemplified from the fact that no sediment 2,4-D in the 2,4-D DMA/polymer plot was found and only negligible sediment 2,4-D in the 2,4-D DMA/invert plot was detected. As a result, viable *M. spicatum* plants grew through the necrotic canopy, subsequently minimizing biomass reduction. The canopy of sparse to moderately dense *M. spicatum* colonies (i.e., 60 to 150 g/m^2 dry weight) was not well developed, allowing the herbicide/adjuvant formulations to penetrate to the underlying biomass. Thus, 2,4-D treatment of *M. spicatum* colonies should occur before total canopy formation using concentrations of 1.2 to 1.4 mg/ℓ 2,4-D acid; and, if the canopy has formed, two treatments per growing season or the use of weighted trailing hoses with lengths suitable to apply part of the formulation below the canopy may be required even when adjuvants are used.

38. Waterborne 2,4-D concentrations were below 0.1 mg/ℓ 24 hr after application and were nondetectable after 4 days in all 2,4-D treatment areas except for the controlled release plots. The controlled release formulations exhibited an initial spike 2,4-D release sufficient to effect partial reduction in the treated areas. However, both formulations failed to provide a continuous release of an effective 2,4-D concentration for a prolonged period. Hall et al. (1982) showed that the long-term 2,4-D threshold concentrations required to control *M. spicatum* in a laboratory study ranged from 0.05 to 0.10 mg/ℓ. Waterborne residues detected in the water 4 days after treatment were much lower than this reported threshold value (<0.01 mg/ℓ) and were virtually nondetectable through the end of the study. However, residues in the sediment were appreciably higher than in the water, indicating that the *M. spicatum* height reduction was probably due to the 2,4-D being absorbed primarily through the *M. spicatum* roots.

39. Negligible 2,4-D residues were detected in the buffer zones outside the treatment plot. Dispersion of 2,4-D was primarily caused by wind-driven currents. With such low 2,4-D concentrations detected in the buffer zones relative to the treatment plot, the adjuvants appeared to minimize dispersion and provided a more sustained herbicide concentration in the vicinity of the target species.

Endothall

40. Moderately sparse to dense *M. spicatum* colonies were not controlled with endothall/adjuvant formulations applied at 10 to 20 kg a.e./ha (i.e., 0.7 to 1.6 mg/ℓ). These formulations also would not sink through the canopy.

This fact was indicated by viable *M. spicatum* plants growing through the canopy that had subsequently dropped below the water surface after treatment. Furthermore, the plant tissues that were in direct contact with the endothall/adjuvant formulations showed little herbicidal effects except for the loss of buoyancy. The endothall/adjuvant application rates were apparently too low to control even moderately sparse *M. spicatum* colonies, suggesting that application rates above 20 kg a.e./ha (i.e., 1.1 mg/ℓ) are warranted. (See Keckement (1969), Frank and Comes (1967), and Serns (1977) for a discussion on effective endothall application rates.) Additionally, adjuvants did not provide enough weight for the formulations to penetrate the canopy and thus also require "below the canopy" application.

41. Although effective control was not evident, the endothall applied without an adjuvant at a rate of 40 kg a.e./ha (i.e. 1.6 mg/ℓ) showed less control than the endothall/invert formulations applied at 0.6 to 1.1 mg/ℓ, suggesting that the inverts did enhance the contact properties of the herbicide. Most *M. spicatum* plants had deposits (i.e. marl and periphyton) on the cuticle of the leaflets which could inhibit penetration of the herbicide. The invert may have allowed a longer contact time of the herbicide to the plant, resulting in penetration of endothall through the deposits, while the endothall only application either became diluted to an ineffective concentration or dispersed from the treated areas.

42. Endothall also persisted in the water for only a short period. Keckemet (1969) states that endothall is a highly biodegradable compound commonly disappearing from the water in 1 to 10 days. Endothall was detected only up to 1 day after treatment in both the treatment plots and the buffer zones in Lake Osoyoos. Westerdahl (1983) studied the fate of endothall on hydrilla in Gatun Lake, Panama, and suggested that endothall dispersed laterally as a density flow resulting in the rapid disappearance of endothall from the water. Much of the endothall applied in Lake Osoyoos may have dispersed down the terrace shelf as a density flow, resulting in the low concentrations in the treatment site.

Diquat

43. The diquat/polymer formulation proved effective for controlling a dense *M. spicatum* colony (i.e., 333 g/m^2 dry weight) at an application rate of 27 kg/ha (i.e., 1.0 mg/ℓ). Calderbank (1972) states that such low application rates are feasible because diquat is rapidly absorbed by the aquatic plants.

Contrary to the results obtained for the 2,4-D DMA/polymer formulation applied in a dense *M. spicatum* colony, the diquat/polymer formulation apparently penetrated the canopy, resulting in a 91-percent reduction of the standing crop 84 days after treatment. Diquat was also virtually nondetectable in both treatment plots and buffer zones, the persistence influenced by the rapid absorption by the aquatic vegetation, soil, and detritus (Frank and Comes 1967; Hiltibran, Underwood, and Fickle 1972; Calderbank 1972; Baker et al. 1975).

Moving Water Applications

44. The 2,4-D/adjuvant applications in flowing water (9 to 15 cms) decreased the height and biomass of dense *M. spicatum* colonies (>300 g/m^2 dry weight) although regrowth was evident in the 2,4-D DMA/invert plot applied at 12 kg a.e. 2,4-D/ha. The 2,4-D that was not in direct contact with the plants apparently moved downstream from the application site and was subsequently detected at the downstream dispersal sampling station. Thus, the adjuvants did not completely eliminate dispersal. Frank (1972) stated that a minimum of 48 hr exposure to 1.0 mg/ℓ 2,4-D is required for complete *Myriophyllum* control. If water velocities exceed 35 cms, then application rates may have to be increased to the maximum label-recommended application rates. This increase will compensate for dilution and dispersion by allowing the toxic concentration to remain on the plant long enough to effect control.

45. These data suggest that the invert formulations were more efficient than the polymer formulations in maintaining the residence time of 2,4-D in flowing water. Higher levels of 2,4-D were detected in the invert plots longer than in the polymer plots. Although the 2,4-D DMA/polymer river plot did show a 78-percent reduction in plant height, the 2,4-D DMA/invert plot was applied at a rate approximately 70 percent less (i.e. 12 kg/ha) than the polymer plot (i.e. 45 kg/ha) and still showed a 40-percent reduction.

46. The formulations leaving the trailing hose in the 2,4-D invert and polymer river plots were sampled and analyzed in the laboratory. The 2,4-D concentration from the 2,4-D DMA/polymer plot was 19.0 mg/ℓ, whereas the highest residue value detected in the plants was 0.5 mg/ℓ, 40 times less than the amount applied. Conversely, the 2,4-D concentration from the 2,4-D DMA/invert plot was 8.4 mg/ℓ and the highest residue value detected in the plants was 2.1 mg/ℓ, only 4 times less than the amount applied. Thus, these data also

indicate that the invert may allow a more sustained contact time of the herbicide to the plant than the polymer.

47. The 2,4-D BEE/granular river plot also showed effective control (i.e. 80-percent reduction in plant height). Studies have shown that over 50 percent of the 2,4-D acid is released from the granules in the first day (Wilkinson 1961). This initial release may be followed by a prolonged release of remaining 2,4-D, minimizing immediate dispersal and dilution of all the 2,4-D acid applied. Because of the granular formulations close association to the soil/water interface, the 2,4-D is more likely to be absorbed by the *M. spicatum* roots and then move readily to the shoots (Funderburk and Lawrence 1963). This was evident in the 1982 study. The initial release of the 2,4-D from the granules apparently was absorbed by the aboveground biomass (i.e. >0.5 mg/ℓ 2,4-D acid in *M. spicatum* tissues the first 4 hr after application), whereas the majority of the remaining formulation was present in the sediment (i.e. 4.0 mg/ℓ 2,4-D acid in sediment 3 days after application) available for absorption by the roots. Unfortunately, interplot contamination did not allow a true assessment of the long-term retention time of the 2,4-D within the 2,4-D BEE/granular river plot.

48. Stream velocity was the only water quality variable affected by the treated *M. spicatum* colonies during the study period. Stream velocity increased as the *M. spicatum* biomass decreased. Thus, one obvious impact *M. spicatum* exhibits on the water is a radical decrease in water velocity.

PART VI: CONCLUSIONS AND RECOMMENDATIONS

Conclusions

49. The following herbicide/adjuvant formulations reduced the biomass or plant height of *M. spicatum* colonies by over 75 percent (i.e., an effective reduction):

Formulation	Application Rate	Percentage of Maximum Label Application Rate	Colony Density g/m^2 dry wt.	Days After Appli-cation	Water Velocity cms
2,4-D BEE/Granular	20 kg a.e/ha (1.2 mg/ℓ)	43	65	84	0
2,4-D DMA/Polymer	33 kg a.e./ha (1.8 mg/ℓ)	74	155	84	0
Diquat/Polymer	27 kg cation/ha (1.0 mg/ℓ)	67	333	84	0
2,4-D BEE/Granular	45 kg a.e./ha (2.8 mg/ℓ)	100	>300	56	35
2,4-D DMA/Polymer	45 kg a.e./ha (3.4 mg/ℓ)	100	>300	56	26

50. The following herbicide/adjuvant formulations did not reduce the biomass and/or plant height of *M. spicatum* colonies by over 75 percent:

Formulation	Application Rate(s)	Percentage of Maximum Label Application Rate	Colony Density g/m^2 dry wt.	Days After Appli-cation	Water Velocity cms
Endothall/Polymer	18 kg a.e/ha (0.7 mg/ℓ)	25	122	84	0
Endothall/Inverts	10 to 20 kg a.e/ha (0.5-1.1 mg/ℓ)	17-38	*	64	0
Endothall	40 kg a.e./ha (1.6 mg/ℓ)	56	*	64	0
Acrylic copolymer matrix in clay (controlled release)	72 to 197 kg a.e 2,4-D/ha (0.03-0.05 mg/ℓ)	--**	*	64	0

(Continued)

* Biomass estimated to be in excess of 300 g/m^2 dry weight.
** -- No maximum application rate currently established.

