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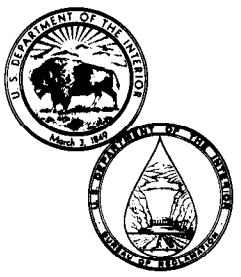
HERBICIDAL RESIDUES AND ENVIRONMENTAL EFFECTS FROM THE EXPERIMENTAL APPLICATION OF TWO 2,4-D FORMULATIONS TO CONTROL EURASIAN WATERMILFOIL

July 1983

Engineering and Research Center



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16. ABSTRACT

In response to the need for an effective and environmentally acceptable herbicide to control eurasian watermilfoil, a serious aquatic weed problem, an interagency research study was initiated involving the USBR (Bureau of Reclamation) and the COE (U.S. Army Corps of Engineers). This document reports the tolerance and label amendment efforts done by the USBR. Experimental small-plot applications of 2,4-D dimethylamine (DMA) and butoxyethanol ester (BEE) at rates of 20 and 40 lb/acre (22.5 and 45 kg/ha) were made at Banks Lake, Washington and Fort Cobb Reservoir, Oklahoma with subsequent water, hydrosoil, invertebrate organism, and fish flesh sample collection and analysis for residues, through 56-days posttreatment.

The temporary potable water tolerance of 0.1 mg/L (0.1 p/m) was exceeded in three 1-day posttreatment samples (of 1680 water samples analyzed) or 0.18 percent of the total samples. No dichlorophenols were found in water, but dimethylnitrosamines in the μg/L range were detected in a few pretreatment and posttreatment samples from both sites. Hydrosoil 2,4-D residues were low, with concentrations ranging from 0 to 0.316 μg/g in DMA treated areas and a maximum of 37 μg/g of soil resulting from a 45 kg/ha (40 lb/acre) BEE application. Trace amounts of 2,4-D dichlorophenol in the μg/L range were found in some hydrosoil pretreatment and posttreatment samples. Herbicide residues in fish flesh were well within the 1 mg/L (1 p/m) food additive tolerance, with no evidence of bioaccumulation.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. *DESCRIPTORS--* — /Weed control/ herbicides/ water analysis/ chemical analysis/ aquatic environment/ dispersion rate/ environmental effects/ water quality/ ecology/ phytoplankton/ zooplankton/ limnology/ gas chromatography

b. *IDENTIFIERS--* — 2,4-D/ Banks Lake/ Columbia Basin Project, WA/ Fort Cobb Reservoir

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July 1983

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UNITED STATES DEPARTMENT OF THE INTERIOR ★ BUREAU OF RECLAMATION

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The authors would also like to recognize Environmental Sciences Section staff members T. J. Parks, J. S. Thullen, J. E. Boutwell, S. J. Grabowski, F. L. Nibling, and V. S. Miyahara for their contributions in field sampling and laboratory support, and S. G. Campbell who processed plankton samples. In addition, members of the Chemistry, Petrography, and Chemical Engineering staff provided analytical data on the inorganic chemical analyses of water samples, authentication of a 2,4-D residue sample, and identification of an inorganic contaminant by mass spectrophotometric analyses.

N. E. Otto and J. C. Pringle, as coprincipal investigators, were responsible for initiation, development, and conduct of the study. D. Sisneros conducted the various 2,4-D herbicide residue analyses required, assisted by T. J. Parks and J. S. Thullen.

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INTRODUCTION

Eurasian watermilfoil is a rooted aquatic weed that is creating serious problems for the management and use of water in reservoirs and other impoundments in the United States and elsewhere in the world. In responding to the need for an effective and acceptable herbicidal control technique, an interagency effort between the USBR (Bureau of Reclamation) and the COE (U.S. Army Corps of Engineers) was initiated to amend established 2,4-D tolerances and registrations. Such an amendment would permit nationwide use of this herbicide in conjunction with water level management and other measures in integrated programs for the control of eruasian watermilfoil in multiple-use reservoirs managed by Federal, state, or local government agencies or by certified applicators under contract to these agencies.

