

**Persistence and Drift
of the Aquatic Herbicide Diquat
Following Application at
Steilacoom and Gravelly Lakes**

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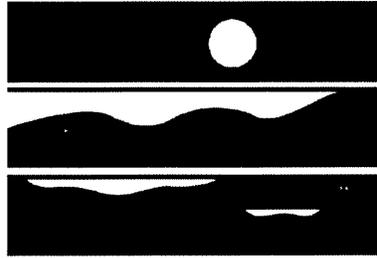
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WASHINGTON STATE
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E C O L O G Y

**Persistence and Drift
of the Aquatic Herbicide Diquat
Following Application at
Steilacoom and Gravelly Lakes**

*by
Dave Serdar*

Environmental Investigations and Laboratory Services Program
Olympia, Washington 98504-7710

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Abstract

Diquat concentrations were monitored in the water column of Steilacoom and Gravelly Lakes following treatment with Reward® to control aquatic weeds. In Steilacoom Lake, diquat persisted at all four sampling sites for at least 12 days, with half-lives of 2.1 to 3.4 days. In Gravelly Lake, diquat persisted for 3 to 6 days following treatment. Diquat concentrations ranged from less than 0.5 to 91.2 µg/L in Steilacoom Lake, and from less than 0.5 to 60 µg/L in Gravelly Lake. Diquat was dispersed or drifted to untreated areas of both lakes, including the Steilacoom Lake outlet (Chambers Creek), within 24 hours after treatment.

Concentrations of diquat resulting from the nearly whole-lake treatment of Steilacoom Lake did not comply with the current Reward® label restriction for drinking water (3 days) based on a maximum allowable level of 10 µg diquat/L. In Gravelly Lake, which was spot-treated, resulting concentrations did comply with the 3-day restriction. Neither lake showed marked changes in water quality, including nutrient and dissolved oxygen concentrations, following treatment with diquat.

Introduction

Background

Diquat [6,7-dihydrodipyrido (1,2-a:2',1'-c) pyrazinediium ion] is a broad-spectrum contact herbicide which is effective at controlling submersed and floating aquatic weeds. The Washington State Department of Ecology (Ecology) does not currently permit the use of diquat in Washington lakes due to a variety of factors, including the lack of critical information about its persistence and drift from treated areas (Ecology, 1992). However, state legislation enacted in 1996 (ESSB 6666) requires Ecology to "...expedite requests for approval for the application of state or federally registered pesticides by licensed pesticide applicators, including the use of herbicides such as copper sulfate or diquat, to control nuisance and noxious weeds in lakes managed under chapter 90.24 RCW." To this end, Ecology is reviewing and acquiring new data on diquat and its potential environmental impact, including persistence and drift in treated lakes, before considering its acceptability for aquatic use.

Objectives

The primary objectives for this project were to (1) determine the persistence of diquat in the water column of lakes following treatment for aquatic weed control, and (2) assess the acceptability of product label restrictions in Washington. Data currently available may not accurately reflect environmental factors in lakes of this region or persistence of diquat under a variety of treatment conditions.

Other objectives were to (3) assess the degree to which diquat drifts from the treatment area and (4) evaluate the accuracy of a model currently being used to predict diquat concentrations in water.

Diquat concentrations were monitored in the water column of Steilacoom and Gravelly Lakes following treatment during 1996. The treatments were permitted to implement the provisions of ESSB 6666.

Description of Lakes and Diquat Applications

Steilacoom Lake

Steilacoom Lake, in western Pierce County, is a shallow, eutrophic lake which is underlain by muck or fine sediments composed primarily of silt and clay (Bennett and Cubbage, 1992; KCM, 1996). The 320 acre lake was formed when a dam was built across Chambers Creek in 1852, inundating a large wetland. Mean depth of the lake is 11 feet with a maximum depth of 20 feet. The lakeshore is fully developed as a residential neighborhood.

Two streams are tributary to Steilacoom Lake: Ponce de Leon Creek to the east and Clover Creek to the south (Figure 1). The lake also receives stormwater through several drains (KCM, 1996). Chambers Creek provides the only surface water outlet, emptying into Puget Sound at Chambers Bay approximately four stream miles from the lake.

Three Washington State Department of Fish & Wildlife (WDFW) salmon/trout hatcheries are located downstream of Steilacoom Lake: Chambers Creek and South Tacoma Hatcheries are located along Chambers Creek upstream of the confluence with Flett and Leach Creeks; Garrison Springs Hatchery is located near Chambers Bay. Chambers Creek Hatchery is the only one among these three that uses Chambers Creek water for fish rearing; South Tacoma and Garrison Springs Hatcheries rely on spring water (Jennifer Shesler, WDFW, personal communication).

Steilacoom Lake was treated with diquat on June 24, 1996 to control *Elodea* (*Elodea canadensis*) and pondweeds (*Potamogeton spp.*). Treatment was done by a licensed professional applicator. Approximately 260 acres were treated via subsurface injection at an application rate of 2 gallons Reward®¹/acre, equivalent to 134 µg diquat cation/L based on a mean depth of 11 feet. Conditions of the pesticide application permit issued by Ecology (Permit No. 96WQ-S168) specified that no diquat was to be applied within a 300-ft radius of the mouths of Ponce de Leon and Clover Creeks, and within a 400-ft radius around the lake outlet. In addition, areas in the southern basin deeper than 17 feet and in the northern basin greater than 15 feet were also to be left untreated. These conditions appeared to have been met based on observations made by Ecology staff (L. Randall and D. Serdar) during the June 24 diquat application.

Copper sulfate was used in Steilacoom Lake on June 5, 1996 -- 19 days prior to the diquat application -- to control algae and prevent potentially toxic algal blooms. Aside from the June 5 copper treatment and the June 24 diquat treatment, no other herbicides or algicides were used in Steilacoom Lake until after July 7, when sampling for diquat was completed.

¹ Reward® is a dibromide salt of diquat manufactured exclusively by Zeneca Inc. It contains 3.73 lbs. diquat dibromide or 2 lbs. diquat cation per gallon.

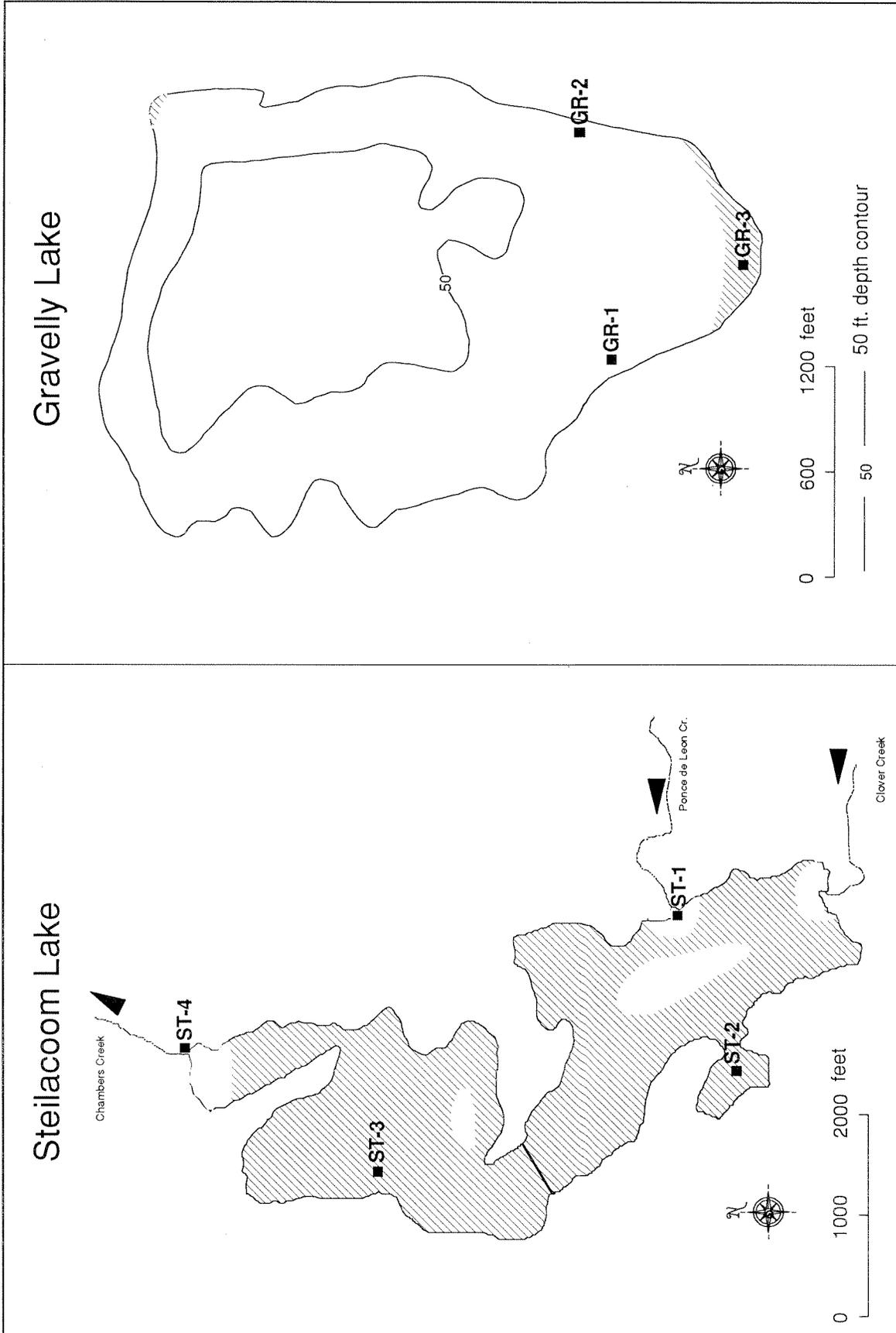


Figure 1. Diquat Sampling Sites at Steilacoom and Gravelly Lakes

- Sampling Site
- ▨ Diquat Treatment Areas

Gravelly Lake

Gravelly Lake is a deep 160 acre natural lake in western Pierce County with a shoreline ringed by houses (Figure 1). As its name implies, it has a gravelly bottom. The lake 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 due to stratification. A previous water quality assessment suggested the lake has fairly high biological productivity (Collings, 1973). Data collected during a 1993 survey led Rector (1996) to conclude that the lake is oligomesotrophic because it exhibited characteristics of a lake both oligotrophic (good water clarity, low density of algae in open water, and low levels of phosphorous in the upper layer) and mesotrophic (sediment release of phosphorous and low oxygen concentrations).