Formulation	Application Rate(s)	Percentage of Maximum Label Application Rate	Colony Density g/m^2 dry wt.	Days After Appli- cation	Water Velocity cms
Kraft Lignin (controlled release)	102 kg a.e. 2,4-D/ha (0.03-0.05 mg/ℓ)	--**	*	64	0
2,4-D DMA/Polymer	34 kg a.e./ha (1.4 mg/ℓ)	75	440	56	0
2,4-D DMA/Invert	34 kg a.e./ha (1.2 mg/ℓ)	75	336	56	0
Acrylic copolymer matrix in clay (controlled release)	178 kg a.e. 2,4-D/ha (0.03 to 0.05 mg/ℓ)	--	*	64	21
2,4-D DMA/Invert	12 kg 2,4-D/ha (0.6 mg/ℓ)	25	537	56	9

* Biomass estimated to be in excess of 300 g/m^2 dry weight.
** -- No maximum application rate currently established.

51. Waterborne endothall and 2,4-D herbicide concentrations became nondetectable in less than a week after treatment, and diquat was not detected in the water 2 weeks posttreatment. All waterborne residues were below 0.1 mg/ℓ 24 hr after treatment.

52. Relatively higher and more sustained 2,4-D levels were detected in the *M. spicatum* plants using 2,4-D DMA/inverts when applied in flowing water than were found using 2,4-D DMA/polymer or 2,4-D BEE/granular.

53. Relatively higher and more sustained 2,4-D levels were detected in the sediment using 2,4-D BEE/granular when applied in flowing water than were found using 2,4-D DMA/polymer or 2,4-D DMA/invert.

54. Regardless of the adjuvant used in flowing water, herbicide dispersal is not totally eliminated. The rate of dispersal will be primarily dependent on water velocity.

Recommendations

55. The following recommendations can be made based on the results of this study:

a. If the canopy is not well developed within a *M. spicatum* colony growing in static water, a 2,4-D concentration of 1.2 to 1.8 mg/ℓ is recommended for controlling the standing crop.

b. Maximum labeled application rates of 2,4-D or a diquat concentration of at least 1.0 mg/ℓ is recommended for controlling dense *M. spicatum* colonies growing in static water. Part of the herbicide formulation must be applied below the canopy to control the underlying biomass.

c. Maximum labeled application rates of endothall should be used to control moderate to dense colonies of *M. spicatum*.

d. Based on the criteria of measured biomass reduction and herbicide retention time, the following herbicide/adjuvant formulations are recommended for controlling *M. spicatum* in flowing water: 45 kg a.e./ha 2,4-D BEE/granular, 45 kg a.e./ha 2,4-D DMA/403 inverting oil, and 45 kg a.e./ha 2,4-D DMA/polymer.

e. Field use of 403 inverting oil is recommended over Spra-mate invert emulsion.

f. Further studies are required to determine if adjuvants will allow the applicator to reduce application rates and still achieve the same degree of *M. spicatum* control.

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Table 1

Areal Distribution of *M. spicatum* in the Study Site from 1979-1982

Date	Location	Area, ha	Number of Colonies	Average Colony Size, m^2
1979	Okanogan River*	1.1	33	325
1980	Okanogan River	2.6	29	947
1981	Okanogan River	2.8	38	738
1982	Okanogan River	2.6	28	935
1979	Lake Osoyoos, west side	1.2	6	1153
1980	Lake Osoyoos, west side	1.9	41	468
1981	Lake Osoyoos, west side	1.5	39	375
1982	Lake Osoyoos, west side	2.6	22	1183
1979	Lake Osoyoos, east side	8.0	43	1858
1980	Lake Osoyoos, east side	23.0	54	4076
1981	Lake Osoyoos, east side	13.1	29	4512
1982	Lake Osoyoos, east side	15.7	21	7467

* Okanogan River from the outlet of Lake Osoyoos to Zosel Mill Dam.

Table 2

Summary of Herbicide Applications

Date	Application Date	Location	Plot Description: Mean Water Size ha	Plot Description: Depth m	Herbicide Brand Name	Herbicide Application Rate	Brand Name Adjuvant	Adjuvant Application Rate ℓ	Water ℓ	Concentration of Active Ingredients In Plot, mg/ℓ	Application Equipment: Dispersing Device	Application Equipment: Nozzle
1980	7/23	Lake Osoyoos	0.4	2.4	Aquathol K	18 kg endothall acid	Nalquatic polymer	10	410	0.7	John Bean Pump, single tank system	None
	7/22	↓	0.4	2.6	Ortho diquat	27 kg diquat cation	Nalquatic polymer	10	440	1.0	John Bean Pump, single tank system	None
	7/22	↓	0.4	1.9	Weedar 64	33 kg 2,4-D acid	Nalquatic polymer	10	393	1.8	John Bean Pump, single tank system	None
	7/22	↓	0.4	1.6	Aqua Kleen	20 kg 2,4-D acid	None	NA*	NA	1.2	Cyclone spreader	NA
1981	7/19	↓	0.2	2.3	Wright St. Univ.**	197 kg, 2,4-D acid	Acrylic copolymer matrix in clay	NA	NA	CR†	↓	↓
	7/18	↓	0.2	2.7	Wright St. Univ.	72 kg 2,4-D acid	Acrylic copolymer matrix in clay	NA	NA	CR	↓	↓
	7/17	Okanogan River	0.4	1.8	Wright St. Univ.	178 kg 2,4-D acid	Acrylic copolymer matrix in clay	NA	NA	CR	↓	↓
	7/19	Lake Osoyoos	0.4	2.7	Westvaco, Inc.†	102 kg 2,4-D acid	Kraft lignin	NA	NA	CR	↓	↓
	7/18	↓	0.4	1.9	Aquathol K	10 kg endothall acid	Spra-mate invert emulsion Xylene	7 20	210	0.5	Modified John Bean Pump, two tank system	No. 6 Raindrop
	7/19	↓	0.4	2.6	↓	13 kg endothall acid	Spra-mate invert emulsion Xylene	12 28	245	0.5	↓	↓
	7/17	↓	0.4	2.2	↓	20 kg endothall acid	Spra-mate invert emulsion Xylene	14 37	250	1.1	↓	↓
	7/16	↓	0.4	1.7	↓	10 kg endothall acid	403 inverting oil	37	243	0.6	↓	↓
	7/17	↓	0.4	2.2	↓	13 kg endothall acid	403 inverting oil	42	260	0.6	↓	↓
	7/17	↓	0.4	1.9	↓	20 kg endothall acid	403 inverting oil	49	281	1.1	↓	↓
	7/18	↓	0.4	2.6	↓	40 kg endothall acid	None	NA	355	1.6	Single tank mix	No. 6 Raindrop
1982	7/12	Okanogan River	0.2	1.6	Aqua Kleen	45 kg 2,4-D acid	None	NA	NA	2.8	Cyclone spreader	NA
	7/15	Okanogan River	0.4	1.3	Weedar 64	45 kg 2,4-D acid	Nalquatic polymer	27	757	3.4	Nalquatic inline suction feed system, three tank system	Straight Stream Jet
	7/16	Okanogan River	0.4	1.9	Weedar 64	12 kg 2,4-D acid	403 inverting oil	69	380	0.6	Minnesota-Warner invert pump, three tank system	No. 8 Raindrop
	7/14	Lake Osoyoos	0.4	2.4	Weedar 64	34 kg 2,4-D acid	Nalquatic polymer	25	700	1.4	Nalquatic inline suction feed system, three tank system	Straight Stream Jet
	7/16	Lake Osoyoos	0.4	2.8	Weedar 64	34 kg 2,4-D acid	403 inverting oil	65	380	1.2	Minnesota-Warner invert pump, three tank system	No. 8 Raindrop

Herbicide Formulations and Application Rates (In hectare equivalent units)

* Not applicable.
** Manufacturer of formulation.
† Controlled release at approximately 0.03 to 0.05 mg/ℓ.