To accomplish this goal, a study was initiated to obtain residue data from experimental small-plot applications of two formulations of 2,4-D under EUP's (Experimental Use Permits) 11683-EUP-2 (liquid amine formulation) and 11683-EUP-3 (granular butoxyethanol ester formulation) granted by the EPA (U.S. Environmental Protection Agency) to the USBR and the COE with effective dates July 10, 1980, to February 28, 1982. This herbicide registration project was conducted with the cooperation of the Agricultural Products Company of Union Carbide Corporation. Union Carbide has a registered product for a similar use pattern for eruasian watermilfoil control on reservoirs managed by the Tennessee Valley Authority.

This is the final report on that portion of the study under the USBR's jurisdiction. Although the EUP became effective July 10, 1980, neither agency was able to initiate the studies until 1981 because of administrative problems. Each agency is reporting study results independently in support of this proposed registration.

APPLICATION

This study provides data that demonstrates the herbicide's dispersion characteristics and environmental effects of 2,4-D butoxy ethanol ester and dimethylamine formulations when applied to reservoirs for control of eruasian watermilfoil. Information in this report is being used to support a petition to obtain legal herbicide registration of 2,4-D for eruasian watermilfoil control.

The data in these investigations demonstrated that these experimental treatments did not adversely affect nontarget aquatic organisms or the aquatic ecosystem. The herbicide residues were considerably below established water, fish flesh, and crop residue tolerances for this herbicide's proposed use pattern.

These two 2,4-D herbicide formulations appear to be effective and environmentally acceptable for control of eurasian watermilfoil in USBR and similar reservoirs, when applied with consideration of water use patterns and at safe distances from potable and irrigation water inlets. The eventual use of the herbicide will depend on the establishment of legal tolerances and herbicide labeling by Federal and state regulatory agencies.

MATERIALS AND METHODS

Reservoir Application Sites

The experimental treatments were made on test plots located at Banks Lake, Washington, and Fort Cobb Reservoir, Oklahoma.

Banks Lake is a 43-km (27-mile) long reservoir with an active storage capacity of 881 945 000 m^3 (715 000 acre-ft). It was designed as an irrigation water-equalizing reservoir feeding Columbia River water into an irrigated area of 212 900 ha (526 000 acres). This reservoir is operated under the jurisdiction of the USBR's Columbia Basin Project. The reservoir also provides return-flow water to produce power during discharges into pump generators located at Franklin Delano Roosevelt Lake.

Fort Cobb Dam and Reservoir are located on the Washita River Basin in south-western Oklahoma approximately 97 km (60 mi) southwest of Oklahoma City near the town of Fort Cobb. This reservoir, with a maximum length of 10.6 km (6.6 mi), has a total capacity of 177 302 000 m^3 (143 740 acre-ft) at full flood control level and was designed as a multipurpose facility to provide municipal and industrial water to the cities of Fort Cobb and Anadarko and to the Western Farmers Electric Cooperative. The city of Chickasha, southeast of Fort Cobb Reservoir, has its own pumping plant near the east abutment of the dam. The reservoir, which does not supply water for irrigation purposes, is operated under the jurisdiction of the Fort Cobb Master Conservancy District.

Experimental Treatments

Herbicide formulations used were a liquid DMA (dimethylamine formulation of 2,4-D), Amchem Corporation's Weedar 64™, EPA Registration

No. 264-2; and granular BEE (butoxyethanol ester formulation of 2,4-D), Amchem Corporation's Aqua-Kleen™, EPA Registration No. 264-109. Applications were made at two rates 22.5 and 45 kg/ha (20 and 40 lb/acre) to a total of four plots at each of the two geographic locations.