Gravelly Lake was treated with diquat on June 14, 1996 to control *Elodea* and pondweeds. Treatment was done by a licensed professional applicator. Diquat application was limited to approximately 8 acres along the southern shoreline and a small, 2-acre embayment in the northeast portion of the lake. The rate of application was 1-2 gallons Reward®/acre equivalent to 110 µg diquat cation/L based on an average depth in the treatment area of 10 feet. Diquat was applied via subsurface injection. On the same day as diquat treatment, copper sulfate was also used along most of the northern, eastern, and southern shoreline for algae control. All areas treated with diquat were also treated with copper. An additional aquatic pesticide, dipotassium endothall (Aquathol K®), was used six days later (June 20) to control pondweeds along 20 acres of shoreline area, including those previously treated with diquat.

Methods

Sample Locations and Sampling Design

Sampling sites for each lake are shown in Figure 1. Details of the sampling site locations are in the Appendix, Table A-1.

Steilacoom Lake

For Steilacoom Lake, samples were collected at four sites:

ST-1 was directly off the mouth of Ponce de Leon Creek. This site was within an untreated buffer and was selected to assess the drift of diquat into a buffer zone.

ST -2 was located at the center of a bay near the southern end of the lake. This bay is shallow, has a fairly narrow opening, and its entire surface area was treated with diquat. The location and morphology of this bay suggests that it is poorly flushed. The selection of **ST-2** was made to determine if diquat concentrations would persist relatively long in this area.

ST-3 was located approximately 300 feet offshore in the northern basin of the lake. It was selected to represent a treated area in the main body of the lake. The northern basin was selected for this site because two other sites (**ST-1** and **ST-2**) had already been chosen in the southern portion of the lake.

ST-4 was located on Chambers Creek directly below the Steilacoom Lake dam. This site was selected to determine if diquat was being transported downstream.

Water was sampled for each parameter within four hours of diquat treatment, 24 hours post-treatment, and at 2, 3, 7, and 12 days post-treatment. Pre-treatment samples were not collected because it has been at least five years since diquat was last used in Steilacoom Lake.

Gravelly Lake

Samples from Gravelly Lake were collected from three sites:

GR-1 was located along the southwestern shoreline, approximately 50 feet offshore. This site was approximately 550 feet from the nearest edge of the treatment area.

GR-2 was located along the shore opposite **GR-1**. Like **GR-1**, it was located in an untreated area at some distance (approximately 500 feet) from the nearest treatment edge.

GR-3 was located approximately 50 feet offshore near the longitudinal center of the area treated with diquat.

Water samples from Gravelly Lake were collected within four hours after treatment was completed, at 24 hours post-treatment, and at 2, 3, 6, and 12 days post-treatment. Pre-treatment samples were not collected because it has been at least five years since diquat was last used in Gravelly Lake.

Sampling Methods and Chemical Analysis

Samples were collected at mid-depth using a plastic Kemmerer bottle and analyzed for diquat, total organic carbon (TOC), ammonia (NH₃), nitrite-nitrate (NO₂-NO₃), orthophosphate (ortho-PO₄), total phosphorous (TP), and total suspended solids (TSS) at the EPA/Ecology Manchester Environmental Laboratory. Temperature, pH, and water clarity (Secchi depth) were measured in the field. Samples for dissolved oxygen (DO) determination were fixed in the field and analyzed at the Ecology Headquarters DO lab using the azide-modified Winkler titration (Ecology, 1993).

Samples were placed in containers and preserved as recommended by the Manchester Laboratory (Ecology, 1994; Table A-2). All samples were stored in ice or refrigerated at 4 ° C and delivered to the lab via courier the following business day.

To prevent contamination of the samples, sampling equipment was cleaned by scrubbing with Liquinox® detergent followed by multiple rinses with hot tap water and deionized water. Samples were collected from untreated areas first, and samples for diquat analysis were individually enclosed in zip-lock bags to prevent cross-contamination. To assess possible contamination in the field, an equipment blank was prepared on-site (at each lake) during the day of herbicide application.

All samples were analyzed according to EPA methods and met holding time and quantitation limit requirements. Diquat was analyzed according to EPA 549.1 using high performance liquid chromatography with ultraviolet detection. Because EPA 549.1 is a drinking water method, the samples first had to be filtered using particle extraction disks.

Data Quality

Quality of the data was assessed through the analysis of field and laboratory blanks, matrix spikes, replicate field samples, duplicate laboratory analyses, holding times, and instrument calibration. Quality assurance memoranda were prepared by the chemists at Manchester and are included in the Appendix.

Instrument calibration and holding times were all within the EPA method criteria. No analytes were detected in equipment or laboratory blanks.

Table 1 shows a summary of the accuracy and precision data for the diquat analysis. Accuracy was assessed through analysis of matrix spikes. Precision was assessed through analysis of duplicate matrix spike samples (lab precision) and replicate field samples (overall precision).

Table 1. Accuracy and Precision Data for Diquat Analysis.

Sample Number	Matrix Spike		Field Replicates	
	Recovery (%)	RPD	($\mu\text{g/L}$)	RPD
27-8643	205/180	13%	na	na
27-8645	25/30	18%	na	na
24-8604/8605	na	na	21.9/9.0	83%
25-8615/8616	na	na	U (0.5)/60	> 200%
26-8623/8624	na	na	1.2/0.6	67%

RPD=Relative Percent Difference (difference/mean x 100%)

U=not detected at concentration in parentheses

na=not analyzed

All of the diquat spikes had poor recoveries. In the case narrative for the diquat analysis (see Appendix), Huntamer suggested that the high recoveries were due to a high native diquat concentration in the sample. However, the additional matrix spike analyses were performed on a sample with an undetectable native diquat level. Unfortunately, no matrix spike recovery limits have been established for this method.

The overall precision data for diquat were only slightly better. Most of the difference came from a pair of replicate field samples which yielded diquat concentrations of 60 $\mu\text{g/L}$ and <0.5 $\mu\text{g/L}$. Since these samples were collected in a diquat-treated area shortly after application, one can only assume that the samples were collected from slightly different locations in the water column and that the diquat was poorly mixed within the treatment area soon after application. Other field replicates yielded more precise results, and duplicate analyses of matrix spikes indicated good laboratory precision.

Overall data quality for nutrients, TOC, and TSS analyses was good (Table A-3). Matrix spike recoveries were generally lower than 100% yet, aside from one low NH_3 spike recovery (68%), were within method acceptance limits of $\pm 25\%$. Precision was good, with overall precision being slightly lower than laboratory precision.

In summary, all of the data are acceptable for use but the diquat data should be viewed with some caution. Because matrix spike recoveries were either very low or very high, it is impossible to determine if the data are biased high, low, or at all. Samples collected during the hours immediately after diquat treatment may not be fully representative of water column concentrations due to incomplete mixing.

Results

Steilacoom Lake

Concentrations of diquat in Steilacoom Lake are shown in Table 2. Diquat was not detected at the two untreated sites within several hours after application. ST-2 had much higher concentrations than ST-3, probably because the water and sampling depth at ST-2 was one-half that of ST-3 and therefore had less capacity for dilution. Diquat was detected at all sites at 24 hours with the highest concentration at the outlet. Among the samples collected, concentrations were at their peak at 24 hours except for ST-3 where dilution and mixing were probably still occurring. By day 12, diquat concentrations at all sites had decreased an order of magnitude from their highest levels.

Half-lives ($t_{1/2}$) of diquat dissolved in the water column were calculated as $-\ln 2/k$ where k is the rate coefficient. k was obtained from the best fit regression equation in the form $C_t = e^{(C_o + kt)}$, where C_t is the concentration remaining at time t (in days), and C_o is the natural logarithm of the peak concentration. Diquat concentrations preceding peak concentrations (*i.e.* at <4 hours for ST-1, ST-2, and ST-4, and at <4 hours and at 24 hours at ST-3) were not used to calculate regression lines. The half-lives shown in Table 2 are probably slight underestimates because they do not account for loss of diquat through the outlet; the flushing rate is a minor factor in overall dissipation of diquat.

Additional water quality parameters monitored during the survey are summarized in Table 3. The complete set of water quality data is in Table A-4. In general, water quality was consistent with previous surveys of Steilacoom Lake (Collings, 1973; KCM, 1996) with the exception of higher-than-normal seasonal NO₂-NO₃ levels. KCM (1996) found summertime levels of NO₂-NO₃ to be less than 200 µg/L but noted that when groundwater flows to the lake are substantial, such as during winter and spring, NO₂-NO₃ levels in the lake are in the range of 1,000 to 2,000 µg/L due to high (>1,000 µg/L) NO₂-NO₃ in groundwater. A wet spring and high lake level observed during June 1996 suggests that substantial groundwater contribution may have persisted longer than normal, accounting for the elevated NO₂-NO₃ levels.

Table 2. Diquat Concentrations in Steilacoom Lake Water ($\mu\text{g/L}$).