Table 3

Summary of Types of Data Collected from 1980-1982

Year	Parameter	Pre-treatment	Sample Frequency																			
			Hours After Application								Days After Application											
			0	1/4	1/2	3/4	1	2	4	8	1	2	4	7	8	14	16	28	32	56	64	84
1980	Water temperature, dissolved oxygen, pH, conductivity, and Secchi disk depth	X*	X	--**	--	--	--	--	--	--	--	--	--	X	--	X	--	X	--	X	--	X
	Herbicide residue in water	X	X	--	--	--	--	--	--	--	--	--	--	X	--	X	--	X	--	X	--	--
	Biomass	X	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	X	--	--	--	X
1981	Herbicide residue in water	X	X	--	--	--	--	--	--	--	X	X	X	--	X	--	X†	--	X†	--	X†	--
	Herbicide residue in soil†	X	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	X	--	X	--
	Plant height	X	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	X	--	X	--
1982	Water temperature, dissolved oxygen, pH, conductivity, photosynthetically active radiation (PAR), alkalinity, hardness, Secchi disk depth, and turbidity	X	X	--	--	--	--	--	--	--	--	--	--	--	X	--	X	--	X	--	X	--
	Water velocity††	--	X	--	--	--	--	--	--	--	--	--	--	--	X	--	X	--	--	--	--	--
	Herbicide residue in water	X	X	X	X	X	X	X	X	X	X	X	X	--	X	--	X	--	X	--	--	--
	Herbicide residue in plants	X	X	--	X	--	X	X	X	X	X	X	X	--	X	--	X	--	X	--	X	--
	Herbicide residue in soil	X	X	--	X	--	X	X	X	X	X	X	X	--	X	--	X	--	X	--	X	--
	Plant height	X	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	X	--	X	--
	Biomass	X	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	X	--	X	--

* Sampled.
** Not sampled.
† Only for controlled-release plots.
†† Only for river plots.

Table 4

Instrumentation Used to Measure Site Variables

Instrument	Site Variable Measured
Martek Mark V digital water quality analyzer coupled to a Martek data logger (the data logger stored the water quality data on cassette tapes. The tapes were then read through a Martek data reader which printed a hard copy of the raw field data)	Water temperature, pH, dissolved oxygen, conductivity
Secchi disk	Water clarity
Titrametric field test kits	Turbidity hardness, alkalinity
Li-Cor photometer	PAR
Marsh-McBirney portable water velocity meter	Depth-specific water velocity
Master flex peristaltic pump (with silicon and bev-a-line tubing)	Herbicide residue in water
Pipe dredge	Herbicide residue in soil
Hand-held scoop dredge	Herbicide residue in plants
Raytheon DE fathometer	Submersed plant height, water depth
WES biomass sampler (see Dardeau and Lazor (1982) and Killgore and Payne (1984) for a description of the biomass sampler)	Submersed plant biomass
Turner Design flurometer	Dye concentrations

Table 5

Mean *M. spicatum* Biomass Values for the 1980 Test Plots

Date	Plot	Wet Weight, g/m^2				Dry Weight, g/m^2				Ash-Free Dry Weight, g/m^2			
		Mean	Standard Deviation	Minimum	Maximum	Mean	Standard Deviation	Minimum	Maximum	Mean	Standard Deviation	Minimum	Maximum
Pretreatment	Reference	2146.2	970.1	981.9	3601.2	313.9	142.2	145.5	505.2	147.9	57.7	82.9	220.2
	2,4-D BEE/granular	420.0	505.6	30.2	1230.6	64.6	76.2	4.1	182.6	15.8	19.9	0.9	48.1
	2,4-D DMA/polymer	1003.1	1192.6	301.3	3123.0	154.9	127.3	32.6	369.9	57.1	58.0	23.1	159.4
	Diquat/polymer	2325.6	1171.8	470.2	3447.4	331.1	159.6	85.8	487.2	122.7	61.2	33.9	203.4
	Endothall/polymer	882.7	546.2	300.0	1598.9	122.2	82.4	35.3	224.8	55.3	48.7	12.8	111.8
Day 28	Reference	1798.7	848.6	1066.1	3243.1	274.9	161.6	148.2	546.6	118.8	116.2	33.9	320.2
	2,4-D BEE/granular	20.4	44.7	0	100.3	2.5	5.6	0	12.5	1.5	5.6	0	8.7
	2,4-D DMA/polymer	445.6	996.3	0	2227.9	85.0	190.1	0	425.0	14.1	31.5	0	70.5
	Diquat/polymer	214.9	157.1	46.8	462.8	24.3	17.3	4.0	50.4	6.7	5.2	1.7	15.3
	Endothall/polymer	488.6	750.0	1.9	1805.8	55.3	90.3	0	215.9	41.3	79.2	0	182.7
Day 84	Reference	1473.8	657.1	521.7	2356.5	163.8	99.5	52.9	324.8	72.7	36.7	19.9	118.3
	2,4-D BEE/granular	67.6	114.2	0	263.6	6.2	9.9	0	22.9	2.4	4.3	0	9.9
	2,4-D DMA/polymer	24.5	38.1	0	86.7	3.1	5.4	0	12.6	2.5	4.1	0	9.4
	Diquat/polymer	325.6	184.2	88.6	544.6	34.3	35.9	7.2	96.2	11.5	5.3	6.7	19.1
	Endothall/polymer	308.1	280.8	21.2	706.1	34.7	42.3	1.5	100.2	19.0	11.9	1.5	30.1

Table 6

Mean Herbicide Residue in Water, Values for the 1980 Test Plots*

Date	Reference (n = 3)				2,4-D BEE/Granular				2,4-D DMA/Polymer				Diquat/Polymer				Endothall/Polymer			
	Mean	Standard Deviation	Min-imum	Max-imum	Mean	Standard Deviation	Mini-imum	Max-imum	Mean	Standard Deviation	Min-imum	Max-imum	Mean	Standard Deviation	Min-imum	Max-imum	Mean	Standard Deviation	Min-imum	Max-imum
Within the Plot (n = 6)																				
Pretreatment	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Day 0	2.1**	6.3	0	19.0	20.8	48.7	0	150	166.1	218.1	0	560	6.7	8.6	0	20.0	40.0	52.9	0	100.1
Day 7	0	0	0	0	0	0	0	0	0	0	0	0	10.0	14.1	0	20.0	0	0	0	0
Day 14	0	0	0	0	0	0	0	0	0	0	0	0	20.0	0	20.0	20.0	0	0	0	0
Day 28	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Day 56	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
30 m Outside the Plot (n = 4)																				
Pretreatment	--†	--	--	--	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Day 0	--	--	--	--	0.12	0.27	0	0.60	12.6	19.8	0	49.0	5.0	12.2	0	30.0	21.0	29.7	0	42.0
Day 7	--	--	--	--	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Day 14	--	--	--	--	0	0	0	0	0	0	0	0	5.0	7.1	0	10	0	0	0	0
Day 28	--	--	--	--	0	0	0	0	0.11	0.18	0	0.22	0	0	0	0	0	0	0	0
Day 56	--	--	--	--	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

* Values in micrograms per litre.
** Endothall acid.
† Not sampled.

Table 7

Mean *M. spicatum* Plant Heights and Water Depth for the 1981 Test Plots

Date	Plot (Ha Equivalent Units)	Average Plant Height, m					Average Water Depth, m				
		Mean	Standard Deviation	Minimum	Maximum	n	Mean	Standard Deviation	Minimum	Maximum	n
Pretreatment	197 kg a.e. poly GMA-2,4-D in clay	2.2	0.4	1.5	2.4	33	2.3	0.1	2.1	2.4	33
	72 kg a.e. poly GMA-2,4-D in clay	1.9	1.0	0	2.7	26	2.7	0.1	2.6	2.9	26
	178 kg a.e. poly GMA-2,4-D in clay	0.5	0.3	0	1.8	80	1.6	0.2	1.2	1.8	80
	112 kg a.e. 2,4-D in lignin	1.5	0.6	0	3.0	37	2.5	0.3	1.5	3.5	37
	20 kg a.e. endothall/Spra-mate	1.2	0.8	0	2.7	68	2.2	0.3	1.2	2.7	68
	13 kg a.e. endothall/Spra-mate	2.4	0.6	0	2.7	36	2.6	0.2	1.8	2.7	36
	10 kg a.e. endothall/Spra-mate	1.6	0.3	0.3	2.1	35	1.9	0.2	1.5	2.1	35
	20 kg a.e. endothall/403	1.7	0.9	0.3	2.4	54	1.9	0.7	0.3	2.4	54
	13 kg a.e. endothall/403	1.7	0.8	0.3	2.4	47	2.2	0.3	1.2	2.6	47
	10 kg a.e. endothall/403	1.5	0.5	0	2.3	46	1.7	0.4	0.3	2.4	46
	40 kg a.e. endothall	1.5	0.4	0	3.6	38	2.6	1.1	1.1	7.6	38
	Lake reference	1.3	0.4	0	2.7	44	3.6	0.1	3.3	3.9	44
	River reference	0.5	0.3	0	1.5	80	1.5	0.5	0.6	2.7	80
Day 28	197 kg a.e. poly GMA-2,4-D in clay	2.0	0.7	1.8	2.3	35	2.1	0.3	1.8	2.3	35
	72 kg a.e. poly GMA-2,4-D in clay	1.9	1.0	0	2.7	30	2.5	0.1	2.4	2.7	30
	178 kg a.e. poly GMA-2,4-D in Clay	0.3	0.4	0	1.5	80	1.4	0.3	0.3	1.7	80
	112 Kg a.e. 2,4-D in lignin	1.0	1.0	0	2.7	30	2.3	0.4	1.5	3.3	30
	20 kg a.e. endothall/Spra-mate	1.1	1.0	0	2.7	61	2.1	0.3	0.9	2.7	61
	13 kg a.e. endothall/Spra-mate	2.2	0.5	0.9	2.7	31	2.3	0.2	1.7	2.7	31
	10 kg a.e. endothall/Spra-mate	1.5	0.4	0	2.1	36	1.8	0.2	0.9	2.1	36
	20 kg a.e. endothall/403	1.3	0.5	0	2.1	49	1.8	0.4	0.3	2.3	49
	13 kg a.e. endothall/403	1.3	0.7	0	2.3	44	2.1	0.5	0.3	2.3	44
	10 kg a.e. endothall/403	1.5	0.6	0	2.3	64	1.7	0.6	0.3	2.4	64
	40 kg a.e. endothall	1.5	0.9	0	3.3	34	2.6	0.8	0.9	5.2	34
	Lake reference	1.4	0.5	0	2.7	41	3.2	0.1	2.9	3.3	41
	River reference	0.7	0.4	0	2.3	90	1.5	0.5	0.6	2.6	90
Day 56	197 kg a.e. poly GMA-2,4-D in clay	1.7	0.3	0	1.8	26	1.7	0.1	1.5	1.8	26
	72 kg a.e. poly GMA-2,4-D in clay	1.6	0.8	0	2.3	29	2.2	0.1	2.1	2.4	29
	178 kg a.e. poly GMA-2,4-D in clay	0.3	0.5	0	1.4	48	1.0	0.4	0.3	1.4	48
	112 kg a.e. 2,4-D in lignin	0.5	0.4	0	1.5	32	1.2	0.3	0.9	1.8	32
	20 kg a.e. endothall/Spra-mate	0.7	0.7	0	2.3	52	1.9	0.3	0.9	2.4	52
	13 kg a.e. endothall/Spra-mate	1.5	0.5	0.1	2.1	31	1.6	0.2	1.2	1.8	31
	10 kg a.e. endothall/Spra-mate	1.0	0.5	0	1.7	36	1.2	0.3	0.9	1.7	36
	20 kg a.e. endothall/403	1.0	0.4	0.3	1.8	40	1.5	0.2	0.6	1.8	40
	13 kg a.e. endothall/403	0.9	0.6	0	1.8	26	1.7	0.3	0.6	1.8	26
	10 kg a.e. endothall/403	1.1	0.5	0	1.8	23	1.3	0.5	0.3	1.8	23
	40 kg a.e. endothall	1.2	0.7	0	2.4	30	2.1	0.9	0.6	3.9	30
	Lake reference	1.1	0.5	0	2.7	40	2.8	0.2	7.4	3.2	40
	River reference	0.6	0.4	0	1.7	34	1.2	0.3	0.3	1.8	34