Applications were originally scheduled to coincide with a period of operational treatment, i.e., late spring, to obtain maximum efficacy. The Banks Lake treatments were accomplished close to this desired timing. However, the experimental applications at Fort Cobb Reservoir were delayed until late August because of legal actions initiated by local citizens during the environmental assessment process. Litigation was eventually ruled upon by a Federal Court, and clearance was given for treatment in mid-August 1981.

The experimental herbicides were applied to Banks Lake by helicopter, using a hydraulic pressure boom sprayer for the DMA liquid formulation and a rotary centrifugal applicator for the BEE formulations. Surface applications were made at Fort Cobb Reservoir using a boat-mounted hydraulic sprayer for the liquid, and a granular-dropping spreader applicator for the ester formulation.

Figures 1 and 2 illustrate the herbicide plot locations on the two reservoirs. Rates of herbicide application, plot sizes, and treatment dates were as follows:

Banks Lake

Plot 1
- Size : 14.8 ha (36.9 acres)
- Formulation : DMA, lot No. S-75-154
- Rate : 22.5 kg/ha, ae (20 per acre, acid equivalent)
- Treatment date: July 8, 1981

Plot 2
- Size : 16.0 ha (39.9 acres)
- Formulation : DMA, lot No. S-75-154
- Rate : 45 kg/ha, ae (40 lbs per acre, acid equivalent)
- Treatment date: July 8, 1981

Plot 3
- Size : 16 ha (40 acres)
- Formulation : BEE, lots No. A 09006-1, -2, and A 0900-7-1
- Rate : 45 kg/ha, ae (40 lbs per acre, acid equivalent)
- Treatment date: July 8, 1981

Plot 4
- Size : 16 ha (40 acres)
- Formulation : BEE, lots No. A 08088-2 and -3
- Rate : 22.5 kg/ha, ae) (20 lbs per acre, acid equivalent)
- Treatment date: July 8, 1981

Fort Cobb Reservoir

Plot 1
- Size : 12.8 ha (32.1 acres)
- Formulation : BEE, lot No. 41929
- Rate : 22.5 kg/ha, ae) (20 lbs per acre, acid equivalent)
- Treatment date: August 24, 1981

Plot 2
- Size : 5.8 ha (14.4 acres)
- Formulation : BEE, lot No. 41929
- Rate : 45 kg/ha, ae (40 lbs per acre, acid equivalent)
- Treatment date: August 25, 1981

Plot 3
- Size : 12.8 ha (32.1 acres)
- Formulation : DMA, lot No. 41440 S 68075
- Rate : 45 kg/ha, ae (40 lbs per acre, acid equivalent)
- Treatment date: August 20, 1981

Plot 4
- Size : 7.6 ha (18.9 acres)
- Formulation : DMA, lot No. 41440 S 68075
- Rate : 22.5 kg/ha, ae (20 lbs per acre, acid equivalent)
- Treatment date: August 19, 1981

Sample Collection

Samples of water hydrosoil, and fish were collected for chemical analysis 1-day pretreatment, and 1-, 4-, 7-, 14-, 28-, and 56-days posttreatment.

Two replicated sample sites were established within the treated plots. Three sample sites were established outside of the herbicide treated area to determine herbicide movement out of the plot. Invertebrate animal population samples were also collected at the same time water quality determinations were made. A summary of the field sampling schedule is given in table 1. The detailed study protocol is presented in the appendix A of this report and includes copies of the supplemental labeling for the EUP and Weedar 64™ and Aqua-Kleen™ labels. Reservoir water,

