PERSISTENCE AND DRIFT OF THE HERBICIDE ENDOTHALL FOLLOWING APPLICATIONS AT LAKELAND VILLAGE AND GRAVELLY LAKES, WASHINGTON

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Water Body Nos. WA-14-1900
WA-12-9020

May 1993

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ACKNOWLEDGEMENTS

We greatly appreciate the cooperation and assistance of the following people: Terry McNab of Aquatic Resources and Doug Dorling of Allied Aquatics provided information on herbicide application; Dale Davis, Chris Maynard, Bob Barnard, Kari Rokstad, and Jim Cubbage of Ecology helped obtain field samples; Gordon Grayum of the Lakeland Village Community Club and Mrs. Main of Gravelly Lake provided access to sampling sites and information about the lakes. Ed Rashin reviewed the report and provided insightful comments and suggestions. Stuart Magoon reviewed the data and Kelly Carruth reviewed and formatted the final copy of this report.

ABSTRACT

Endothall concentrations were monitored in the water column of two western Washington lakes following typical summer treatment to control aquatic macrophytes. Concentrations measured immediately after treatment (Day 1) were near the target application levels of 1 - 4 ppm (1,000 - 4,000 μg/L), but decreased to less than 50 μg/L after 24-to-48 hours. Trace concentrations of endothall were detected in untreated areas of the lakes 2-to-8 days following application. By Day 16, concentrations at all sampling sites were near or below detection limits (10 - 20 μg/L). No substantial changes in water clarity, dissolved oxygen, or other water quality parameters were observed during the course of the study.

INTRODUCTION

The Washington State Department of Ecology (Ecology) currently permits the use of herbicides containing the chemical endothall for the control of aquatic nuisance plants. The dipotassium salt of endothall is the active ingredient in the herbicides Aquathol® and Aquathol K® produced exclusively by Atochem of North America. Although the persistence of endothall in warm water lakes has been well documented (Reinert and Rodgers, 1987; Reinert *et al.*, 1988), little is known about the persistence of endothall in surface waters of Washington. There are also concerns that endothall may be transported downstream in treated lakes with surface water outlets.

At the request of the Water Quality Program, the Toxics, Compliance, and Ground Water Investigations Section has conducted surveys of endothall concentrations in two western Washington lakes: Lakeland Village Lake in eastern Mason County near Allyn and Gravelly Lake near Tacoma, Washington. The purpose of the surveys was to assess the persistence and drift of endothall in the water column following typical treatment by a professional applicator.

DESCRIPTION OF LAKES AND ENDOTHALL APPLICATIONS

Lakeland Village

Lakeland Village Lake is a shallow 40 acre lake partially formed by construction of an earthen dam in the early 1960s. The lake is fed through underwater springs and small inflow channels at the north end. One of these channels is overflow from ponds in an adjacent golf course. Mean depth of the lake is about 8 feet and maximum depth is about 12 feet. The shoreline is largely developed, which, in addition to golf course drainage, probably has an enriching effect and contributes to eutrophication.

The lake was treated with liquid Aquathol® on June 22, 1992, to control curly leaf pondweed (*Potamogeton*) and elodea (*Elodea canadensis*). Figure 1 shows treatment areas for both lakes. Target water concentration of the treatment area was 1 - 2.5 parts per million (ppm). Lakeland Village had not been treated with endothall in the recent past.

Prior to treatment, aquatic weed biomass appeared to be low. Submersed macrophytes were present at relatively low density, mainly in the south arm of the lake.

Gravelly Lake

Gravelly Lake is a deep 160 acre lake in western Pierce County with a shoreline completely ringed by houses. It is in a closed basin - there is no surface water inlet or outlet channel. Mean depth is 38 feet and maximum depth is 55 feet. Anoxic conditions are found in the hypolimnion (below 40 feet) during summer and autumn. A previous water quality