Table 8

Mean Herbicide Residue Values for the 1981 Controlled-Release Plots (n = 3)*

	197 kg a.e. Poly GMA-2,4-D in Clay/ha				72 kg a.e. Poly GMA-2,4-D in Clay/ha				178 kg a.e. Poly GMA-2,4-D in Clay/ha				112 kg a.e. 2,4-D in Lignin/ha			
Date	Mean	Standard Deviation	Min-imum	Max-imum	Mean	Standard Deviation	Min-imum	Max-imum	Mean	Standard Deviation	Min-imum	Max-imum	Mean	Standard Deviation	Min-imum	Max-imum
Pretreatment	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Day 0	25.9	18.5	15.1	47.4	7.7	7.5	1.9	16.1	1.1	1.3	0.2	2.3	24.3	41.0	0.2	71.7
Day 1	8.5	6.7	1.5	14.9	18.5	24.5	0.2	46.3	0.4	0.7	0	1.2	16.2	10.1	5.2	25.1
Day 2	--	--	--	--	--	--	--	--	0	0	0	0	--	--	--	--
Day 4	0.5	0.4	0.4	0.6	0.3	0.2	0	0.3	0	0	0	0	9.7	3.4	7.2	13.5
Day 8	0.4	0.1	0.3	0.4	0.3	0.1	0.3	0.3	1.3	0.1	1.2	1.3	7.9	11.1	0.8	20.8
Day 16	0.7	0.2	0.4	0.8	0.3	0.1	0.2	0.4	0.5	0.1	0.4	0.6	2.6	2.3	0.7	5.0
Day 32	0.3	0.1	0.2	0.3	0.3	0.1	0.3	0.3	0.4	0.1	0.3	0.5	1.3	0.2	1.1	1.5
Day 64	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0

* Values in micrograms per litre.

Table 9

Mean Herbicide Residue Values for the 1981 Endothall/Invert Plots (n = 3)*

Plot	Before Treatment				Treatment Day				Day 1			
	Mean	Standard Deviation	Minimum	Maximum	Mean	Standard Deviation	Minimum	Maximum	Mean	Standard Deviation	Minimum	Maximum
40 kg a.e. endothall acid/ha	0	0	0	0	12.4	21.4	0	37.1	0	0	0	0
20 kg a.e. endothall/403 inverting oil/ha	0	0	0	0	37.5	6.5	30.0	41.2	16.2	10.1	0	22.1
13 kg a.e. endothall/403 inverting oil/ha	0	0	0	0	317.7	550.0	0	953.0	0	0	0	0
10 kg a.e. endothall/403 inverting oil/ha	0	0	0	0	0	0	0	0	0	0	0	0
20 kg a.e. endothall/Spra-mate invert emulsion/ha	0	0	0	0	30.0	43.6	0	80.0	0	0	0	0
13 kg a.e. endothall/Spra-mate invert emulsion/ha	0	0	0	0	40.0	41.6	0	80.4	5.4	9.3	0	16.1
10 kg a.e. endothall/Spra-mate invert emulsion/ha	0	0	0	0	0	0	0	0	0	0	0	0

Plot	Day 2				Day 4				Day 8			
	Mean	Standard Deviation	Minimum	Maximum	Mean	Standard Deviation	Minimum	Maximum	Mean	Standard Deviation	Minimum	Maximum
40 kg a.e. endothall acid/ha	0	0	0	0	0	0	0	0	0	0	0	0
20 kg a.e. endothall/403 inverting oil/ha	0	0	0	0	0	0	0	0	0	0	0	0
13 kg a.e. endothall/403 inverting oil/ha	0	0	0	0	0	0	0	0	0	0	0	0
10 kg a.e. endothall/403 inverting oil/ha	0	0	0	0	0	0	0	0	0	0	0	0
20 kg a.e. endothall/Spra-mate invert emulsion/ha	0	0	0	0	0	0	0	0	0	0	0	0
13 kg a.e. endothall/Spra-mate invert emulsion/ha	0	0	0	0	0	0	0	0	0	0	0	0
10 kg a.e. endothall/Spra-mate invert emulsion/ha	0	0	0	0	0	0	0	0	0	0	0	0

* Values in micrograms per litre.

Table 10

Herbicide Concentrations Outside of 1981 Test Plots (n = 1)*

Date	Sample Location	Herbicide	Herbicide Concentration μg/ℓ	Date	Sample Location	Herbicide	Herbicide Concentration μg/ℓ
Day 0	A1	Endothall	0	Day 2	A1	Endothall	0
		2,4-D	0			2,4-D	0
	A2	Endothall	0		A2	Endothall	0
		2,4-D	0			2,4-D	3.92
	A3	2,4-D	0		A3	2,4-D	0.24
	A4	Endothall	0		A4	Endothall	0
	A5	Endothall	18.6		A5	Endothall	0
	A6	Endothall	0		A6	Endothall	0
	A7	Endothall	0		A7	Endothall	0
	A8	2,4-D	3.53		A8	2,4-D	1.86
Day 1	A1	Endothall	0	Day 3	A1	2,4-D	0
		2,4-D	0		A2	2,4-D	0
	A2	Endothall	11.5		A3	2,4-D	0
		2,4-D	0.27				
	A3	2,4-D	3.0	Day 4	A8	2,4-D	0
	A4	Endothall	1.2	Day 8	A8	2,4-D	0
	A5	Endothall	0	Day 16	A8	2,4-D	0
	A6	Endothall	0	Day 32	A8	2,4-D	0.31
	A7	Endothall	0	Day 64	A8	2,4-D	0
	A8	2,4-D	3.26				

* See Figure 2 for sample locations.

Table 11

Concentration of 2,4-D in Sediment for the 1981 Controlled-Release Plots*

Date	197 kg a.e. Poly GMA-2,4-D in Clay/ha (Lake)					178 kg a.e. Poly GMA-2,4-D in Clay/ha (River)					112 kg a.e. 2,4-D in Lignin/ha				
	n	Mean	Standard Deviation	Minimum	Maximum	n	Mean	Standard Deviation	Minimum	Maximum	n	Mean	Standard Deviation	Minimum	Maximum
Pretreatment	3	0	0	0	0	3	0	0	0	0	3	0	0	0	0
Day 32	4	182.2	92.2	92.2	29.5	3	9.0	7.9	0.1	15.1	3	1.7	1.4	0.1	2.8
Day 64	2	157.5	25.6	138.0	177.0	3	0	0	0	0	3	0	0	0	0

* Values in micrograms per kilogram.