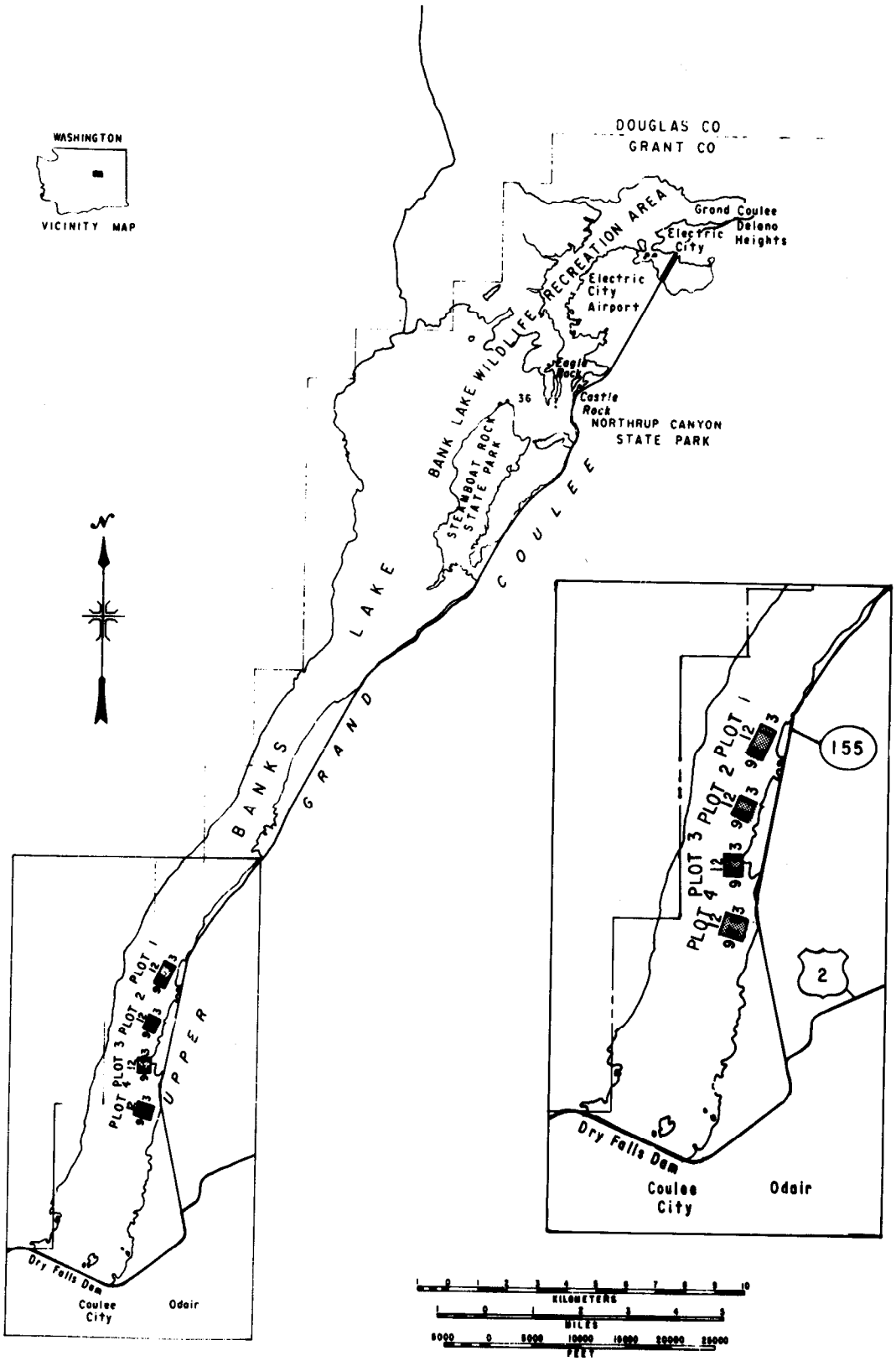


Figure 1.—Banks Lake.