	ST-1	ST-2	ST-3	ST-4
< 4 hours	U (0.5)	84.9	15.4	U (0.5)
24 hours	57.1	87.0	47.1	91.2
2 days	43.1	74.1	77.7	73.9
3 days	54.7	58.7	32.5	35.5
7 days	14.5	7.2	18.0	16.0
12 days	6.8	3.1	3.2	4.7
$t_{1/2}$	3.4 days	2.1 days	2.4 days	2.6 days

U=not detected at concentration in parentheses

Table 3. Range of Water Quality Values Obtained During the 1996 Diquat Monitoring in Steilacoom Lake.

	ST-1	ST-2	ST-3	ST-4
Temp. (C)	17.5 - 21.9	18.5 - 21.7	19.1 - 20.4	19.5 - 23.1
pH	7.85 - 9.00	8.37 - 8.98	8.24 - 9.04	8.34 - 9.01
Secchi depth (ft)	4 (full depth)	5 (full depth)	5 - 8	na
DO (mg/L)	11.0 - 13.1	10.9 - 13.1	11.5 - 14.7	na
TOC (mg/L)	1.9 - 2.5	2.4 - 2.7	2.2 - 2.6	2.4 - 2.8
NH ₃ ($\mu\text{g/L}$)	U(10) - 18	U(10) - 29	U(10) - 17	U(10) - 20
NO ₂ -NO ₃ ($\mu\text{g/L}$)	438 - 745	329 - 515	393 - 619	341 - 512
ortho-PO ₄ ($\mu\text{g/L}$)	U(10)	U(10)	U(10)	U(10)
TP ($\mu\text{g/L}$)	15 - 25	12 - 32	12 - 28	11 - 32
TSS (mg/L)	2 - 3	U(1) - 4	2 - 5	2 - 17

na=not analyzed

U=not detected at concentration in parentheses

Gravelly Lake

Concentrations of diquat in Gravelly Lake are shown in Table 4. Diquat was not detected at the untreated sites (GR-1 and GR-2) within several hours of application to the lake. As mentioned previously, replicate samples collected near the longitudinal center of the treatment area (GR-3) several hours after diquat application showed vastly different results -- undetectable diquat concentrations in one sample and 60 $\mu\text{g/L}$ in the other. The difference was probably due to sampling in slightly different locations during a period when the herbicide had little time to become mixed within the water column.

Diquat had drifted to sample sites in untreated areas by 24 hours post-treatment and persisted near the 1 µg/L level at all three sites for at least three days. By the sixth day following application, diquat concentrations were below detection limits at all sites except for GR-2 which had 0.5 µg diquat/L on Day 12. Diquat half-life values were not calculated for Gravelly Lake due to its low concentrations and lack of persistence in the water column.

Table 4. Diquat Concentrations in Gravelly Lake Water (µg/L)

	GR-1	GR-2	GR-3
< 4 hours	U (0.5)	U (0.5)	U (0.5)/60
24 hours	0.6	1.3	2.8
2 days	1.6	1.3	1.3
3 days	0.6	1.0	0.9
6 days	U (0.5)	U (0.5)	U (0.5)
12 days	U (0.5)	0.5	U (0.5)

U=not detected at concentration in parentheses

Water quality parameters monitored during the survey (Table 5) were at levels typically expected in Gravelly Lake during June or July (Collings, 1973; Bortleson, 1976; Rector, 1996). Water clarity, as measured by Secchi readings, increased by 70-90% during the course of the survey, probably as a result of algae die-off from copper treatment. Other than Secchi depths, water quality parameters remained stable during the survey.

Table 5. Range of Water Quality Values Obtained During the 1996 Diquat Monitoring in Gravelly Lake.

	GR-1	GR-2	GR-3
Temp. (C)	18.4 - 20.5	18.0 - 20.7	18.1 - 21.0
pH	7.75 - 8.39	7.85 - 8.41	7.63 - 8.34
Secchi depth (ft)	13 - 25	15 - 25	15 - 25
DO (mg/L)	10.6 - 11.4	10.3 - 10.8	10.4 - 11.0
TOC (mg/L)	1.5 - 1.9	1.5 - 1.9	1.5 - 1.9
NH ₃ (µg/L)	U(10) - 16	U(10) - 15	U(10) - 14
NO ₂ -NO ₃ (µg/L)	471 - 495	476 - 502	476 - 516
ortho-PO ₄ (µg/L)	U(10)	U(10)	U(10)
TP (µg/L)	U(10)	U(10) - 12	U(10) - 19
TSS (mg/L)	U(1) - 3	U(1) - 2	U(1) - 2

U=not detected at concentration in parentheses

Discussion

Persistence and Fate

Several studies have shown diquat to be short-lived in aquatic systems where it is rapidly sorbed to sediments, suspended particulate matter, and plants (Simsiman *et al.*, 1976). Its rate of disappearance is therefore controlled largely by the availability of sorbents. Reinert and Rodgers (1987) reviewed persistence and fate studies on diquat and found water half-lives to typically be in the range of one to four days which agrees closely with the present study findings.

Once diquat has sorbed to sediments or plant material, it may either remain intact or undergo microbial decomposition. Simsiman and Chesters (1976) found that diquat sorbed to plants decomposed rapidly when the plant material decayed, thus promoting microbial degradation of the diquat. However, diquat bound to sediments, especially those containing clay minerals, may persist intact for months and may exhibit sharply reduced bioavailability. Simsiman and Chesters (1976) reported that in laboratory incubation experiments using only sediment and abundant overlying water, adsorption of diquat to sediment was rapid, the rate of microbial degradation was slow, and diquat persisted in sediment for at least 180 days. Other fates for diquat such as volatilization, chemical decomposition and photodegradation are negligible in the aquatic environment (Simsiman *et al.*, 1976; Reinert and Rodgers, 1987).

Steilacoom and Gravelly Lakes were chosen to study diquat persistence in part because they represent two systems with very different physiographies, bottom sediments, and trophic conditions. Given equal treatments, it is expected that diquat would persist in the water column of Gravelly Lake longer than Steilacoom Lake because of the higher abundance of sorbents. However, the lakes were not treated similarly, nor were the treatments conducted in a controlled scientific manner. Approximately 80% of Lake Steilacoom's surface area was treated with diquat, representing an almost equal proportion of the lake's volume. In contrast, only about 6% of Gravelly's surface area was treated, representing approximately 1.5% of the lake volume.

No samples of sorbing material (sediments, suspended particulate matter, or plant material) were collected during this survey to assess the fate of diquat. However, the data suggest that dispersion and drift were probably the major processes affecting the concentration of diquat in the water column of both lakes for at least 2 to 3 days following treatment. Assuming complete mixing of diquat in Steilacoom Lake water, with no loss through sorption or through the outlet, final diquat concentrations would theoretically be 108 µg/L based on a lake volume of 3.53×10^9 L (3,500 acre-ft; this probably underestimates the actual volume since the lake level was higher than normal). Fifty percent or more of this theoretical whole-lake concentration remained

for at least three days post-treatment at ST-1 and ST-2, and for 2 to 3 days post-treatment at ST-3 and ST-4. By seven days post-treatment, most of the diquat was probably bound to sediments or to dead and dying plant material, with a smaller percentage having been flushed through the outlet.

The fate of diquat in Gravelly Lake is more difficult to assess. Theoretical whole-lake concentrations assuming complete mixing and no loss through sorption would be $1.8 \mu\text{g/L}$ based on a lake volume of $7.39 \times 10^9 \text{ L}$ (6,000 acre-ft; also probably an underestimate). Drift and dispersion clearly occurred during the first two days post-treatment when concentrations were nearly uniform at all three sampling sites. Unlike Steilacoom Lake, however, it is improbable that the diquat in Gravelly Lake was mixed uniformly in the water column because only a small area was treated and Gravelly Lake probably lacks the necessary circulating currents for complete mixing.

Compliance with Label Restrictions

As part of its data submission to support the use of Reward® in Washington, Zeneca, Inc. will be using the EPA Exposure Analysis Modeling System (EXAMS II) to predict the fate of diquat in several lakes, including Steilacoom and Gravelly Lakes. EXAMS II combines a chemical fate and transport model with a hydraulic model, and uses parameters of system geometry, dispersion coefficient, advection velocity, plant mass, suspended sediment concentration, biosorption coefficient, sediment adsorption coefficient, and application rate to predict diquat concentrations.

One of the main goals of the modeling effort will be to determine the validity of timing restrictions specified under the Reward® label. The label for Reward® used in the Steilacoom and Gravelly Lake treatments is included in the Appendix. In a recently issued supplemental label, swimming restrictions were lifted, and the drinking water timing restriction was decreased from 14 to 3 days based on a 2 gallon/acre application rate. The timing restriction for drinking water assumes that diquat concentrations will be reduced to $10 \mu\text{g/L}$ three days following application. This did not occur in Steilacoom Lake.

Based on the rate coefficients calculated for Steilacoom Lake, predicted diquat concentrations would not descend to $10 \mu\text{g/L}$ for 10 days post-application at ST-1, 9 days at ST-4, and 8 days at ST-2 and ST-3 (although the measured concentration at ST-2 was below $10 \mu\text{g/L}$ by Day 7). Since neither Steilacoom Lake nor Chambers Creek are sources of drinking water (Gary Porter, Tacoma-Pierce County Health Department, personal communication), this specific label restriction does not apply. However, this example illustrates the need for *in situ* monitoring since label restrictions are based on very generalized circumstances and dissipation rates may vary widely. If either Steilacoom Lake or Chambers Creek were used for drinking water, the previous drinking water label restriction of 14 days would be protective of human health whereas the current 3-day restriction would not be adequate.

Toxicity to Aquatic Life

While results of this survey show that buffers do little to protect against the drift of diquat into “protected” areas, the concentrations of diquat measured in both Steilacoom and Gravelly Lakes probably do not pose a threat to most aquatic animals. Median lethal concentrations (LC50s) for fish subject to acute exposure generally range from 8,000 to 30,000 µg diquat /L (Bond *et al.*, 1960; Surber and Pickering, 1962; Hiltibran, 1967; Lorz *et al.*, 1979).