Table 12

Mean M. spicatum Biomass Values for the 1982 Test Plots (n = 15)

Date	Plot	Wet Weight, g/m^2 Mean	Standard Deviation	Minimum	Maximum	n	Dry Weight g/m^2*	Ash-Free Dry Weight g/m^2**
Pretreatment	River reference	5,104.4	1,632.8	1632.9	7,076.0	15	731.2	301.6
	Lake reference	1,886.8	689.6	1088.6	3,447.2	15	267.9	108.5
	River - 2,4-D DMA/invert	3,756.0	1,432.4	1632.9	6,803.8	15	537.1	220.7
	Lake - 2,4-D DMA/polymer	3,084.4	888.8	725.7	5,080.2	15	440.4	180.4
	Lake - 2,4-D DMA/invert	2,358.8	1,016.0	181.4	3,991.6	15	335.9	136.8
Day 32	River reference	4,536.0	2,540.0	1360.8	8,618.2	15	653.2	267.5
	Lake reference	1,801.8	1,016.0	453.6	4,535.9	15	255.7	103.4
	River - 2,4-D DMA/invert	2,540.0	1,923.2	362.9	6,350.3	15	362.0	147.7
	Lake - 2,4-D DMA/polymer	1,542.0	671.2	725.7	2,721.5	15	218.3	87.8
	Lake - 2,4-D DMA/invert	1,324.4	943.6	362.8	3,810.2	15	186.9	74.7
Day 64	River reference	3,992.0	1,814.4	1360.8	8,708.9	15	571.1	234.8
	Lake reference	1,785.6	1,260.4	816.5	3,447.3	15	253.3	102.4
	River - 2,4-D DMA/invert	1,850.8	998.0	0	3,810.2	15	262.6	106.4
	Lake - 2,4-D DMA/polymer	2,177.2	780.0	453.6	3,175.1	15	309.7	125.9
	Lake - 2,4-D DMA/invert	1,482.8	1,230.8	365.9	4,535.9	15	209.7	84.2

* Value determined from the equation $y = 0.144(x) + (-3.78)$.
** Value determined from the equation $y = 0.06(x) + (-4.69)$.

Table 13

Mean *M. spicatum* Plant Height and Water Depth for the 1982 Test Plots

		Average Plant Height, m					Average Water Depth, m				
Date	Plot	Mean	Standard Deviation	Minimum	Maximum	n	Mean	Standard Deviation	Minimum	Maximum	n
Pretreatment	River reference	1.4	0.7	0.1	2.4	48	1.8	0.3	1.4	2.7	48
	River - 2,4-D BEE/granular	1.5	0.3	0.8	2.1	32	1.6	0.2	1.3	2.1	32
	River - 2,4-D DMA/polymer	0.9	0.8	0	2.4	48	1.3	0.6	0.6	2.7	48
	River - 2,4-D DMA/invert	1.5	0.8	0	3.0	80	1.9	0.5	1.2	3.2	80
	Lake reference	1.9	0.5	0	2.9	40	3.2	0.2	2.7	3.7	40
	Lake - 2,4-D DMA/polymer	2.2	0.6	0.6	2.7	30	2.4	0.3	1.5	2.7	30
	Lake - 2,4-D DMA/invert	2.7	0.5	0	2.9	33	2.8	0.1	2.4	2.9	33
Day 32	River reference	1.5	0.7	0	2.4	46	1.9	0.3	1.5	2.4	46
	River - 2,4-D BEE/granular	0.7	0.5	0	1.7	38	1.5	0.4	0.6	2.1	38
	River - 2,4-D DMA/polymer	0.7	0.6	0	1.8	48	1.3	0.3	0.8	1.8	48
	River - 2,4-D DMA/invert	1.3	0.7	0	2.7	64	1.7	0.4	0.9	2.7	64
	Lake reference	2.2	0.5	0	3.2	40	3.2	0.2	2.7	3.7	40
	Lake - 2,4-D DMA/polymer	2.1	0.5	0.3	2.7	40	2.5	0.2	1.8	2.7	40
	Lake - 2,4-D DMA/invert	2.3	0.9	0	3.0	35	2.9	0.1	2.7	3.0	35
Day 64	River reference	1.3	0.6	0	2.1	42	1.9	0.4	1.2	2.7	42
	River - 2,4-D BEE/granular	0.3	0.3	0	0.9	32	1.5	0.5	0.6	2.1	32
	River - 2,4-D DMA/polymer	0.2	0.3	0	1.2	42	1.3	0.4	0.8	2.1	42
	River - 2,4-D DMA/invert	0.9	0.6	0	2.1	59	1.7	0.5	0.9	3.0	59
	Lake reference	2.1	0.5	0.9	3.2	43	3.2	0.2	2.7	3.7	43
	Lake - 2,4-D DMA/polymer	1.9	0.5	0.5	2.7	31	2.4	0.2	1.8	2.7	31
	Lake - 2,4-D DMA/invert	1.9	1.1	0	2.9	38	2.9	0.1	2.7	3.0	38

Table 14

Mean Herbicide Residue Values* in Water for the 1982 Test Plots

Okanogan River

Date	2,4-D BEE/Granular** Mean	Standard Deviation	Minimum	Maximum	n	2,4-D DMA/Polymer† Mean	Standard Deviation	Minimum	Maximum	n	2,4-D DMA/Invert Mean	Standard Deviation	Minimum	Maximum	n
Within the Plot															
Pretreatment	0	0	0	0	1	0	0	0	0	1	0	0	0	0	1
0-30 min	1.4	0.2	1.2	1.6	3	3.7	6.3	0	11	3	20.1	29.4	0	53.8	3
31-60 min	0	0	0	0	3	0	0	0	0	4	20.1	34.8	0	60.2	3
1-2 hr	0	0	0	0	3	9.0	2.2	7.1	11.4	3	24.5	45.5	0	116.0	6
2-4 hr	1.1	1.5	0	2.8	3	6.5	6.7	0	13.4	3	1.2	2.0	0	3.5	3
4-8 hr	0	0	0	0	3	0	0	0	0	3	0	0	0	0	2
Day 1	0	0	0	0	3	0	0	0	0	3	0	0	0	0	3
Day 2	0	0	0	0	3	0	0	0	0	3	0	0	0	0	3
Day 3	13.2	8.5	3.4	18.4	3	--	--	--	--	--	--	--	--	--	--
Day 4	0	0	0	0	3	0	0	0	0	3	0	0	0	0	3
Day 8	0	0	0	0	3	0	0	0	0	3	0	0	0	0	3
Day 16	0	0	0	0	3	0	0	0	0	3	0	0	0	0	3
Day 32	0	0	0	0	3	0	0	0	0	3	0	0	0	0	3
Downstream of Plot															
0-30 min	7.5	13.1	0	27.1	4	68.7	58.1	5.5	158.0	5	5.6	9.6	0	22.3	5
31-60 min	2.8	2.1	0	5.2	4	61.6	84.1	0	183.0	6	11.0	10.7	0	22.6	5
1-2 hr	1.6	2.8	0	4.9	4	0.9	2.3	0	5.6	6	1.0	2.0	0	4.0	4
2-3 hr	--††	--	--	--	--	0	0	0	0	2	3.9	3.4	0	6.2	3
3-4 hr	0	0	0	0	3	0	0	0	0	3	0	0	0	0	4

Lake Osoyoos

Date	2,4-D DMA/Polymer Mean	Standard Deviation	Minimum	Maximum	n	2,4-D DMA/Invert Mean	Standard Deviation	Minimum	Maximum	n
Within the Plot										
Pretreatment	0	0	0	0	1	0	0	0	0	1
0-30 min	573.3	246.4	369.0	953.0	6	--	--	--	--	--
31-60 min	426.6	234.2	55.4	742.0	6	--	--	--	--	--
1-2 hr	222.0	186.9	91.0	436.0	3	194.4	159.5	13.2	314.0	3
2-4 hr	0	0	0	0	3	--	--	--	--	--
4-8 hr	30.9	12.3	19.8	44.2	3	129.0	143.3	35.6	294.0	3
Day 1	1.6	2.8	0	4.8	3	78.2	71.3	14.1	155.0	3
Day 2	0	0	0	0	3	18.8	9.6	8.7	27.8	3
Day 3	--	--	--	--	--	--	--	--	--	--
Day 4	0	0	0	0	3	5.4	5.4	0	10.9	3
Day 8	0	0	0	0	3	0	0	0	0	3
Day 16	0	0	0	0	3	0	0	0	0	3
Day 32	0	0	0	0	3	0	0	0	0	3

Lake Osoyoos – Outside of Plot

Date	North Mean ± 50	n	South Mean ± 50	n	North, n = 1
0-30 min	0.8 ± 1.6	4	--	--	--
31-60 min	31.4 ± 40.5	4	--	--	--
1-2 hr	77.4 ± 65.6	5	8.81 ± 51.9	3	--
2-3 hr	38.0 ± 0	1	34.0 ± 0	1	194.4
3-4 hr					--
4-8 hr					129.9
Day 1					78.2
Day 2					18.8
Day 4					5.4
Day 5					0
Day 6					0

* In micrograms per litre.
** Herbicide residue was detected at only one downstream sampling station. Values are reported for only that station.
† Twenty-six minutes after the 2,4-D DMA/polymer application, 890 ± 487.1 µg/ℓ of 2,4-D acid was detected between the 2,4-D BEE/granular and 2,4-D DMA/polymer plot.
†† Not sampled.