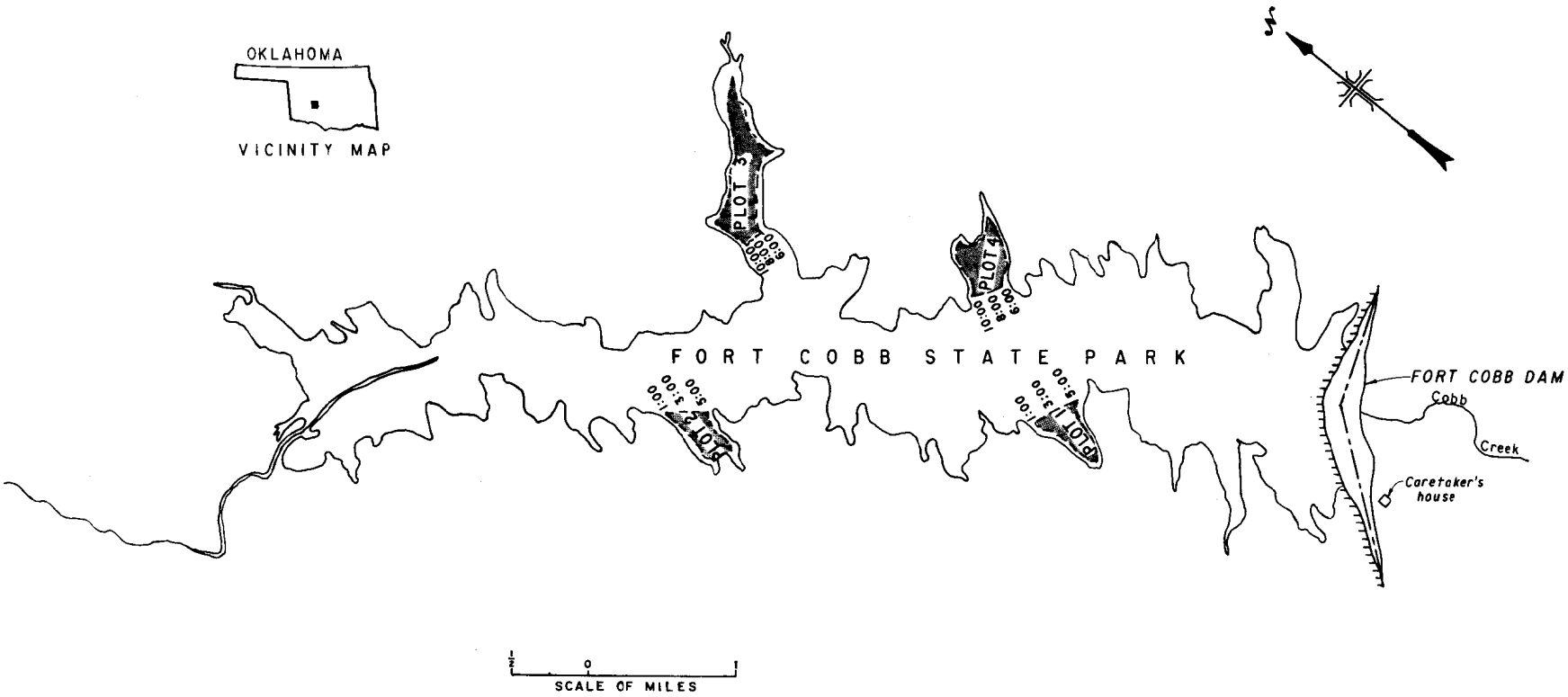


Figure 2.—Fort Cobb Reservoir.

hydrosoil, and fish fillet samples were stored in ice immediately upon collection and transported for deep freezing within 4 to 5 hours of collection. These frozen samples were transported by airfreight to the USBR analytical laboratory in Denver, Colorado using insulated shipping cases containing dry ice. Samples were received at the Denver laboratory within 12 to 24 hours after shipment. All samples were maintained in frozen condition until ready for analysis.

Invertebrate animal samples were collected in vertical hauls utilizing a Wisconsin plankton sampling net. Samples were immediately preserved in formalin solution for subsequent organism identification and enumeration by procedures published in Standard Methods for Examination of Water and Wastewater [1]*. Diversity indices were calculated using Wilhm and Dorris' formula for determining $\bar{d}$ [2].