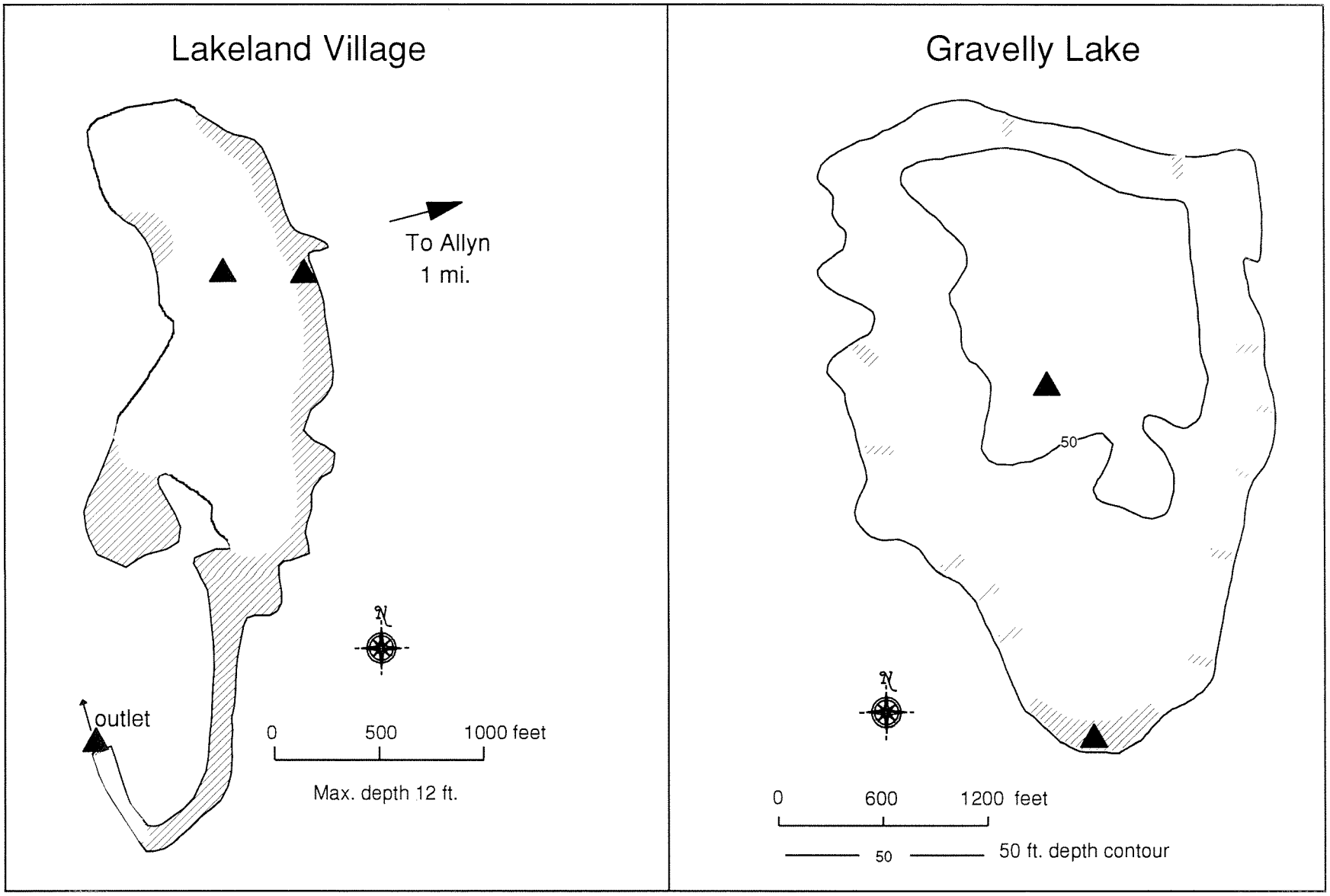

Figure 1. Endothall Sampling Sites at Lakeland Village and Gravelly Lakes

▲ Sampling Site ▨ Treatment Areas

assessment suggested fairly high biological productivity of the lake (Collings, 1973), yet aquatic macrophytes appeared to be scarce during the present study.

Treatment with liquid and granular Aquathol® was done on August 11, 1992, to control pondweed (*Potamogeton*). Most of the herbicide was applied near the southern shore. Spot treatments were done around the shoreline, mostly in the vicinity of private docks. Target endothall concentration of the treatment area was 3 - 4 ppm.

Two months prior to the August application, a small area in the northeast corner of Gravelly Lake was treated with Aquathol K®. Six weeks following that treatment, the Washington State Department of Health and Tacoma-Pierce Health Department collected water column samples from the vicinity of the treatment area because a young girl became sick after swimming in the lake. Ecology had the samples analyzed for endothall, but none was found at a detection limit of 10 μg/L (parts per billion).

METHODS

Sample Collection

Sampling sites for each lake are shown in Figure 1. One site within the treated area and one site in the center lake was monitored at each lake. At Lakeland Village, monitoring was also conducted at the lake outlet.