Table 15

Mean Herbicide Residue Values* in *M. spicatum* for the 1982 Test Plot (n = 3)

River

Date	2,4-D BEE/Granular Mean	2,4-D BEE/Granular Standard Deviation	2,4-D BEE/Granular Minimum	2,4-D BEE/Granular Maximum	2,4-D DMA/Polymer Mean	2,4-D DMA/Polymer Standard Deviation	2,4-D DMA/Polymer Minimum	2,4-D DMA/Polymer Maximum	2,4-D DMA/Invert Mean	2,4-D DMA/Invert Standard Deviation	2,4-D DMA/Invert Minimum	2,4-D DMA/Invert Maximum
Pretreatment	0	0	0	0	0	0	0	0	0	0	0	0
30 min	897.3	1169.5	0	2220.0	104.6**	135.7	0	328.0	2160.2**	2551.3	371.0	6610.0
1 hr	531.7	428.6	218.0	1020.0	49.2	64.3	0	122.0	577.1	406.2	186.2	997
2 hr	881.3	138.1	725.0	987.0	13.4	21.5	1.0	38.2	524.0	569.1	1.0	1130.0
4 hr	941.0	946.5	0	1893.0	87.7	95.1	24.2	197.0	158.0	140.8	1.0	273
8 hr	--	--	--	--	--	--	--	--	--	--	--	--
1 day	147.4	71.8	64.8	195.0	502.0	396.4	178.0	944.0	127.1	62.4	59.2	181.9
2 days	194.4††	184.6	63.9	325.0	161.7	43.2	121.0	207.0	79.0	6.1	74.8	86.0
3 days	118.6	116.2	23.1	248.0	--	--	--	--	--	--	--	--
4 days	638.6	791.1	128.0	1550.0	55.0††	76.4	1.0	109.0	18.6	30.5	1	53.9
8 days	64.2††	24.5	46.9	81.5	207.3	196.1	0	390.0	1.0	0.5	0	1.4
16 days	52.1	28.8	34.6	85.4	0	0	0	0	0	0	0	0
32 days	29.2	50.5	0	87.5	0	0	0	0	540.6	858.1	0	1530.0
64 days	0	0	0	0	0	0	0	0	0	0	0	0

Lake

Date	2,4-D DMA/Polymer Mean	2,4-D DMA/Polymer Standard Deviation	2,4-D DMA/Polymer Minimum	2,4-D DMA/Polymer Maximum	2,4-D DMA/Invert Mean	2,4-D DMA/Invert Standard Deviation	2,4-D DMA/Invert Minimum	2,4-D DMA/Invert Maximum
Pretreatment	0	0	0	0	0	0	0	0
30 min	1890.0	121.6	1750.0	1970.0	--†	--	--	--
1 hr	1856.3	1858.8	139	3830.0	--	--	--	--
2 hr	--	--	--	--	398.3	93.1	312.0	497.0
4 hr	299.3	167.1	107.0	409.0	623.3	402.3	321.0	1080
8 hr	--	--	--	--	284.3	183.6	134.0	489.0
1 day	165.7	166.5	62.8	357.8	434.9	604.1	36.7	1130
2 days	342.0††	72.1	291.0	393.0	591.6††	605.8	163.2	1020
3 days	--	--	--	--	--	--	--	--
4 days	112.2	55.3	54.6	165.0	318.0††	113.1	238	398
8 days	198.0	110.9	130	326	210.5	177.2	62.6	407.0
16 days	25.4††	34.6	1	49.9	31.6	19.0	28.9	44.2
32 days	29.5	26.7	0	52.0	31.9	29.8	1.0	60.5
64 days	0	0	0	0	0	0	0	0

* In micrograms per kilogram.
** n = 6.
† Not sampled.
†† n = 2.

Table 16

Mean Herbicide Residue Values* in Sediment for the 1982 Test Plots (n = 3)

	River												Lake							
	2,4-D BEE/Granular				2,4-D DMA/Polymer				2,4-D DMA/Invert				2,4-D DMA/Polymer				2,4-D DMA/Invert			
Date	Mean	Standard Deviation	Minimum	Maximum	Mean	Standard Deviation	Minimum	Maximum	Mean	Standard Deviation	Minimum	Maximum	Mean	Standard Deviation	Minimum	Maximum	Mean	Standard Deviation	Minimum	Maximum
Pretreatment	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	13.7	23.8	0	41.2
30 min	343.3	489.6	0	904.0	0	0	0	0	0	0	0	0	--**	--	--	--	--	--	--	--
1 hr	862.7	840.4	0	1,679.0	25.0	43.3	0	75.0	0	0	0	0	0	0	0	0	--	--	--	--
2 hr	1,331.7	2,216.2	0	3,890.0	0	0	0	0	29.3	50.8	0	88.0	0	0	0	0	33.3	57.7	0	100.0
4 hr	1,741.3	2,883.7	0	5,070.0	19.0	32.9	0	57.0	0	0	0	0	--	--	--	--	--	--	--	--
1 day	268.0	365.5	52.0	690.0	0	0	0	0	0	0	0	0	0	0	0	0	40.0	69.3	0	120.0
2 days	4,333.3	4,454.6	0	8,900.0	44.7	77.4	0	134.0	0	0	0	0	0	0	0	0	0	0	0	0
3 days	4,136.7	6,292.4	340.0	11,400.0	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
4 days	132.0	228.6	0	396.0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
8 days	340.0†	184.6	210.0	471.0	63.3	109.7	0	190.0	0	0	0	0	0	0	0	0	0	0	0	0
16 days	2,336.7	4,047.2	0	7,010.0	0	0	0	0	--	--	--	--	--	--	--	--	--	--	--	--
32 days	80.7	91.4	0	180.0	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
64 days	0	0	0	0	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--

* In micrograms per kilogram.
** Not sampled.
† n = 2.

Table 17

Summary of Water Velocity Values for the 1982 Test Plots

Date	Plot	Cross-Sectional Area of Plot, m^2	Average Stream Velocity in Plot cms	Average Stream Velocity Outside Plot cms (n = 2)	Flow Rate in Plot m^3/sec	Discharge at USGS Gaging Station at Zosel Mill Dam, m^3/sec
Day 0	Reference	51.7	3.0 ± 2.6 (6)*	19.8 ± 10.6	1.6	36.8
	2,4-D BEE/granular	43.2	15.3 ± 4.3 (6)	35.0 ± 10.7	6.4	36.8
	2,4-D DMA/polymer	89.4	13.7 ± 5.6 (8)	25.9 ± 10.7	12.2	39.9
	2,4-D DMA/invert	47.6	9.1 ± 4.7 (6)	22.8 ± 6.4	4.3	39.9
Day 8	Reference	55.7	7.3 ± 9.6 (4)	19.8 ± 10.8	4.1	47.8
	2,4-D BEE/granular	49.4	18.2 ± 4.3 (6)	38.1 ± 2.1	9.0	47.8
	2,4-D DMA/polymer	93.5	16.2 ± 15.0 (6)	33.5 ± 21.5	15.1	47.8
	2,4-D DMA/invert	60.3	9.4 ± 4.2 (6)	21.3 ± 12.9	5.7	47.8
Day 16	Reference	50.1	5.7 ± 5.3 (6)	18.2 ± 4.3	2.8	35.4
	2,4-D BEE/granular	39.2	21.6 ± 13.8 (6)	35.0 ± 6.5	8.4	35.4
	2,4-D DMA/polymer	60.0	19.8 ± 6.6 (6)	28.9 ± 10.8	11.9	38.5
	2,4-D DMA/invert	44.6	7.4 ± 3.9 (6)	18.2 ± 12.9	3.3	38.5

* Numbers in parentheses indicate sample observation number.