Observations were made of existing rooted macrophytes within the treated plots by "grab sampling" techniques. Samples were identified and empirical estimates of relative density were made. In situ determinations of water quality parameters were made including pH, dissolved oxygen, conductivity, and temperature at each sampling site. A Hydrolab Corporation model 4000 data acquisition system was used to measure these parameters.

Additional water samples were collected and frozen for water chemistry analysis to determine pH, all major ions, conductivity, and total dissolved solids according to procedures in the National Handbook of Recommended Methods for Water Data Acquisition [3]. The results included in appendix B, were used to determine possible limnological changes that might occur as a result of the experimental treatments.

Determination of 2,4-D and Product Residues

Water samples were analyzed for 2,4-D acid residues and 2,4-dichlorophenol in simultaneous sample runs using Waters Associates Sep-Pak C18™ cartridges for concentration of components, followed by analysis on a Waters Associates HPLC (high performance liquid chromatograph) [4]. On completion of HPLC analysis, aliquots of representative samples were analyzed using a gas-liquid chromatograph to authenticate the comparability of the two techniques. Also, 2,4-D chromatograms were authenticated using comparative standards in mass spectrophotometric analysis. Separate analyses were made to determine the possible occurrence of dimethylnitrosamines (Appendix A, attachment 4). Hydrosoils were extracted, and possible residues of 2,4-D acid and any dichlorophenol were determined using HPLC analytical procedures developed by the authors as given in the Appendix, Attachment 5. Fish flesh analysis used HPLC modified procedures published by Hesselburg and Johnson [5].

Detailed residue analysis procedures are included in the "Protocol for 2,4-D Residue Dissipation Studies" in the appendix. Also included are analytical traces typical of water, hydrosoil, and fish flesh analytical techniques. The 2,4-dichlorophenol content of each water sample was determined in conjunction with parent 2,4-D acid determinations.

RESULTS AND DISCUSSION

2,4-D Residues in Water

Sample collection through 56 days' posttreatment was completed at Banks Lake on September 2, 1981, and at Fort Cobb Reservoir on October 15, 1981. Residue determinations of 2,4-D in water were completed on all samples through 14 days' posttreatment using HPLC analytical procedures. The methodology and a typical chromatogram are presented in the Study Protocol in appendix A of this report. As a result of low residue levels found in 14-day posttreatment samples, later samples were not analyzed except for some profile data representing typical 28- and 56-day residue levels. The resulting residues of 2,4-D found in each of the samples collected are presented in tables 2 through 9.

Pretreatment water samples collected from Banks Lake had no detectable 2,4-D residues. However, trace amounts of 2,4-D residue at levels of one microgram per liter (μg/L) or less were found at Fort Cobb Reservoir. It is assumed that because Fort Cobb Reservoir is located in an intensive agricultural area that some surface drainage contamination could account for this minor background residue. Banks Lake is more remote from an intensively cultivated area and would be less apt to have a 2,4-D residue background.

Dissipation of 2,4-D in Water

The data in tables 2 through 9 are presented in figures 3 through 10. Figures 3 through 6 depict

* Numbers in brackets refer to entries in the bibliography.

Table 1.—*1981 2,4-D study, Fort Cobb Reservoir, Oklahoma, and Banks Lake, Washington, summary of field sampling schedule.*