Endothall samples were collected prior to treatment (Day 0), approximately one hour after treatment was complete (Day 1), and on Days 2 or 3, 4, 8, 16, and 36 (Lakeland Village only). All samples were surface grabs except for Gravelly Lake where samples were also obtained from a depth of 50 feet at center lake using a Kemmerer bottle. Replicate samples were collected at all sites for each day of sampling at Lakeland Village, and on Days 0-through-4 from center surface and south shore sites at Gravelly Lake.

Samples for endothall analysis were collected in 40 mL amber glass vials with teflon septa caps, solvent and acid cleaned appropriate for analysis of EPA priority pollutants. After sample collection, each sample container was rinsed with clean water and placed in a polyethylene bag to reduce potential for cross-contamination. Samples were immediately placed on ice for transport to the laboratory.

Chemical Analysis

Endothall was analyzed at A & S Environmental Laboratory in Reading, Pennsylvania. Analysis was by gas chromatography according to EPA Method 548 "Analysis of Endothall in Drinking Water." Samples for nutrients (nitrogen and phosphorous), total organic carbon (TOC), pH, specific conductance, and turbidity were analyzed at the Ecology Manchester Environmental Laboratory or Analytical Resources Inc. in Seattle following methods outlined

in Ecology (1991). Dissolved oxygen (DO) samples were fixed in the field and analyzed at Ecology's Tumwater DO lab.

Quality of the Endothall Data

Results of the endothall analyses were reviewed at the Manchester Laboratory by Stuart Magoon. The data were evaluated for qualitative and quantitative accuracy, validity, and usefulness.

Samples and extracts were stored in the dark at 4°C until analyzed. Most samples were extracted within seven days of collection as specified in the method. A few of the Gravelly Lake samples were extracted one day beyond the seven day limit. Analysis of the extracts was completed within six days of extraction. A & S has demonstrated the extracts are stable for up to three months. Two of the Lakeland Village samples were broken during shipment to A & S Laboratory.

Accuracy and precision of the data were evaluated by recoveries of an endothall reference standard spiked in lake water (matrix spikes) and through duplicate analyses of field samples. These data are summarized in Tables 1 (Lakeland Village) and 2 (Gravelly Lake). No quality control limits have been established for recovery or precision for this method.

Matrix spike recoveries of 74.0% to 88.5% were achieved during the Lakeland Village study. Recoveries were more variable in Gravelly Lake samples (71.9% to 118%), probably because the spiking level was much lower. The mean relative percent for field replicates was 42% (Appendices A.1 and A.2). Overall, there was good agreement between duplicate analyses of matrix spikes and field samples.

Three types of blanks were analyzed: a bottle blank, transfer blanks, and laboratory (method) blanks. The bottle blank consisted of a sample container filled with organic-free water at the Manchester Laboratory and carried unopened along with the Lakeland Village field samples. Transfer blanks were prepared at both lakes following endothall application by pouring blank water from one sample container to another.

Blank results (Appendix A) indicated that laboratory contamination may have been responsible for some of the endothall reported in lower level samples. One of the transfer blanks had 7 μg/L endothall. Endothall concentrations of 5 to 30 μg/L were reported for some method blanks. Method detection limits for the Lakeland Village and Gravelly Lake samples were 10 μg/L and 20 μg/L, respectively. In the opinion of Stuart Magoon, endothall results in the range of 10 to 35 μg/L for Lakeland Village and 20 to 45 μg/L for Gravelly Lake "should be used with caution since laboratory contamination may be contributing to the result."

Table 1. Accuracy and Precision of Endothall Data: Lakeland Village

1. Matrix Spikes of Lake Water

Sample Set	PPM Added	PPM Found	Recovery	RPD
June 22 – 25	10.0	8.04	80.3 %	
	10.0	7.99	79.8 %	
			80.1 %	<1 %
June 29	10.0	8.00	80.0 %	
	10.0	7.70	77.0 %	
			78.5 %	4 %
July 7	10.0	7.40	74.0 %	
	10.0	8.85	88.5 %	
			81.3 %	18 %
July 27	10.0	8.37	83.7 %	
	10.0	8.59	85.9 %	
			84.8 %	3 %

2. Duplicate Analysis of Field Samples (ug/L)

Sample Set	Analysis #1	Analysis #2	RPD
June 22 – 25	630	640	2 %
	26	27	4 %
	26	24	8 %
June 29	59	57	3 %
July 7	28.5	30.5	3 %
July 27	<10	<10	-