APPENDIX A: WATER QUALITY MEASUREMENTS

Table A1

Mean and Standard Deviation of Water Quality Values for the 1980 Test Plots

Date	Plot	Temperature °C	Conductivity μmhos/cm	Dissolved Oxygen mg/ℓ	pH	Secchi Disk, m (n = 3)
			Water Quality Values by Date (n = 9)			
Pretreatment	Reference	22.8 ± 0.4	246 ± 8.0	11.3 ± 0.5	8.6 ± 0.1	1.1 ± 0.1
	2,4-D BEE/granular	21.6 ± 0.3	272 ± 7.7	9.5 ± 0.5	8.6 ± 0.1	1.9 ± 0.1
	2,4-D DMA/polymer	21.5 ± 0.5	279 ± 12.9	9.5 ± 0.9	8.7 ± 0.1	1.8 ± 0.1
	Diquat/polymer	22.1 ± 0.1	237 ± 0.4	7.8 ± 0.9	8.6 ± 0.1	1.8 ± 0.0
	Endothall/polymer	22.1 ± 0.2	283 ± 6.2	9.6 ± 0.2	8.7 ± 0.0	1.8 ± 0.5
Day 0	Reference	26.2 ± 0.1	273 ± 2.7	9.6 ± 0.8	8.6 ± 0.1	0.9 ± 0.1
	2,4-D BEE/granular	25.6 ± 0.1	270 ± 1.0	10.2 ± 0.4	8.6 ± 0.1	1.5 ± 0.0
	2,4-D DMA/polymer	26.3 ± 0.3	272 ± 15.2	10.1 ± 0.2	8.6 ± 0.1	1.8 ± 0.1
	Diquat/polymer	27.7 ± 0.1	275 ± 2.3	10.1 ± 0.2	8.6 ± 0.1	1.8 ± 0.2
	Endothall/polymer	24.7 ± 0.2	272 ± 2.6	9.3 ± 0.6	8.6 ± 0.1	1.3 ± 0.1
Day 7	Reference	26.1 ± 0.3	246 ± 5.6	9.7 ± 0.1	8.4 ± 0.1	0.8 ± 0.1
	2,4-D BEE/granular	25.1 ± 0.2	256 ± 2.1	8.5 ± 0.9	8.6 ± 0.1	0.9 ± 0.1
	2,4-D DMA/polymer	25.2 ± 0.1	241 ± 3.4	9.4 ± 0.3	8.6 ± 0.0	0.8 ± 0.2
	Diquat/polymer	25.1 ± 0.1	242 ± 1.7	9.6 ± 0.5	8.7 ± 0.1	1.0 ± 0.1
	Endothall/polymer	25.2 ± 0.1	245 ± 1.1	9.7 ± 0.2	8.6 ± 0.1	1.1 ± 0.1
Day 14	Reference	23.1 ± 0.1	245 ± 1.5	8.8 ± 0.7	8.6 ± 0.1	1.1 ± 0.1
	2,4-D BEE/granular	22.6 ± 0.1	233 ± 1.3	8.2 ± 0.5	8.6 ± 0.1	1.0 ± 0.0
	2,4-D DMA/polymer	22.6 ± 0.1	237 ± 1.3	8.6 ± 0.4	8.6 ± 0.1	1.2 ± 0.0
	Diquat/polymer	22.8 ± 0.1	230 ± 1.1	9.4 ± 0.3	8.6 ± 0.0	1.3 ± 0.1
	Endothall/polymer	23.0 ± 0.2	231 ± 1.3	10.1 ± 0.6	8.7 ± 0.1	1.3 ± 0.1
Day 28	Reference	21.6 ± 0.2	240 ± 3.2	8.3 ± 0.1	8.2 ± 0.1	1.2 ± 0.0
	2,4-D BEE/granular*	--	--	--	--	--
	2,4-D DMA/polymer*	--	--	--	--	--
	Diquat/polymer	21.2 ± 0.1	239 ± 4.1	6.9 ± 1.2	7.9 ± 0.2	1.4 ± 0.1
	Endothall/polymer	22.1 ± 0.1	241 ± 1.3	8.1 ± 0.4	7.9 ± 0.1	1.3 ± 0.1
Day 56	Reference	17.9 ± 0.2	225 ± 6.2	8.6 ± 0.6	7.9 ± 0.1	1.5 ± 0.1
	2,4-D BEE/granular	17.7 ± 0.1	226 ± 0.4	9.0 ± 0.1	8.0 ± 0.0	1.4 ± 0.1
	2,4-D DMA/polymer	17.6 ± 0.3	233 ± 14.5	8.3 ± 0.2	8.0 ± 0.1	2.0 ± 0.2
	Diquat/polymer	18.9 ± 0.1	208 ± 3.2	7.2 ± 1.4	8.0 ± 0.1	2.0 ± 0.0
	Endothall/polymer	18.5 ± 0.1	206 ± 2.5	7.2 ± 0.9	8.1 ± 0.1	2.0 ± 0.0
Day 84	Reference	16.6 ± 0.1	229 ± 1.0	8.7 ± 0.3	8.1 ± 0.1	1.5 ± 0.1
	2,4-D BEE/granular	17.3 ± 0.2	223 ± 3.4	8.7 ± 1.4	8.2 ± 0.1	1.6 ± 0.3
	2,4-D DMA/polymer	16.8 ± 0.2	220 ± 4.6	8.9 ± 0.2	8.2 ± 0.1	1.3 ± 0.1
	Diquat/polymer	16.6 ± 0.1	230 ± 1.6	9.0 ± 0.4	8.1 ± 0.1	1.3 ± 0.2
			Water Quality for all Dates (n = 63)			
	Reference	21.7 ± 3.7	250 ± 4.5	8.9 ± 1.0	8.3 ± 0.3	1.2 ± 0.3
	2,4-D BEE/granular**	22.2 ± 3.1	252 ± 28.1	9.1 ± 0.8	8.5 ± 0.2	1.4 ± 0.4
	2,4-D DMA/polymer**	21.3 ± 3.9	245 ± 31.1	9.1 ± 0.7	8.4 ± 0.3	1.4 ± 0.4
	Diquat/polymer	22.1 ± 3.3	241 ± 22.3	8.4 ± 1.2	8.1 ± 0.3	1.6 ± 0.4
	Endothall/polymer	22.2 ± 2.8	250 ± 30.8	8.5 ± 1.2	8.2 ± 0.3	1.5 ± 0.5

* Data not taken.

** n = 45.

Table A2

Water Quality Values for the 1982 Test Plots

Date	Plot	Temperature °C	Conductivity µmhos/cm	Dissolved Oxygen mg/ℓ	pH	Total Alkinity mg/ℓ $CaCO_3$	Total Alkinity mg/ℓ $CaCO_3$	Turbidity JTU*	Photosynthetically Active Radiation (PAR) Above Water µE/sec/m^2	Photosynthetically Active Radiation 0.5 m Below Water Surface, µE/sec/m^2	Secchi Disk m
					Water Quality by Date (n = 1)						
Pretreatment	River reference	23.0	264	10.0	8.6	125	110	5	1600	400	2.5
	River - 2,4-D BEE/granular	21.8	250	9.4	8.5	122	120	5	4500	800	2.0
	River - 2,4-D DMA/polymer	21.4	246	9.0	8.5	130	115	5	6100	500	2.0
	River - 2,4-D DMA/invert	20.4	233	9.0	8.6	120	100	5	215	60	2.0
	Lake reference	23.3	252	10.0	8.6	125	110	5	2100	750	2.5
	Lake - 2,4-D DMA/polymer	21.2	240	10.0	8.8	130	100	10	620	150	2.0
	Lake - 2,4-D DMA/invert	20.7	246	11.5	8.6	115	95	5	1500	80	2.0
Day 16	River reference	23.7	233	9.0	8.6	120	100	5	550	250	2.0
	River - 2,4-D BEE/granular	24.4	225	9.1	8.8	120	100	5	550	110	1.3
	River - 2,4-D DMA/polymer	24.2	227	9.2	8.7	130	115	5	570	200	1.5
	River - 2,4-D DMA/invert	24.1	233	9.0	8.6	125	105	5	680	190	1.5
	Lake reference	23.8	235	8.4	8.6	135	110	5	850	220	2.3
	Lake - 2,4-D DMA/polymer	23.4	231	8.5	8.8	115	105	10	900	250	1.5
	Lake - 2,4-D DMA/invert	23.0	225	9.4	8.7	110	100	25	620	80	1.5
Day 32	River reference	22.9	225	8.2	8.9	120	105	5	1500	240	2.5
	River - 2,4-D BEE/granular	23.5	233	9.3	8.8	115	100	5	480	140	1.5
	River - 2,4-D DMA/polymer	23.5	231	9.0	8.8	115	100	5	1400	350	1.5
	River - 2,4-D DMA/invert	23.1	225	8.7	8.9	120	100	5	1350	500	1.5
	Lake reference	22.6	225	7.2	8.7	115	100	5	1500	420	2.5
	Lake - 2,4-D DMA/polymer	22.4	221	9.1	9.2	120	100	5	1500	500	2.5
	Lake - 2,4-D DMA/invert	22.1	219	8.6	8.4	120	100	5	1600	250	2.0
Day 64	River reference	18.4	217	9.6	8.5	130	110	7	1500	420	2.0
	River - 2,4-D BEE/granular	17.9	208	7.2	8.9	125	115	7	1500	850	1.8
	River - 2,4-D DMA/polymer	18.2	219	8.5	8.5	135	120	7	1500	875	1.8
	River - 2,4-D DMA/invert	18.3	219	8.9	8.5	130	115	7	1500	840	1.8
	Lake reference	18.5	217	8.5	8.5	125	105	10	1500	400	2.0
	Lake - 2,4-D DMA/polymer	16.9	213	8.5	9.0	115	100	20	3100	950	2.0
	Lake - 2,4-D DMA/invert	16.8	217	7.2	8.3	135	115	20	3000	990	2.0
		Water Quality for all Dates (n = 4)							Average Percent Reduction of PAR (n = 28)		
All dates for river plots	River reference	22.0 ± 2.4	235 ± 20.5	8.3 ± 1.3	8.6 ± 0.2	126.2 ± 7.5	106.2 ± 4.8	5.5 ± 1.0	71.2 ± 2.5	2.25 ± 0.3	
	River - 2,4-D BEE/granular	21.9 ± 2.8	229 ± 17.4	8.7 ± 9.0	8.7 ± 0.2	120.5 ± 4.2	108.8 ± 10.3	6.2 ± 2.5	69.0 ± 18.0	1.7 ± 0.3	
	River - 2,4-D DMA/polymer	21.8 ± 2.7	231 ± 11.3	8.9 ± 0.3	8.7 ± 0.1	127.5 ± 8.7	127.5 ± 8.7	6.0 ± 2.0	68.5 ± 20.1	1.7 ± 0.2	
	River - 2,4-D DMA/invert	21.5 ± 2.6	228 ± 6.8	9.0 ± 0.1	8.6 ± 0.2	123.7 ± 4.8	105.0 ± 7.1	6.0 ± 2.0	62.7 ± 13.2	1.7 ± 0.2	
All dates for lake plots	Lake reference	22.1 ± 2.4	232 ± 15.1	8.5 ± 1.1	8.6 ± 0.1	125.0 ± 8.2	106.2 ± 4.8	6.2 ± 2.5	70.1 ± 4.6	2.3 ± 0.2	
	Lake - 2,4-D DMA/polymer	20.1 ± 2.9	226 ± 11.8	9.0 ± 0.7	8.9 ± 0.2	120.0 ± 7.1	101.2 ± 2.5	11.2 ± 6.0	73.5 ± 7.1	2.0 ± 0.4	
	Lake - 2,4-D DMA/invert	20.1 ± 2.7	227 ± 13.3	9.2 ± 1.8	8.5 ± 0.2	120.0 ± 10.8	102.5 ± 8.7	13.7 ± 10.3	70.1 ± 3.5	1.8 ± 0.2	

* JTU = Jackson Turbidity Units.

APPENDIX B: HERBICIDE ANALYSIS TECHNIQUES AND RECOVERY RATES

Introduction

The 1980 herbicide analyses were conducted by AmTest, Inc., using the techniques referenced. The 1981 and 1982 herbicide analyses were conducted by the Washington State Pesticide Laboratory using their techniques described herein. Table B1 provides recovery rates for 1980-1982.