Fish in the early life stages appear to be much more sensitive to diquat than adults. Paul *et al.* (1994) found that, for walleye (*Stizostedion vitreum*), LC50s were directly related to age and inversely related to length of exposure. Median lethal diquat concentrations ranged from 750 µg/L for 8-10 day-old fish exposed for 96 hours to 7,800 µg/L for 84-86 day-old fish exposed for 24 hours. One to two week-old smallmouth bass (*Micropterus dolomieu*) and largemouth bass (*M. salmoides*) tested by these investigators showed a higher tolerance to diquat but demonstrated a similar response to length of exposure (LC50s of 3,900-110,000 µg/L for smallmouth bass and 4,900-15,000 µg/L for largemouth bass). Hiltibran (1967) found that fry of smallmouth bass, bluegill (*Lepomis macrochirus*), and lake chubsucker (*Erimyzon sucetta*) die within one to four days of exposure to 2,500 µg/L or 1,300 µg/L diquat while small bluegills (1-3 inch) showed no mortality in 10,000 µg/L diquat for 12 days. Allen and Meekan (1968) reported the use of diquat at 2,000 µg/L to combat *Dermocystidium* fungus infecting pre-spawning chinook salmon (*Oncorhynchus tshawytscha*) at the Priest Rapids Spawning Channel on the Columbia River, Washington. Although one-hour treatments several times per week proved to substantially reduce fungus-induced mortality, no other effects were observed, for instance to eggs, fry, or other non-target organisms.

Most aquatic invertebrates appear to have similar tolerances to diquat as those exhibited by fish (Simsiman *et al.*, 1976). However, the amphipod *Hyaella azteca* is probably the most sensitive aquatic species tested to date. Wilson and Bond (1969) reported a 96-hr LC50 of 48 µg/L and a 48-hr LC50 of 120 µg/L for this species. These data indicate that diquat concentrations found in Steilacoom Lake following treatment would probably be lethal to *Hyaella azteca*. Interestingly enough, Wilson and Bond (1969) also found that LC50s for *Hyaella azteca* exposed to diquat increased by two orders of magnitude when mud was added to test chambers. The authors concluded that the diquat became irreversibly bound to the mud, thereby reducing its bioavailability.

Although acute toxicity tests have been standardized and prove useful in comparing different chemicals and species, the results generally do not address sublethal or secondary effects. For instance, Lorz *et al.* (1979) exposed coho salmon smolts (*O. kisutch*) to 500-3,000 µg diquat /L for 96 or 285 hours and released them to a stream. Migration capability was measured by counting the number of smolts reaching a checkpoint 6.4 km downstream. They found that migration was inhibited at all levels of exposure, and that significant migration inhibition was observed at concentrations lower than those required to cause

direct mortality. The same investigators challenged coho smolts with seawater following freshwater diquat exposures of 250-20,000 µg/L. Dose-dependent mortality was observed at 5,000-20,000 µg diquat/L, levels much higher than those expected to occur based on the label application rates. However, salmon are recognized as an increasingly precious resource and diquat treatments may overlap with the seasonal window for several species undergoing smoltification, one of the most the vulnerable life stages for pacific salmon. Zeneca, Inc. has therefore agreed to conduct additional seawater challenge testing of diquat-exposed chinook salmon during the 1997 smolting season (Jill Slate, Zeneca, Inc., written communication). Ecology also plans to conduct a thorough review of the effects of diquat on fish, aquatic invertebrates, and the aquatic ecosystem. This review, due June 30, 1997, will include an analysis of any sublethal and secondary effects which may occur as a result of diquat's use as an aquatic herbicide.

There a few data available on the toxicity of diquat to non-target aquatic plant species. In their Reregistration Eligibility Decision (RED) fact sheet, EPA concludes that "Additional studies are required to determine diquat dibromide's toxicity to non-target aquatic and terrestrial plants." (EPA, 1995). However, since diquat dibromide is a non-selective herbicide, toxicity to non-target species can probably be expected. Furthermore, since concentrations in Steilacoom Lake outlet were as high as in treated areas of the lake, toxicity to plants in Chambers Creek would be expected.

Conclusions

- Diquat used for aquatic weed control in Steilacoom Lake persisted in the water column for at least 12 days following application. In Gravelly Lake, diquat persisted for 3 to 6 days post-treatment, except at one untreated area where diquat was detected at a very low concentration 12 days post-treatment.
- Peak concentrations of diquat in treated and untreated areas of both lakes were generally found from 1 to 2 days post-application. Peak concentrations at the four sampling sites in Steilacoom Lake ranged from 57.1 to 91.2 µg diquat/L. In Gravelly Lake, peak concentrations of diquat at three sampling sites were 1.3 to 60 µg/L.
- Dispersion and drift were the processes most affecting the water column concentrations of diquat for 2 to 3 days following application. Diquat dissipated much more rapidly in Gravelly Lake, where only 6% of the lake surface was treated compared to Steilacoom Lake where 80% of the surface area was treated. Diquat half-lives in Steilacoom Lake ranged from 2.1 to 3.4 days.
- In both lakes, diquat was found in untreated areas 24 hours after treatment. Diquat was found in the Steilacoom Lake outlet (Chambers Creek) at concentrations similar to those in treated areas of the lake even though no diquat was used within 400 feet of the outlet.
- The current Reward® label restriction for drinking water (3 days) would not be sufficient to protect human health in Steilacoom Lake based on a maximum allowable level of 10 µg diquat/L. In Gravelly Lake, the 3-day restriction would protect human health. These findings are based on a nearly whole-lake treatment in Steilacoom Lake and a spot treatment in Gravelly Lake.
- Diquat concentrations following treatment in Steilacoom and Gravelly Lakes probably do not pose a threat to most aquatic animals. However, diquat concentrations in Steilacoom Lake would probably be lethal to the amphipod *Hyalella azteca*. In addition, it is likely that toxicity to aquatic plants occurred in Chambers Creek following diquat treatment of Steilacoom Lake.
- Diquat treatments in Steilacoom and Gravelly Lakes did not cause marked changes in water quality, including nutrient and dissolved oxygen concentrations. However, any changes in water quality due to diquat treatment may have been masked by additional herbicides and algicides applied prior to or during the present survey.

Recommendations

- Expand buffer areas, use adjuvants, or otherwise modify diquat treatments to prevent drift and dispersion of diquat to “protected” areas. Certain adjuvants mixed with herbicide formulations are reportedly effective at preventing dispersion and increasing contact with target plant material.
- The drinking water restriction on the Reward® label should be returned to 14 days since the dissipation of diquat in water varies widely depending on treatment conditions and limnological factors. The current 3-day drinking water restriction does not appear to be adequate to protect human health.
- A thorough assessment of downstream transport should be included with future diquat applications in Steilacoom Lake or other lakes with surface water outlets. The present survey measured diquat concentrations at the lake outlet but did not include an assessment of its fate and persistence in downstream waters or possible attenuation within the outlet stream.
- Ecology’s review of the effects of diquat on the aquatic ecosystem should include any available data on toxicity to non-target aquatic plant species. If no data can be found, Ecology should consider conducting laboratory or field studies, or require such studies be conducted to obtain these data.
- Diquat concentrations in bottom sediments should be monitored if diquat is to be used on a regular long-term basis.

Acknowledgements

I appreciate the cooperation and assistance of the following people:

- ◇ Doug Dorling of Allied Aquatics did the diquat applications on Steilacoom and Gravelly Lakes
- ◇ Mrs. Main of Gravelly Lake provided access to sampling sites
- ◇ Loree Randall of Ecology's Southwest Regional Office provided useful information about the lakes and helped coordinate herbicide applications
- ◇ Dickey Huntamer of the Manchester Laboratory performed the diquat analysis
- ◇ Debbie Lacroix of Manchester did the general chemistry
- ◇ Greg Pelletier, Art Johnson, Dale Norton, Larry Goldstein and Steve Saunders reviewed the report
- ◇ Joan LeTourneau proofread and formatted the report

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Appendices

Table A-1. Depths and Positions of Sampling Sites

Site	Water Depth (ft)	Sample Depth (ft)	Latitude	Longitude
ST-1	4	2	47°09'43"N	122°31'49"W
ST-2	4.5	2	47°09'38"N	122°32'13"W
ST-3	10	5	47°10'14"N	122°32'28"W
ST-4	-	-	47°10'40"N	122°32'04"W
GR-1	12	6	47°08'40"N	122°31'54"W
GR-2	12	6	47°08'42"N	122°31'36"W
GR-3	12	6	47°08'33"N	122°31'48"W

Table A-2. Requirements for Sample Containers, Preservatives, Holding Times, Analytical Methods, and Quantitation Limits.

Parameter	Sample Container	Preservative	Holding Time	Method	Practical Quantitation Limit
Diquat	1 L amber PVC	none	7 days*	EPA 549.1	0.5 ug/L
TOC	60 mL clear PE	H ₂ SO ₄ to pH<2	28 days	EPA 415.1	1 mg/L
NH ₃	125 mL clear PE	H ₂ SO ₄ to pH<2	28 days	EPA 350.1	10 ug/L
NO ₂ -NO ₃	125 mL clear PE	H ₂ SO ₄ to pH<2	28 days	EPA 353.2	10 ug/L
ortho-PO ₄	125 mL amber PE	none	48 hours	EPA 365.3M	10 ug/L
TP	125 mL clear PE	H ₂ SO ₄ to pH<2	28 days	EPA 365.3	10 ug/L
TSS	1 L clear PE	none	7 days	EPA 160.2	1 mg/L

* until extraction

MANCHESTER ENVIRONMENTAL LABORATORY
7411 Beach Drive E , Port Orchard Washington 98366

CASE NARRATIVE

August 14, 1996

Subject: Diquat in Lakes

Samples: 96 -248601 to -248602, -248604 to -248609, -258610 to -258619, -268620 to -268640
-278641 to -278645 and -288650 to -288653.