RPD = relative percent difference; range as percent of mean

Table 2. Accuracy and Precision of Endothall Data: Gravelly Lake

1. Matrix Spikes of Lake Water

Sample Set	PPM Added	PPM Found	Recovery	RPD
August 11 – 14	0.60	0.68	80.0 %	
	0.60	0.63	71.9 %	
			75.9 %	10 %
August 18	0.25	0.30	118 %	
	0.25	0.28	110 %	
			114 %	7 %
August 26	0.25	0.24	98.0 %	
	0.25	0.23	93.0 %	
			95.5 %	5 %

2. Duplicate Analysis of Field Samples (ug/L)

Sample Set	Analysis #1	Analysis #2	RPD
August 11 – 14	37	30	21 %
	20	20	0 %
August 18	<20	<20	–
August 26	<20	<20	–

RPD = relative percent difference; range as percent of mean

RESULTS AND DISCUSSION

Lakeland Village

Endothall concentrations measured before and after the Lakeland Village application are summarized in Table 3. The laboratory reported trace amounts of endothall as being present in the two pre-treatment samples. This finding would have to be attributed to sample contamination, either in the field or laboratory.

A mean endothall concentration of 1,280 μg/L was measured along the east shoreline of the lake immediately following application (Day 1). This agrees well with the intended treatment level of 1 to 2.5 parts per million (1,000 - 2,500 μg/L). No endothall was detectable at the center of the lake or at its outlet on Day 1.

Endothall levels appeared to decrease rapidly within 24-hours of application. By Day 2, concentrations within the treated area were approximately 3% or less than the previous day. There was evidence to indicate endothall at the center of the lake on the second day. One of the two samples collected there had 77 μg/L (the other was broken in shipment).

Beyond Day 2, endothall was rarely detected at levels much above those measured in pre-treatment samples or in blanks. Maximum credible concentrations of approximately 50 μg/L were measured on Day 8 along the east shore and at the lake center. By Day 16 endothall concentrations in lake water could not be differentiated from blank water. At no time during the study were substantial concentrations of endothall observed at the lake outlet.

Table 4 summarized the water quality measurements taken at Lakeland Village during the endothall monitoring period. No meaningful changes in water clarity, dissolved oxygen, or other parameters were observed. As previously noted, only small amounts of dying plant material were seen at either lake.

Gravelly Lake

Rapid dispersion/degradation of endothall was also observed following its application to Gravelly Lake (Table 5). Concentrations measured within the treated area of the lake averaged 1,020 μg/L soon after treatment, similar to findings at Lakeland Village, but substantially lower than the targeted treatment level of 3 - 4 ppm.

Samples were not collected at Gravelly Lake the day following treatment. However, by the second day after application (Day 3) and for the remainder of the 16-day monitoring period, almost no significant endothall concentrations were found. One surface water sample from the center of the lake on Day 8 was reported to contain 47 μg/L of endothall (companion samples also lost in shipment). There was apparently no migration of endothall to the lake bottom (center lake) during the survey.

Table 3. Summary of Endothall Results for Lakeland Village, June 22 – July 27, 1992
(ug/L; mean ± range for replicate samples)

Day	0*	1	2	4	8	16	36
East Shore	20±8	1,280±640	34±8	25±0	53±5	17±12	<10
Center	NA	<10	77	19±13	48±36	<10	<10
Outlet	NA	<10	11±1	18±4	30±12	<10	<10

*pre-treatment samples
= sample contamination may have contributed to result

Table 4. Water Quality at Lakeland Village, June 22 – July 27, 1992 (n = 6)

Location	Sample Depth (ft.)	Secchi Depth (ft.)	Temp. (C)	D.O. (mg/L)	pH (units)	Specific Conductance (umhos/cm)
East Shore (9 ft. deep)	1	>9	21.4–25.7	7.5–8.9	7.4–7.8	53.0–57.2
	8	–	21.3–23.1	7.5–10.3	–	–
Center Lake (12 ft. deep)	1	10–12	21.3–25.6	7.4–8.7	7.3–7.7	52.9–57.6
	11	–	20.5–23.0	7.1–10.0	–	–
Outlet	–	–	20.0–25.0	7.2–8.0	7.3–7.4	53.4–57.9