Herbicide Analysis References for 1980

References used for 1980 herbicide analysis are listed below.

2,4-D

U. S. Environmental Protection Agency, 1979 (Mar). "EPA Method 615 for Chlorinated Herbicides," Interim Methods, Attachment C, Cincinnati, Ohio.

Diquat

U. S. Department of Health, Education, and Welfare. 1977. "FDA Pesticide Analytical Manual Vol. II," Diquat Method A, Washington, D. C.

Endothall

Carlson, R., Whitaker, R., and Landskov, A. 1978. Analytical Methods for Pesticides and Plant Growth Regulators, Academic Press, New York, pp 327-340.

Herbicide Analysis Techniques for 1981
Endothall in Water

1. Pour 200 ml sample into 400-ml beaker. Add boiling chip. For recovery standard, pour 200 ml distilled water into a 400-ml beaker. Add 20 µg of endothall standard by pipeting 1.0 ml of a standard solution of endothall.
2. Add 2 drops of HCl.
3. Evaporate sample to 25-50 ml on a hot plate.
4. Add 100 ml glacial acetic acid.
5. Evaporate to 25-50 ml on a hot plate.
6. Add 25 ml glacial acid.
7. Evaporate to 10-15 ml on a hot plate.
   Note: Do not let sample go to dryness.
8. Add 100 mg 2-chloroethylamine hydrochloride and 100 mg of sodium acetate.

9. Heat for 1-1/2 hr with hot plate on low setting.

10. Use 75 ml distilled water to transfer sample to a 125-ml separatory funnel.

11. Extract sample four times with 20-ml portions of dichloromethane. Combine extracts in a 125-ml Erlenmeyer flask.

12. Transfer extract to a 125-ml separatory funnel.

13. Wash sample with 30 ml of 0.1 N NaOH and again with 30 ml of distilled water.

14. Transfer washed extract to KD concentrator flask.

15. Evaporate to 3-5 ml on steam bath.

16. Continue to evaporate just to dryness on N-evap.

17. Add 1.0 ml of methanol to dissolve sample residue.

18. *Analyze by gas chromatography (GC). The minimum reporting level is 1 μg/ℓ.

* Must use Hall Detector on halogen mode.

Herbicide Analysis Techniques for 1982

2,4-D in water

1. Accurately measure approximately 1000 ml of water in a graduated cylinder. Record the volume of sample. Add deionized, carbon-filtered water to make a final volume of 100 ml.

2. Transfer the water to a 200-ml separatory funnel and add 3 ml of concentrated sulfuric acid. Swirl the contents of the funnel to mix.

3. Add 175 ml of diethyl ether to the funnel and shake for 1 min.

4. Drain the water into a beaker.

5. Drain the ether layer into a 250-ml Erlenmeyer flask and add a 10-ml beaker full of anhydrous Na_2SO_4. Let stand for 30 min.

6. Quantitatively transfer the ether to a KD concentrator fitted with a 10-ml tube.

7. Using the steam cabinet concentrate the ether to about 3 ml.

8. Quantitatively transfer the ether to a 12-ml graduated centrifuge tube.

9. Using the water bath and a stream of nitrogen take the ether almost (but not quite) dry.

10. Make the extract to 1 ml with methyl alcohol.

11. Analyze by high pressure liquid chromatography (HPLC). Minimum reporting level is approximately 1 μg/ℓ.

2,4-D in plants

1. Weigh out 5 to 10 g of plant material and note exact amount. Place in 1-ℓ Erlenmeyer and add 10 ml of 25% H_2SO_4. Add 100 ml of diethyl ether and 100 ml petroleum ether and shake, on a wrist shaker, for 15 min.
2. Decant ether solvents through 2-in. Na_2SO_4 column into KD flask fitted with 10-ml concentrator. A second diethyl ether extraction should be made by adding 100 ml ether, shake for 20 min on shaker, and decant into KD. (To avoid H_2SO_4 getting into Na_2SO_4 column, you may wish to decant into 50-ml graduated cylinder to separate ether/aqueous phases.)
3. Blow down ether in concentrator tube to 0.5 ml. Add about 1.5 ml CH_3OH/borontrifluoride mixture, mix well, and allow to stand at 50°C for 30 min with tightly fitted stopper in water bath.
4. Add 5 ml of 5% Na_2SO_4 solution to derivatized solution and extract with 2 ml benzene portions with 1 min of shaking and repeat for a total of three extractions. Combine all three extracts in 15-ml centrifuge tubes and add 0.25 g Na_2SO_4. Allow to stand 30 min while making florisil columns.
5. Prepare florisil columns in capillary pipettes; place about 4 cm of florisil and 2 cm of Na_2SO_4 in the pipette, which has been fitted with a small glass wool retaining plug. (Make wool plug as small as possible.)
6. Add phenoxyacid-ester solution to column and rinse several times with 0.5 ml benzene rinses and collect the first 5.0 ml of the eluate.
7. Analyze by GC. Use 175°C column temperature with OV17/OV210 and/or SE30/OV210 columns. The minimum reporting level is approximately 1.0 µg/ℓ. The retention time for 2,4-D ME ester is 6 min.

2,4-D in sediment

1. Place 15-25 g accurately weighed soil sample into a 500-ml Erlenmeyer glass-stoppered flask. Weigh another sample and dry to determine percent moisture.
2. Prepare a spike sample with 2.0 µg 2,4-D, which is added after 10.0 g clean substrate is moistened with distilled water.
3. Add 10 ml of 25% H_2SO_4 to each soil or sediment sample. Add 20 ml methanol and mix sample well.
4. Add 100 ml of 50% ethyl ether and 50% petroleum ether and 5 g Na_2SO_4.
5. Place on shaker for 20 min. Be sure to agitate strongly enough to ensure that soil and aqueous layers are thrown into the organic phase with each throw of the wrist-action shaker.

6. Decant solvent into KD which is fitted with a 25-ml concentrator tube. Pass solvent through Na_2SO_4; 2-in. depth in 25-mm chromatography column (no stopcock).

7. If emulsion forms, then centrifuge slurry to separate organic phase.

8. Repeat steps 3 through 7.

9. Rinse twice with 50-ml portions of 50% ethyl ether/petroleum ether and combine with extract in KD flask.

10. Add 1 ml benzene and concentrate on stream table to about 10 ml and continue concentrating to 0.5 ml on N_2 bath. (Stop and cap, if needed.)

11. Add 1 ml boron trifluoride/methanol reagent, mix on vortex mixer, and place in 50°C water bath in tightly stoppered tube for 30 min.

12. Remove from water bath and allow to cool. Add 5 ml of 5% Na_2SO_4 solution. Add 3 ml benzene and gently agitate for 1 min and allow to separate. Repeat extraction once more with 1 ml benzene.

13. Combine benzene extracts in concentrator tube and prepare a florisil column. Using a disposable pipette, place a loose glass wool plug, 3 cm of florisil, and 2 cm of Na_2SO_3 in the column.

14. Add the benzene extract to this column and rinse through with 0.5 ml benzene rinses. Collect first 5.0 ml of benzene eluate. The 2,4-D methyl ester will be eluted in this fraction.

15. The 2,4-D will elute in 6 min. Analyze by GC. Use 180°C, from an OV17/OV210 column. The minimum reporting level is approximately 50 μg/ℓ.

Table B1

Herbicide Analysis Recovery Rates

Date	Herbicide	Sample Type	Number of Samples*	Quality Control Spike Samples (Percent Recovery) n	Mean	Standard Deviation
1980	2,4-D	Water	225	8	78.7	15.3
	Endothall	Water	85	9	89.8	21.1
	Diquat	Water	101	7	79.9	11.0
1981	2,4-D	Water	122	19	94.3	3.5
	2,4-D	Sediment	26	2	78.0	4.2
	Endothall	Water	137	33	83.8	20.4
1982	2,4-D	Water	345	38	82.6	11.9
	2,4-D	Sediment	161	17	66.4	14.9
	2,4-D	Plant	300	22	84.6	12.9

* Sample number includes within treatment plot, outside of treatment plot, reference plot, and water intake samples.

APPENDIX C: LINEAR CORRELATION AND PERCENT MOISTURE FOR SELECTED AQUATIC PLANTS SAMPLED IN 1980

Linear Correlation and Percent Moisture for Selected Aquatic Plants Sampled in 1980

	Wet Weight (x) - Dry Weight (y)			Wet Weight (x) - Ash-Free Dry Weight (y)			
Species	Equation	R^2	n	Equation	R^2	n	Percent Moisture
M. spicatum	y = 0.144(x) + (-3.78)	0.95	60	y = 0.06(x) + (-4.69)	0.86	60	87.0 ± 4.5
Chara sp.	y = 0.20 (x) + (-4.02)	0.97	15	y = 0.13(x) + (-7.17)	0.83	15	81.3 ± 8.7
Elodea canadensis	y = 0.17 (x) + (-1.94)	0.99	22	y = 0.10(x) + (-2.41)	0.97	22	87.7 ± 3.5
Potamogeton crispus	y = 0.11 (x) + 0.20	0.95	14	y = 0.05(x) + 0.38	0.64	14	84.3 ± 3.5
Potamogeton pussillus	y = 0.12 (x) + (-1.31)	0.93	13	y = 0.07(x) + (-0.93)	0.92	13	89.9 ± 4.0
Potamogeton pectinatus	y = 0.13 (x) + 0.79	0.99	8	y = 0.07(x) + (-0.43)	0.99	8	85.2 ± 4.9