Case No. 1405 -96

Officer: Dave Serdar

By: Dickey D. Huntamer 
Organics Analysis Unit

DIQUAT

ANALYTICAL METHODS:

The samples were extracted and analyzed using solid phase extraction by EPA Method 549.1.

HOLDING TIMES:

All sample and extraction holding times were within the recommended limits.

BLANKS:

Laboratory blanks were extracted and analyzed with each set of samples. No Diquat was detected in the laboratory blanks.

SURROGATES:

Currently there are no surrogates used with this method.

MATRIX SPIKE AND MATRIX SPIKE DUPLICATE:

Two sets of matrix spikes were analyzed with the samples. One from each lake studied.

Matrix spike recoveries for sample 9627864 LMX1 were 205% and for LMX2 180%.. The high native concentration in the sample, 18 ug/L, may be a factor in calculating the recoveries. The matrix spike was added at a level to achieve a final concentration of 2 ug/L. This is only one tenth of the native amount present. Consequently a small signal is being imposed on top of large signal which could introduce inaccuracies in the measurements.

Matrix spike recoveries for samples 96278645 LMX1 and LMX2 were 25% and 30% respectively. No native Diquat was detected in the matrix spike source water, 96278645. No matrix spike recovery limits have been established for this method.

FORTIFIED LABORATORY BLANKS:

Four samples of deionized water were spiked with Diquat (2 ug/L), extracted and analyzed using Method 549.1. Diquat recoveries on these fortified laboratory blanks were 85%, 100%, 110% and 75%.

ANALYTICAL COMMENTS:

The adaptation of an EPA drinking water method 549.1 to the analysis of natural lake water samples posed some difficulties. Water from both lakes contained particulate matter. The particulate matter resulted in some long extraction time for some samples. In some cases over six hours was needed to process a one liter sample even with the use of filter aid to prevent clogging. Neither lake was turbid and the water was generally clear although Steilacoom Lake had a slight green cast to it. Gravelly Lake was cleaner and posed the least problem with more rapid filtering times.

Although these samples were relatively light in particulate matter any increase in the amount of particulate matter could pose serious problems with sample extractions. This would result in excessively long times for filtering and higher method detection limits due to the decreased sample volumes processed

There were no significant problems with the analysis and the data is acceptable for use as qualified.

DATA QUALIFIER CODES:

- U - The analyte was not detected at or above the reported value.
- J - The analyte was positively identified. The associated numerical value is an estimate.
- UJ - The analyte was not detected at or above the reported estimated result.
- REJ - The data are unusable for all purposes.
- EXP - The result is equal to the number before EXP times 10 to the power of the number after EXP. As an example 3EXP6 equals 3×10^6 .
- NAF - Not analyzed for.
- N - For organic analytes there is evidence the analyte is present in this sample.
- NJ - There is evidence that the analyte is present. The associated numerical result is an estimate.
- E - This qualifier is used when the concentration of the associated value exceeds the known calibration range.
- bold** - The analyte was present in the sample. (Visual Aid to locate detected compound on report sheet.)

Washington State Department of Ecology
Manchester Laboratory

August 13, 1996

TO: Dave Serdar

FROM: Debbie J. Lacroix, Chemist 

SUBJECT: General Chemistry Quality Assurance memo for the Diquat in Lakes Project

SUMMARY

The data generated by the analysis of these samples can be used noting the qualifications below. Total phosphorus sample 96268624 was flagged with a "J" indicating an estimated result.

SAMPLE INFORMATION

Samples 96248601-53 from the Diquat in Lakes Project were received by the Manchester Laboratory on 6/17-7/8-96 in good condition.

HOLDING TIMES

All analyses were performed within applicable EPA holding times.

ANALYSIS PERFORMANCE

Instrument Calibration

Where applicable, instrument calibration was performed before each analysis and verified by initial and verification standards and blanks. All initial and continuing calibration verification standards were within the relevant EPA control limits. All balances are calibrated yearly with calibration verification performed monthly.

Procedural Blanks

All procedural blanks were within acceptable limits.

Spiked Sample Analysis

All spike recoveries were within the acceptance window of +/- 25 %.

Precision Data

The results of the duplicate analysis of samples were used to evaluate the precision on this sample set. The Relative Percent Differences (RPD) were within their acceptance windows of +/- 20 % except for TSS samples 96268629 and 96268636. The results of these sample were close to the reporting limit making for a high amount of imprecision.

Laboratory Control Sample (LCS) Analyses

LCS analyses were within their acceptance windows of +/- 20 %.

Other Quality Assurance Issues

Total phosphorus sample 96268624 was flagged with a "J" indicating an estimated result due to an irregular peak shape. The sample was re-analyzed and produced the same results. Therefore, the sample is considered an estimate.

Please call Debbie Lacroix at SCAN 871-8812 with any questions or concerns about this project.

cc: Bill Kammin
Project File

Table A-3. Accuracy and Precision Data for General Chemistry Analysis.

Parameter	Sample Number	Matrix Spike Recovery	Laboratory Duplicate RPD	Field Replicate RPD
TOC	25-8615	87%	na	na
"	27-8643	82%	na	na
"	24-8609	na	0%	na
"	25-8613	na	0%	na
"	26-8622	na	0%	na
"	26-8627	na	4%	na
"	26-8636	na	4%	na
"	28-8651	na	0%	na
"	24-8604\05	na	na	0%
"	25-8615\16	na	na	12%
"	26-8623\24	na	na	0%
NH ₃	24-8606	90%	na	na
"	25-8619	90%	na	na
"	26-8624	68%	na	na
"	26-8634	75%	na	na
"	27-8644	99%	na	na
"	28-8653	98%	na	na
"	25-8613	na	7%	na
"	25-8617	na	0%	na
"	26-8628	na	0%	na
"	26-8638	na	0%	na
"	25-8615\16	na	na	29%
NO ₂ -NO ₃	24-8606	104%	na	na
"	25-8616	87%	na	na
"	25-8619	92%	na	na
"	26-8624	94%	na	na
"	26-8634	85%	na	na
"	27-8644	80%	na	na
"	28-8653	88%	na	na
"	24-8601	na	1%	na
"	25-8613	na	1%	na
"	25-8617	na	1%	na
"	26-8620	na	1%	na
"	26-8628	na	1%	na
"	26-8630	na	1%	na
"	26-8638	na	1%	na
"	27-8641	na	1%	na
"	27-8650	na	2%	na
"	24-8604\05	na	na	2%
"	25-8615\16	na	na	5%
"	26-8623\24	na	na	1%

Table A-3 (Cont'd)

Parameter	Sample Number	Matrix Spike Recovery	Laboratory Duplicate RPD	Field Replicate RPD
Ortho-PO ₄	24-8605	84%	na	na
"	25-8616	107%	na	na
"	25-8618	100%	na	na
"	26-8624	96%	na	na
"	26-8629	106%	na	na
"	26-8634	99%	na	na
"	26-8640	108%	na	na
"	27-8644	76%	na	na
"	28-8653	114%	na	na
TP	24-8606	105%	na	na
"	25-8616	86%	na	na
"	25-8619	91%	na	na
"	26-8624	92%	na	na
"	26-8634	92%	na	na
"	27-8644	91%	na	na
"	28-8653	99%	na	na
"	26-8620	na	78%	na
"	26-8628	na	24%	na
"	26-8638	na	9%	na
"	27-8641	na	25%	na
"	27-8650	na	32%	na
"	24-8604\05	na	na	21%
"	26-8623\24	na	na	62%
TSS (mg/L)	24-8601	na	0%	na
"	25-8612	na	0%	na
"	25-8616	na	0%	na
"	25-8619	na	0%	na
"	26-8629	na	29%	na
"	26-8636	na	0%	na
"	27-8644	na	0%	na
"	28-8653	na	29%	na
"	24-8604\05	na	na	0%
"	25-8615\16	na	na	0%
"	26-8623\24	na	na	40%

RPD=Relative Percent Difference (difference/mean x 100%)

na=not analyzed

Table A-4. Data Collected During the 1996 Diquat Survey in Steilacoom and Gravelly Lakes.
See Figure 1 for Location of Sampling Sites.