Location	Sample Depth (ft.)	Turbidity (NTU)	TOC (mg/L)	NH3 (mg/L)	NO3/NO2 (mg/L)	Tot. P (mg/L)
East Shore (9 ft. deep)	1	.05–2.0	3.8–4.8	<.01–.82	<.01–<.05	<.01–0.02
	8	–	–	–	–	–
Center Lake (12 ft. deep)	1	.05–1.6	3.8–5.2	.02–1.2	<.01–<.05	<.01–0.03
	11	–	–	–	–	–
Outlet	–	1.0–1.5	3.3–5.6	<.01–.22	<.01–<.05	<.01–.02

Table 5. Summary of Endothall Results for Gravelly Lake, August 11 – 26, 1992
(ug/L; mean ± range for replicate samples)

Day	0*	1	3	4	8	16
S. Shore, Surface	<20	1,020±30	32±2	30±2	23	<20
Center, Surface	<20	<20	27±1	30±0	47	<20
Center, Bottom	NA	<20	NA	<20	<20	<20

*pre-treatment samples
= sample contamination may have contributed to result

Table 6. Water Quality at Gravelly Lake, August 11 – 26, 1992 (n = 5)

Location	Sample Depth (ft.)	Secchi Depth (ft.)	Temp. (C)	D.O. (mg/L)	pH (units)	Specific Conductance (umhos/cm)
South Shore (12 ft. deep)	1	>12	21.7–23.2	8.9–9.5	8.1–8.5	148–156
	11	–	22.0–24.5	8.9–10.2	–	–
Center Lake (52 ft. deep)	1	24–34	22.0–23.6	8.9–9.3	8.1–8.5	145–151
	50	–	10.9–12.8	0	–	–

Location	Sample Depth (ft.)	Turbidity (NTU)	TOC (mg/L)	NH3 (mg/L)	NO3/NO2 (mg/L)	Tot. P (mg/L)
South Shore (12 ft. deep)	1	.40–.75	1.7–2.4	.02–.05	.16–.20	<.01
	11	–	–	–	–	–
Center Lake (52 ft. deep)	1	.40–1.1	1.6–1.7	.02–.06	.16–.19	<.01–.02
	50	–	3.0–4.7	–	–	–

Water quality conditions in Gravelly Lake are summarized in Table 6. As with Lakeland Village, there were no substantial changes in conventional water quality parameters following application of endothall. Samples collected at center lake indicated the bottom waters were anoxic prior to herbicide application.

Endothall Persistence in Other Studies

The monitoring efforts at Lakeland Village and Gravelly Lake indicated endothall concentrations decreased to limits of detection (10 - 20 μg/L) within 16 days of application. Most of the endothall had dispersed or degraded by 24-to-48 hours. Table 7 compares these results to other studies of endothall's persistence in freshwater.

Endothall's persistence in lakes and ponds is consistently reported to be short, ranging from 2.5 to 36 days. Reinert *et al.*, (1988) attribute its disappearance from the lakes primarily to dilution, dispersion, biodegradation, and biotransformation. Adsorption to sediment does not appear to be a major fate process, although sediment characteristics, such as organic content and microbial populations, are probably important factors in degradation (Sikka and Rice, 1973; Simsiman *et al.*, 1976). Depletion of dissolved oxygen, which was not observed at Lakeland Village or Gravelly Lake, may slow loss of endothall. For example in the Simsiman and Chesters experiment listed in Table 7, endothall disappeared rapidly after persisting for 30 days following "the restoration of oxygenated and oxidizing conditions."

The fate of endothall in the present study was not determined. Based on results of previous studies, microbial degradation was probably the ultimate fate process (Reinert and Rodgers, 1987). However, detectable concentrations of endothall at the lake centers suggest that dispersion accounted for some of the disappearance from the treated area during the first week following application.

SUMMARY AND RECOMMENDATIONS

Substantial concentrations of endothall were found in treated areas of both Lakeland Village and Gravelly Lakes immediately following herbicide application (Day 1). Mean endothall concentrations on Day 1 were 1.0 - 1.3 ppm, about 25 - 100% of target application rates. During the 24-to-48 hours following treatment, endothall concentrations dropped to about 3% of the Day 1 concentrations. Some of the endothall dispersed to the non-treated areas in both lakes, but there was no migration of endothall to the bottom waters of Gravelly Lake. Conventional water quality parameters were not substantially affected in either lake following herbicide treatment.

Results of this survey agreed well with those of previous studies of endothall persistence in freshwater. Past studies have shown aquatic endothall concentrations reduced to non-detectable concentrations in 2.5-to-36 days following treatment, compared to 16 days for the present study.