Samples to be collected per plot at each sample time	Total number of samples
In or near each herbicide plot:	
1. Water.—Collect triplicate 1-liter samples from 0.3-m (1-ft) below the surface and near the bottom from two separate stations (designated as sample site numbers 1 and 2) within each of the treated plots. In addition, collect triplicate 1-L samples from two depths at three separate sampling stations adjacent to each plot. These sampling sites at Banks Lake are designated sample site numbers 9, 12 and 3. Sample sites outside of the treated plots at Fort Cobb Reservoir were arranged in a row on the reservoir side outside of the treated bays and were designated as sample site numbers 1:00, 3:00, and 5:00 on BEE and 6:00, 8:00 and 10:00 on DMA plots. (See figs. 1 and 2)	30
2. Hydrosoils.—Using the same two stations within each treated plot, select two random sampling sites within a 9- to 18-m (10- to 20-yd) radius. Obtain a single sample from each of the two stations within the treated plot and from each of the two random sites.	4
3. Invertebrates.—Sample with a Wisconsin net using vertical hauls from a depth of 3-m (10-ft) to the water surface. Collect duplicate samples from one site within each plot. Also, collect duplicate samples from one site outside each plot, but within 90- to 180-m (100- to 200- yd).	4
4. Water quality.—Obtain a 1-L water sample for total chemical analysis. Sample, analyze, and record while on the plot (using Hydrolab) the DO (dissolved oxygen), pH, conductivity, and water temperature.	
5. Fish.—In the vicinity of the treated plots, obtain six samples of two to five species of resident game fish, each of which are 178- to 254-mm (7- to 10-in) length, using gill net or electrofishing. (Duplicate nets may be required to collect selected species at different depths).	6
At or near the outlet works:	
1. Water.—Collect triplicate 1-L samples at middepth.	3
2. Invertebrates.—Collect duplicate samples from one location near the water sampling station. Collect samples with vertical hauls from a depth of 3-m (10-ft) to the water surface.	2
3. Water quality.—Obtain a 1-L water sample for total chemical analysis. Sample and record DO, pH, conductivity, temperature, and ORP (oxidation/reduction potential).	1
4. Hydrosoils.—None.	0
5. Fish.—Obtain six samples of two to five species of resident game fish, each 178- to 254-mm (7- to 10-in) in length, using gill net or electrofishing in the vicinity of the outlet works. (Duplicate nets may be required to collect selected species at different depths.	6

2,4-D dissipation patterns for the four experimental plots at Banks Lake. Figures 7 through 10 illustrate 2,4-D dissipation patterns in water at the Fort Cobb Reservoir study sites. The plotted values given in these graphs are the means of the three sample replications. Data from sample sites 1 and 2 collected within each herbicide treated plot were combined into one graph. Also, data from samples taken from outside each plot are included to show the characteristics of herbicide movement outside of the study plots. Surface to 0.3 m (1-ft) samples are plotted separately from near-bottom samples to illustrate possible differences in dissipation in the treated water column over 14 days. In addition, the statistical computation of the combined replicates within treated plots (sites 1 and 2) was developed to determine concentration versus time to demonstrate a concentration decay curve for each treatment. The regression equation and the correlation coefficients are given on each figure. Each herbicide treatment and rate of application is discussed individually.

BANKS LAKE, plot 1, 22.5 kg/ha, ae (20 lb/acre), DMA (table 2, fig. 3).—Surface water residues within the treated plot 1-day posttreatment averaged 199.8 μg/L (199.8 p/b) with one sample reaching 227.0 μg/L. This level, although exceeding the 100 μg/L (0.1 p/m) established water tolerance for 2,4-D in potable water and the 100 μg/L (0.1 p/m) level accepted in irrigation canal water, rapidly declined to an average near 5 μg/L (5 p/b) in 4 days and dissipated to levels averaging 2 μg/L (2 p/b) by 14 days. At 28 days, this level dropped to 1 μg/L (1 p/b) or less. Bottom water sample residues were very low with means never exceeding 3 μg/L (3 p/b), declining to a low at 14 days' posttreatment of 1.9 μg/L (1.9 p/b), similar to the surface samples.

The residue decay curves for the treated plots show rapid decline within four days, with only trace amounts detectable at 7 and 14 days. This decline suggests that DMA dissipation is by simple diffusion throughout the water column, with movement in a fairly uniform pattern to areas outside the treated plot.