Date	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	24-Jun-96	24-Jun-96	24-Jun-96	24-Jun-96	14-Jun-96	14-Jun-96	14-Jun-96
24 hrs	25-Jun-96	25-Jun-96	25-Jun-96	25-Jun-96	15-Jun-96	15-Jun-96	15-Jun-96
2 days	26-Jun-96	26-Jun-96	26-Jun-96	26-Jun-96	16-Jun-96	16-Jun-96	16-Jun-96
3 days	27-Jun-96	27-Jun-96	27-Jun-96	27-Jun-96	17-Jun-96	17-Jun-96	17-Jun-96
6/7 days	1-Jul-96	1-Jul-96	1-Jul-96	1-Jul-96	20-Jun-96	20-Jun-96	20-Jun-96
12 days	7-Jul-96	7-Jul-96	7-Jul-96	7-Jul-96	26-Jun-96	26-Jun-96	26-Jun-96
Sample No.	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	26-8620	26-8622	26-8623\24 ^a	26-8621	24-8601	24-8602	24-8604\05 ^a
24 hrs	26-8626	26-8627	26-8628	26-8629	24-8607	24-8608	24-8609
2 days	26-8633	26-8634	26-8635	26-8636	25-8611	25-8610	25-8612
3 days	26-8637	26-8638	26-8639	26-8640	25-8614	25-8613	25-8615\16 ^a
6/7 days	27-8641	27-8642	27-8643	27-8644	25-8618	25-8617	25-8619
12 days	28-8650	28-8651	28-8652	28-8653	26-8631	26-8630	26-8632
Temp. (C)	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	18.3	18.5	19.1	19.5	19.7	20.7	21.0
24 hrs	19.4	20.4	19.7	21.2	20.5	20.3	21.0
2 days	19.5	20.9	19.6	22.1	18.4	18.0	18.8
3 days	17.5	20.1	19.1	19.5	19.1	18.4	18.1
6/7 days	21.9	21.7	20.4	23.1	20.0	20.2	20.8
12 days	19.8	20.8	19.9	20.3	19.3	19.4	20.3
pH	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	8.98	8.98	9.04	9.01	8.39	8.31	8.34
24 hrs	9.00	8.91	8.97	8.72	8.21	8.09	7.63
2 days	8.70	8.81	8.91	8.75	8.00	7.85	8.00
3 days	7.85	8.37	8.24	8.34	8.07	7.93	7.81
6/7 days	8.67	8.79	8.65	8.57	7.95	8.08	8.28
12 days	8.52	8.40	8.56	8.50	7.75	8.41	7.73
Secchi depth (ft)	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	4(full)	5(full)	5	na	13	15	15
24 hrs	4(full)	5(full)	7	na	16	16	18
2 days	4(full)	5(full)	8	na	20	16	18
3 days	4(full)	5(full)	8	na	17	19	19
6/7 days	4(full)	5(full)	7	na	21	18	18
12 days	4(full)	5(full)	5	na	25	25	25
DO (mg/L)	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	13.1	13.0	14.7	na	10.9	10.8	10.7\10.7 ^a
24 hrs	12.2	12.8	13.9	na	11.0	10.7	11.0
2 days	12.3	12.2	na	na	10.7	10.3	10.5
3 days	11.0	10.9	11.5	na	11.4	10.6	10.4
6/7 days	12.0	13.1	13.1	na	10.6	na	10.8
12 days	12.4	11.8	12.4	na	10.7	10.5	10.5
TOC (mg/L)	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	1.9	2.5\2.5 ^c	2.2\2.2 ^a	2.6	1.8	1.8	1.9\1.9 ^c
24 hrs	2.2	2.6\2.7 ^b	2.6	2.7	1.8	1.8	1.9\1.9 ^c
2 days	2.1	2.7	2.4	2.7\2.8 ^b	1.8	1.9	1.8
3 days	2.5	2.6	2.3	2.6	1.6	1.7\1.7 ^b	1.6\1.8 ^a
6/7 days	2.1	2.4	2.3	2.4	1.9	1.5	1.5
12 days	2.3	2.5\2.5 ^b	2.4	2.4	1.5	1.7	1.6

Table A-4 (Cont'd)

NH ₃ (ug/L)	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	U(10)\U(10) ^b	U(10)	U(10)\U(10) ^a	15	U(10)\U(10) ^b	U(10)	U(10)\U(10) ^a
24 hrs	13	U(10)	13\13 ^b	18	12	U(10)	11
2 days	18	29	17	16	U(10)	10	14
3 days	U(10)	20\20 ^b	11	20	U(10)	13\14 ^b	16\12 ^a
6/7 days	U(10)\U(10) ^b	U(10)	U(10)	U(10)	16	15\15 ^b	13
12 days	U(10)\U(10) ^b	U(10)	U(10)	15	U(10)	U(10)\U(10) ^b	U(10)
NO ₂ -NO ₃ (ug/L)	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	66\1659 ^b	494	477\472 ^a	453	477\482 ^b	480	480\472 ^a
24 hrs	745	504	517\522 ^c	472	495	476	516
2 days	649	502	616	472	471	487	507
3 days	637	513\517 ^b	619	512	481	504\499 ^c	514\489 ^d
6/7 days	528\531 ^b	401	464	449	472	498\497 ^c	477
12 days	434\443 ^b	329	393	341	486	482\481 ^c	501
Ortho-PO ₄ (ug/L)	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	U(10)\U(10) ^b	U(10)	U(10)\U(10) ^a	U(10)	U(10)\U(10) ^b	U(10)	U(10)\U(10) ^a
24 hrs	U(10)\U(10) ^b	U(10)	U(10)	U(10)	U(10)	U(10)	U(10)\U(10) ^b
2 days	U(10)	U(10)	U(10)	U(10)	U(10)	U(10)\U(10) ^c	U(10)
3 days	U(10)\U(10) ^c	U(10)	U(10)	U(10)	U(10)	U(10)\U(10) ^c	U(10)\U(10) ^a
6/7 days	U(10)\U(10) ^c	U(10)	U(10)	U(10)	U(10)	U(10)\U(10) ^c	U(10)
12 days	U(10)\U(10) ^c	U(10)	U(10)	U(10)	U(10)	U(10)\U(10) ^c	U(10)
TP (ug/L)	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	25\11 ^b	24	19\10 J ^e	32	U(10)\U(10) ^b	12	17\21 ^a
24 hrs	16	12	23\18 ^b	23	U(10)	U(10)	U(10)
2 days	15	29	23	11	U(10)	U(10)\U(10) ^b	U(10)
3 days	15	34\31 ^b	12	15	U(10)	U(10)\U(10) ^b	U(10)\U(10) ^a
6/7 days	21\27 ^b	22	23	23	U(10)	U(10)\U(10) ^b	U(10)
12 days	29\21 ^b	31	28	28	U(10)	U(10)\U(10) ^b	U(10)
TSS (mg/L)	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	3	U(1)	2\3 ^a	17	3\3 ^c	2	2\2 ^d
24 hrs	2	3	5	4\3 ^b	2	1	1
2 days	3	3	4	2\2 ^b	1	1	1\1 ^b
3 days	3	3	3	3	2	1	1\1\1 ^{a,c}
6/7 days	3	4	4	3\3 ^b	1	U(1)	1\1 ^c
12 days	3	4	4	3\4 ^b	U(1)	U(1)	U(1)
Diquat (ug/L)	ST-1	ST-2	ST-3	ST-4	GR-1	GR-2	GR-3
< 4 hrs	U(0.5)	84.9	21.9\9.0 ^a	U(0.50)	U(0.5)	U(0.5)	U(0.5)/60 ^a
24 hrs	57.1	87.0	47.1	91.2	0.6	1.3	2.8
2 days	43.1	74.1	77.7	73.9	1.6	1.3	1.3
3 days	54.7	58.7	32.5	35.5	0.6	1.0	1.2\0.6 ^a
6/7 days	14.5	7.2	18.0	16.0	U(0.5)	U(0.5)	U(0.5)
12 days	6.8	3.1	3.2	4.7	U(0.5)	0.5	U(0.5)

na=not analyzed

J=not detected at concentration in parentheses

j=estimated concentration

^afield replicates^blaboratory duplicates

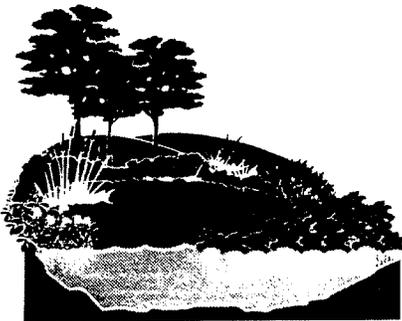
CONDITIONS OF SALE AND
LIMITATION OF WARRANTY AND LIABILITY

NOTICE: Read the entire Directions for Use and Conditions of Sale and Limitation of Warranty and Liability before buying or using this product. If the terms are not acceptable, return the product at once, unopened, and the purchase price will be refunded.

ZENECA Professional Products

REWARD[®]
Aquatic and Noncrop Herbicide

COMPLETE DIRECTIONS FOR USE



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The Directions for Use of this product should be followed carefully. It is impossible to eliminate all risks inherently associated with the use of this product. Crop injury, ineffectiveness or other unintended consequences may result because of such factors as manner of use or application, weather or crop conditions, presence of other materials or other influencing factors in the use of the product, which are beyond the control of ZENECA or Seller. All such risks shall be assumed by Buyer and User, and Buyer and User agree to hold ZENECA and Seller harmless for any claims relating to such factors.

ZENECA warrants that this product conforms to the chemical description on the label and is reasonably fit for the purposes stated in the Directions for Use, subject to the inherent risks referred to above, when used in accordance with directions under normal use conditions. This warranty does not extend to the use of this product contrary to label instructions, or under abnormal conditions or under conditions not reasonably foreseeable to or beyond the control of Seller or ZENECA, and Buyer and User assume the risk of any such use. ZENECA MAKES NO WARRANTIES OF MERCHANTABILITY OR OF FITNESS FOR A PARTICULAR PURPOSE NOR ANY OTHER EXPRESS OR IMPLIED WARRANTY EXCEPT AS STATED ABOVE.

In no event shall ZENECA or Seller be liable for any incidental, consequential or special damages resulting from the use or handling of this product. **THE EXCLUSIVE REMEDY OF THE USER OR BUYER, AND THE EXCLUSIVE LIABILITY OF ZENECA AND SELLER FOR ANY AND ALL CLAIMS, LOSSES, INJURIES OR DAMAGES (INCLUDING CLAIMS BASED ON BREACH OF WARRANTY, CONTRACT, NEGLIGENCE, TORT, STRICT LIABILITY OR OTHERWISE) RESULTING FROM THE USE OR HANDLING OF THIS PRODUCT, SHALL BE THE RETURN OF THE PURCHASE PRICE OF THE PRODUCT OR, AT THE ELECTION OF ZENECA OR SELLER, THE REPLACEMENT OF THE PRODUCT.**

ZENECA and Seller offer this product, and Buyer and User accept it, subject to the foregoing conditions of sale and limitations of warranty and of liability, which may not be modified except by written agreement signed by a duly authorized representative of ZENECA.