Table 7. Endothall's Persistence in Lakes and Ponds

Type of Waterbody	Formulation Applied	Initial Concentration (ppm)	Time to Non-Detect (days)	Reference
Lake	dipotassium salt	1 – 4	16	present study
Lake	dipotassium salt	2	3	Reinert et al. (1988)
Lake	dipotassium & diamine salts	2	7	Gangstad (1983)
Simulated Lake	endothall (99.9%)	3	30	Simsiman & Chesters (1975)
Experimental Pool	dipotassium salt	0.03 – 4.5	15 – 21	Reinert et al. (1985)
Pond	dipotassium salt	5	12 – 18	Holmberg & Lee (1976)
Pond	dipotassium salt	2	28 – 36	Sikka & Rice (1973)
Pond	dipotassium salt	0.3 – 1.4	8 – 20	Yeo (1970)
Pond	diamine salt	1	24	Frank & Comes (1967)
Pond	not specified	0.3 – 10	2.5 avg/4 max	Hiltibran (1962)

The available data indicate that, under similar scenarios in western Washington, endothall does not persist in the water column of treated lakes nor does it significantly affect water quality. Results of this survey also suggest that significant downstream transport is not likely to occur. However, caution should be used when treating lakes prone to dissolved oxygen depletion because of the potential for slow endothall degradation.

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APPENDICES

Appendix A.1 Endothall Data Collected at Lakeland Village (ug/L)

Location	Day	Date ('92)	Time	Sample #1	Sample #2	Relative Percent Difference
East Shore	0*	22 June	1220	12 JB	27 B	77%
	1	22 June	1500	635	1920	100%
	2	23 June	1215	26 B	43	49%
	4	25 June	1250	25 B	25 B	0%
	8	29 June	1150	58	48	19%
	16	7 July	1220	29 B	10 U	–
	36	27 July	1340	10 U	10 U	–
Center Lake	1	22 June	1455	–	10 U	–
	2	23 June	1150	–	77	–
	4	25 June	1215	10 U	33 B	–
	8	29 June	1120	84	11 JB	150%
	16	7 July	1220	10 U	10 U	–
	36	27 July	1315	10 U	10 U	–
Lake Outlet	1	22 June	1530	10 U	10 U	–
	2	23 June	1105	10 NJ	12 NJB	18%
	4	25 June	1130	13 JB	22 B	51%
	8	29 June	1000	41	18 B	78%
	16	7 July	1130	10 U	10 U	–
	36	27 July	1230	10 U	10 U	–
Bottle Blank	0	–	–	10 U	–	–
Transfer Blank	2	23 June	1215	8 NJ	–	–
Method Blank	0 – 4	–	–	7 J	–	–
	8	–	–	10 U	–	–
	16	–	–	10 U	–	–
	36	–	–	10 U	–	–

*pre–treatment samples
J = analyte positively identified, value is an estimate
U = analyte was not detected at or above reported result
NJ = evidence that analyte is present, value is an estimate
B = blank results indicate possible contamination

Appendix A.2 Endothall Data Collected at Gravelly Lake (ug/L)

Location	Day	Date ('92)	Time	Sample #1	Sample #2	Relative Percent Difference
South Shore	0*	11 August	1010	20 U	20 U	–
	1	11 August	1430	990	1050	6%
	3	13 August	1100	34 B	29 B	16%
	4	14 August	1125	32 B	28 B	13%
	8	18 August	1045	23 B	–	–
	16	26 August	1050	20 U	–	–
Center, Surface	0*	11 August	1100	20 U	20 U	–
	1	11 August	1400	20 U	20 U	–
	3	13 August	1030	28 B	26 B	7%
	4	14 August	1040	30 B	30 B	0%
	8	18 August	1020	47	–	–
	16	26 August	1015	20 U	–	–
Center, Bottom	1	11 August	1400	–	–	–
	3	13 August	1030	20 U	–	–
	4	14 August	1040	20 U	–	–
	8	18 August	1020	20 U	–	–
	16	26 August	1015	20 U	–	–
Transfer Blank	4	14 August	–	20 U	–	–
Method Blank	0 – 4	–	–	8 J	–	–
	8	–	–	5 J	–	–
	16	–	–	30 NJ	–	–

*pre-treatment samples
J = analyte positively identified, value is an estimate
U = analyte was not detected at or above reported result
NJ = evidence that analyte is present, value is an estimate
B = blank results indicate possible contamination