REWARD[®] is a trademark of a ZENECA Group Company.
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**DO NOT USE THIS PRODUCT
FOR REFORMULATION
KEEP OUT OF REACH OF CHILDREN
WARNING—AVISO**

Si usted no entiende la etiqueta, busque a alguien para que se la explique a usted en detalle. (If you do not understand the label, find someone to explain it to you in detail.)

ACTIVE INGREDIENT:
Diquat dibromide [6,7-dihydrodipyrido (1,2-a:2',1'-c) pyrazinedium dibromide]... 36.4%
INERT INGREDIENTS..... 63.6%
TOTAL..... 100.0%

Contains 2 lbs. diquat cation per gal. as 3.73 lbs. salt per gal.
EPA Reg. No. 10182-353

STATEMENT OF PRACTICAL TREATMENT
IF SWALLOWED: IMMEDIATELY give water or milk to drink and induce vomiting by inserting finger in throat. Do not induce vomiting or give anything by mouth to an unconscious person. Take person and product container to the nearest hospital or physician fast. **PROMPT TREATMENT IS ESSENTIAL TO COUNTERACT POISONING** and should be initiated before signs and symptoms of injury appear.

IF ON SKIN: IMMEDIATELY wash with soap and water. See a doctor if diquat contacts a skin cut, abrasion or area of irritation.

IF IN EYES: IMMEDIATELY wash eyes with water for at least 15 minutes and get medical attention.

IF INHALED: IMMEDIATELY get away from spray mist. Stop and check spray procedure. See a doctor if irritation persists.

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**TO PREVENT ACCIDENTAL POISONING,
NEVER PUT INTO FOOD, DRINK OR
OTHER CONTAINERS AND USE STRICTLY
IN ACCORDANCE WITH ENTIRE LABEL**

NOTE TO PHYSICIANS: Call ZENECA Medical Emergency Information Network 1-800-F-A-S-T-M-E-D (327-8633) at any hour to obtain toxicology information and a diquat analysis. To be effective, treatment for diquat poisoning must begin **IMMEDIATELY**. Treatment consists of binding diquat in the gut with suspensions of activated charcoal or bentonite clay, administration of cathartics to enhance elimination, and removal of diquat from the blood by charcoal hemoperfusion or continuous hemodialysis.

**FOR 24-HOUR EMERGENCY MEDICAL
ASSISTANCE CALL 1-800-F-A-S-T-M-E-D
(327-8633)**
**FOR CHEMICAL EMERGENCY: Spill, leak, fire,
exposure, or accident call CHEMTREC 1-800-
424-9300.**

**PRECAUTIONARY STATEMENTS
HAZARDS TO HUMANS AND
DOMESTIC ANIMALS
WARNING**

MAY BE FATAL IF SWALLOWED, INHALED OR ABSORBED THROUGH THE SKIN. CAUSES SUBSTANTIAL, BUT TEMPORARY, BUT TEMPORARY, EYE INJURY. CAUSES SKIN IRRITATION. CONTACT WITH IRRITATED SKIN, OR A CUT, OR REPEATED CONTACT WITH INTACT SKIN MAY RESULT IN POISONING. Do not get in eyes, on skin or on clothing. Do not breathe spray mist. Do not feed forage from treated crops to livestock. Keep livestock and pets out of treated fields and crop areas.

PERSONAL PROTECTIVE EQUIPMENT

Applicators and other handlers must wear:

- Coveralls over short-sleeved shirt and short pants or coveralls over long-sleeved shirt and long pants.
- Waterproof gloves.
- Chemical-resistant footwear plus socks.
- Protective eyewear.
- Chemical-resistant headgear for overhead exposure.
- Chemical-resistant apron when cleaning equipment, mixing, or loading.

EXCEPTION: Applicators for AQUATIC SUB-SURFACE APPLICATIONS must wear (Note—Mixers and Loaders for this application method must still wear the personal protective equipment (PPE) as described in the above section):

- Short-sleeved shirt and short pants.
- Waterproof gloves.
- Chemical-resistant footwear plus socks.

Discard clothing and other absorbent materials that have been drenched or heavily contaminated with this product's concentrate. Do not reuse them. Follow manufacturer's instructions for cleaning/maintaining PPE. If no such instructions for washables, use detergent and hot water. Keep and wash PPE separately from other laundry.

When handlers use closed systems, enclosed cabs, or aircraft in a manner that meets the requirements listed in the Worker Protection Standard (WPS) for agricultural pesticides [40 CFR 170.240(d)(4-6)], the handler PPE requirements may be reduced or modified as specified in the WPS.

User Safety Recommendations

Users should:

- Wash hands before eating, drinking, chewing gum, using tobacco or using the toilet.
- Remove clothing immediately if pesticide gets inside. Then wash thoroughly and put on clean clothing.
- Remove PPE immediately after handling this product. Wash the outside of gloves before removing. As soon as possible, wash thoroughly and change into clean clothing.

**ENVIRONMENTAL HAZARDS
(AQUATIC AND NONAQUATIC USES)**

This pesticide is toxic to wildlife. Do not apply directly to water except as specified on this label. **For Terrestrial Uses**, do not apply directly to water, or to areas where surface water is present or to intertidal areas below the mean high water mark. Do not contaminate water when disposing of equipment washwaters. Treatment of dense weed areas may result in oxygen loss from decomposition of dead weeds. This loss of oxygen may cause fish suffocation. Therefore, treat only 1/3 to 1/2 of the dense weed areas at a time and wait 14 days between treatments.

Necessary approval and/or Permits should be obtained prior to application if required. Consult the responsible State Agencies (i.e., Fish and Game agencies or Department of Natural Resources) before making applications to public waters.

DIRECTIONS FOR USE

It is a violation of Federal law to use this product in a manner inconsistent with its labeling.

Do not apply this product in a way that will contact workers or other persons, either directly or through drift. Only protected handlers may be in the area during application. For any requirements specific to your State or Tribe, consult the agency responsible for pesticide regulation.

READ ENTIRE LABEL. USE STRICTLY IN ACCORDANCE WITH PRECAUTIONARY STATEMENTS AND DIRECTIONS, AND WITH APPLICABLE STATE AND FEDERAL REGULATIONS.

DIRECTIONS

REWARD® Aquatic and Noncrop Herbicide is a nonvolatile herbicidal chemical for use as a general herbicide to control weeds in noncrop and aquatic areas. Absorption and herbicidal action is usually quite rapid with effects visible in a few days. REWARD Aquatic and Noncrop Herbicide controls weeds by interfering with photosynthesis within green plant tissue. Weed plants should be succulent and actively growing for best results. Rinse all spray equipment thoroughly with water after use. **AVOID SPRAY DRIFT** to crops, ornamentals, and other desirable plants during application as injury may result. Application to muddy water may result in reduced control. Minimize creating muddy water during application. Use of dirty or muddy water for diquat dilution may result in reduced herbicidal activity. **Avoid applying under conditions of high wind and wave action.**

Do not apply this product through any type of irrigation system.

NON-AGRICULTURAL USE REQUIREMENTS

The requirements in this box apply to uses of this product that are NOT within the scope of the Worker Protection Standard for agricultural pesticides (40 CFR Part 170). The WPS applies when this product is used to produce agricultural plants on farms, forests, nurseries, or greenhouses.

Keep all unprotected persons out of operating areas or vicinity where there may be drift.

For terrestrial uses, do not enter or allow entry of maintenance workers into treated areas, or allow contact with treated vegetation wet with spray, dew or rain, without appropriate protective clothing until spray has dried.

For aquatic uses, do not enter treated areas while treatments are in progress.

Do not allow swimming in treated areas within 24 hours after treatment.

Certain states may require more restrictive reentry intervals; consult your State Department of Agriculture for further information.

Written or oral warnings regarding use of protective clothing and accidental exposure must be given to workers who are expected to be in treated areas or in areas about to be treated. Oral warnings must be given if there is reason to believe that written warnings cannot be understood by workers.

When oral warnings are given, warnings shall be given in a language customarily understood by workers and must indicate the specific period of time that treated areas may not be entered without protective clothing. Oral warnings must also include appropriate first aid instructions in case of accidental exposure. These instructions are given in the STATEMENT OF PRACTICAL TREATMENT at the beginning of this label.

**NON-AGRICULTURAL USE
REQUIREMENTS (cont'd)**

Written warnings must include the following information, "WARNING. Area treated with REWARD Aquatic and Noncrop Herbicide on (date of application). Do not enter treated areas without appropriate protective clothing until spray has dried (or other reentry interval if your State has a more restrictive interval for this product). If accidental exposure occurs, follow the instructions below." (Written warnings must include the STATEMENT OF PRACTICAL TREATMENT given at the beginning of this label).

NONCROP OR NONPLANTED AREAS: Rights-Of-Way, including Railroads, Highways, Roads, Dividers and Medians, Pipelines, Public Utility Lines, Including Pumping Stations, Transformer Stations and Substations, Around Electric Utilities, Commercial Buildings, Manufacturing Plants, Storage Yards, Rail Yards, Fence Lines and Parkways, Edges and Nonflooded Portions of Ponds, Lakes and Ditches. Also Around Ornamental Gardens, Walkways, Patios, Beneath Greenhouse Benches, Along Driveways and on or Around Golf Courses. To Kill Undesirable Above-Ground Grass and Broadleaf Weed Growth—1 to 2 qts. plus 8-16 oz. of a 75% nonionic spreader per 100 gals. water (4 teaspoonfuls REWARD Aquatic and Noncrop Herbicide plus 1 teaspoonful of a 75% nonionic spreader to 1 gal. water). Apply for full coverage and thorough weed contact. Apply to young weeds since control decreases as weeds mature. Retreatment may be necessary to control grasses and established weeds. Avoid spray contact with foliage of food crops or ornamental plants.

TURF RENOVATION (All Turf Areas Except Commercial Sod Farms)

To desiccate golf course turf and other turf areas prior to renovation, apply 1 to 2 quarts of Diquat Herbicide plus 8-16 ounces of a 75% nonionic spreader per 100 gallons of water (4 teaspoons of Diquat Herbicide plus 1 teaspoon of a 75% nonionic spreader per 1 gallon of water) using ground spray equipment. Apply for full coverage and thorough contact with the turf grass. Apply only when the turf is dry, free from dew and incidental moisture.

Avoid spray contact with, or spray drift to, foliage of ornamental plants of food crops.

Do not graze livestock on treated turf or feed treated thatch to livestock.

DORMANT ESTABLISHED BERMUDAGRASS (Nonfood or Feed Crop)

For control of emerged annual broadleaf and grass weeds, including Little Barley*, Annual Bluegrass, Bromes including Rescuegrass, Sixweeks fescue, Henbit, Buttercup, and Carolina Geranium in established dormant bermudagrass lawns, parks, golf courses, etc.

Apply 1 to 2 pts. REWARD Aquatic and Noncrop Herbicide per acre in 20 to 100 gallons spray mix by ground as a broadcast application. Add 16-32 oz. of a nonionic surfactant per 100 gallons spray mixture.

Bermudagrass must be dormant at application. Application to actively growing bermudagrass may cause delay or permanent injury. Users in the extreme Southern areas should be attentive to the extent of dormancy at the time of application.

*For control of Little Barley, apply REWARD Aquatic and Noncrop Herbicide prior to the mid boot stage.

AQUATIC USE DIRECTIONS

U.S., except Florida: For application only to ponds, lakes, and drainage ditches where there is little or no outflow of water and which are totally under the control of the product's user.

FOR USE ONLY BY THE CORPS OF ENGINEERS OR OTHER FEDERAL OR STATE PUBLIC AGENCIES, OR BY CONTRACTORS OR LICENSEES (CERTIFIED APPLICATORS) UNDER THEIR DIRECT CONTROL.

Florida: For application only to ponds, lakes, and drainage ditches where there is little or no outflow of water and which are totally under the control of the product's user.

U.S., including Florida: For application to ponds, lakes, reservoirs, marshes, bayous, drainage ditches, canals, streams, rivers and other slow moving or quiescent bodies of water for control of aquatic weeds.

Necessary approval and/or Permits should be obtained if required. Consult the responsible State Agencies (i.e., Fish and Game agencies or Department of Natural Resources) before making applications.

Treated water may be used according to the following table or until such time as an approved assay (example: PAM II Spectromatic Method) shows that the water does not contain more than 0.01 part per million of diquat dibromide (calculated as the cation):

WATER USE RESTRICTIONS FOLLOWING APPLICATIONS WITH REWARD AQUATIC AND NONCROP HERBICIDE (Days)

Application Rate **	Drinking	Swimming	Livestock Consumption	Irrigation Turf/ Nonfood Crop	Irrigation Food Crops
2 gal./surface acre	14 days	1 day	14 days	5 days	14 days
1 gal./surface acre	14 days	1 day	14 days	5 days	14 days
0.75 gal./surface acre	14 days	1 day	14 days	5 days	14 days
0.50 gal./surface acre	14 days	1 day	14 days	5 days	14 days
Spot Spray* (< 0.5 gal./surface acre)	14 days	1 day	14 days	5 days	14 days

*Rates refer to total surface area for spot spray.
 **For water with an average depth less than 2 feet, use no more than 1 gal./surface acre.

No applications are to be made in areas where commercial fish processing, resulting in the production of fish protein concentrate or fish meal, is practiced. Before application, coordination and approval of local and/or State authorities must be obtained.

Apply REWARD Aquatic and Noncrop Herbicide in accordance with the following table:

WEED SPECIES	SUBSURFACE OR BOTTOM PLACEMENT GALS/SURFACE ACRE	SURFACE GALS/SURFACE ACRE
Bladderwort (<i>Utricularia</i> spp.)	1-2	2
Coontail (<i>Ceratophyllum demersum</i>)	2	2
Elodea (<i>Elodea</i> spp.)	2	2
Naiad (<i>Najas</i> spp.)	1-2	2
Pondweeds ¹ (<i>Potamogeton</i> spp.)*	2	2
Watermilfoils (<i>Myriophyllum</i> spp.)	1-2	2
Hydrilla (<i>Hydrilla verticillata</i>)	2	2
Waterlettuce ² (<i>Pistia Stratiotes</i>)	NA	0.5-0.75
Waterhyacinth ³ (<i>Eichhornia crassipes</i>)	NA	0.5-0.75

¹Diquat controls *Potamogeton* species except Richardson's pondweed (*P. richardsonii*). For control of *P. robbinsii*, applications must be made when the plants are in the early stages of growth such as in Spring and early Summer.

²For salvinia, waterlettuce and water hyacinth, use the labeled rate of REWARD Aquatic and Noncrop Herbicide in 150 gallons water plus 1 pt. of 75% nonionic surfactant per acre for surface sprays and for aerial application for waterlettuce and water hyacinth control, apply the labeled rate of REWARD Aquatic and Noncrop Herbicide in 10 to 24 gallons water plus 1 qt. of 75% nonionic surfactant per acre.

(continued on next page)

APPLICATION: In mixed weed populations, use the high rate of application as indicated by weeds present.

SUBSURFACE APPLICATIONS: Where the submersed weed growth, especially Hydrilla, has reached the water surface, apply either in a water carrier or an invert emulsion through boom trailing hoses carrying nozzle tips to apply the dilute spray below the water surface to insure adequate coverage.

BOTTOM PLACEMENT: Where the submersed weeds, especially Hydrilla, Bladderwort, and Coontail growth have reached the water surface or where water is slowly moving through the submersed weed growth that has reached the water surface, especially Hydrilla, Bladderwort, and Coontail, control may be enhanced when applied in an invert emulsion carrier injecting diluted REWARD Aquatic and Noncrop Herbicide near the bottom with weighted hoses. The addition of a copper-based herbicide will improve control. Where algae are present along with the submersed weeds, pretreatment with copper sulphate at recommended rates is advised for best results.

SURFACE APPLICATION: Apply REWARD Aquatic and Noncrop Herbicide either as concentrate slowly poured directly from the container in strips or as a spray in sufficient carrier. Applications should be made to ensure complete coverage of the weed areas. In mixed weed populations, use the high rate of application as indicated by weeds present. For waters less than 2 feet in average depth, use a maximum of 1 gallon REWARD Aquatic and Noncrop Herbicide per surface acre.

STORAGE AND DISPOSAL

PROHIBITIONS: Do not contaminate water, food or feed by storage, disposal or cleaning of equipment. Open dumping is prohibited.

STORAGE: Keep pesticide in original container. Do not put concentrate or dilute into food or drink containers. Do not contaminate feed, foodstuffs or drinking water. Do not store or transport near feed or food. Store at temperature above 32°F. For help with any spill, leak, fire or exposure involving this material, call CHEMTREC (1-800-424-9300).

PESTICIDE DISPOSAL: Pesticide wastes are toxic. Improper disposal of excess pesticide, spray mixture, or rinsate is a violation of Federal Law. If these wastes cannot be disposed of by use according to label instructions, contact your State Pesticide or Environmental Control Agency, or the Hazardous Waste representative at the nearest EPA Regional Office for guidance.

CONTAINER DISPOSAL: Triple rinse (or equivalent). Do not reuse container. Incinerate, burn, or puncture and dispose of in a sanitary landfill, or dispose of by other procedures allowed by State and local authorities. If burned, stay out of smoke.

WEED SPECIES	SUBSURFACE OR BOTTOM PLACEMENT GALS/SURFACE ACRE	SURFACE GALS/SURFACE ACRE
Pennywort ¹ (<i>Hydrocotyle</i> spp.)	NA	0.5-0.75
Salvinia ² (<i>Salvinia</i> spp.)	NA	0.5-0.75
Duckweed ³ (<i>Lemna</i> spp.)	NA	1
Cattails ⁴ (<i>Typha</i> spp.)	NA	1-2
Algae ⁵ (<i>Spirogyra</i> spp. & <i>Pithophora</i> spp.)	1-2	2

¹For Pennywort and cattail control, apply in 100 gallons of water plus 1 pt. 75% nonionic surfactant per acre for full coverage and thorough weed contact. Repeat treatments may be necessary to control regrowth. For best results, apply before flowering (cattail).

²For duckweed control, apply as an overall spray in 50-150 gallons of water plus 1 pt. 75% nonionic surfactant per acre. Retreatment may be necessary for plants missed in previous applications and regrowth.

³For suppression of certain filamentous algae species including *Spirogyra* and *Pithophora*, apply according to the submersed use directions. For water less than 2 feet in average depth, use a maximum of 1 gallon REWARD Aquatic and Noncrop Herbicide per surface acre.

STORAGE AND DISPOSAL (cont'd)**FOR BULK AND MINI-BULK CONTAINERS:**

CONTAINER DISPOSAL: Reseal container and offer for reconditioning, or triple rinse (or equivalent) and offer for recycling or reconditioning, or clean in accordance with manufacturer's instructions.

CONTAINER PRECAUTIONS: Before refilling, inspect thoroughly for damage, such as cracks, punctures, bulges, dents, abrasions and damaged or worn threads on closure devices.

REFILL ONLY WITH REWARD AQUATIC AND NONCROP HERBICIDE.

The contents of this container cannot be completely removed by cleaning. Refilling with materials other than REWARD Aquatic and Noncrop Herbicide will result in contamination and may weaken container.

After filling and before transporting, check for leaks.

Do not refill or transport damaged or leaking container.

CONTAINER IS NOT SAFE FOR FOOD, FEED OR DRINKING WATER!