This diffusion was shown in untreated sample sites 3, 9, and 12. The higher concentrations at sites 3 and 12 were similar at surface 1-day posttreatment to the within-plot sites. This result can be attributed to a strong posttreatment prevailing wind from the southwest.

Regression curves for the surface samples show an excellent correlation of time versus concentration, with an $R^2 = 0.909$, indicating uniform diffusion of the 2,4-D residue. The bottom samples demonstrated more variability, but with a good correlation of $R^2 = 0.678$. Both decay curves could be considered useful for predictive purposes. It should be emphasized that the residue degradation mechanism within this plot would be primarily dispersion through wind action and convective mixing. Sorption loss would be minor because of minimal aquatic weed stands at the time of treatment. No temperature profile thermocline activity was expected in this plot because of the shallow depths, from 0.76- to 1.22-m (2.5- to 4-ft).

BANKS LAKE, plot 2, 45 kg/ha, ae (40 lb/acre) DMA (table 3, fig. 4).—The within-plot surface water residues treated at the 45 kg/ha rate averaged 96.9 μg/L (96.9 p/b) with a single maximum of 113 μg/L. Similar to the 22.5 kg/ha (20-lb/acre) treatment rate in plot 1, the 2,4-D residue rapidly decreased to an average level of 6.8 μg/L and declined to 2.6 μg/L in 14 days. This 2-μg/L level continued through 28 days, but no residue was detectable at 56 days. Bottom water samples exhibited considerably higher residue values 1-day posttreatment with a mean of 24.4 μg/L, and a single high value of 55 μg/L. After this time, however, the dissipation rate was similar to the 22.5 kg/ha rate except for a slight increase from 1.4 μg/L at 7 days to 2.4 μg/L at 14 days. A slight persistence was suggested by a 56-day posttreatment bottom water sample residue value of 2 μg/L. The surface water decay curve was very similar for the 22.5- and 45-kg/ha rates. The 45-kg/ha application rate produced a higher residue level initially in the bottom water, but there was little difference at the surface.

Untreated sample sites 3, 9 and 12 exhibited dissipation curves similar to the treated-plot area, indicating similar outward dispersion from the treated areas as found in plot 1. Some increase in the bottom water 2,4-D residues was observed at sample site 12 after 7 days, which follows the treated-plot trend. The waterflow pattern in the reservoir, which is from north to south in this equalizing reservoir, may have carried higher residue concentrations toward site 12. However, the residue was very low at 8.5 μg/L.

Water depths in the treated-plot area averaged 1- to 1.5-m (3- to 5-ft) and vegetation standing crop was minimal, similar to plot 1.

Regression curves of dissipation levels over the 14-day period show excellent correlation with

Table 2.—*Banks Lake, Washington, Plot 1[a], 2,4-D residue levels (mg/L) 1-, 4-, 7-, and 14-days, posttreatment.*

Site	Depth[d] m (ft)	Residue levels							
		1 day[e]		4 day		7 day		14 day	
[b] 1	.3, .3, .3, .3	0.2120		0.0080		0.0042		0.0034	
	(1, 1, 1, 1)	0.1839	(±0.0187)	0.0054	(±0.0018)	0.0035	(±0.0007)	0.0013	(±0.0017)
		0.1765		0.0046		0.0050		0.0012	
1	.76, 1.2, 1, 1	0.0060		0.0080		0.0065		0.0018	
	(2.5, 4, 3, 3)	0.0052	(±0.0006)	–	(±0.0030)	0.0015	(±0.0035)	0.0034	(±0.0009)
		–[f]		0.0037		–		0.0020	
[b] 2	.3, .3, .3, .3	0.2270		0.0046		0.0047		–	
	(1, 1, 1, 1)	0.2079	(±0.0177)	0.0051	(±0.0006)	0.0041	(±0.0007)	0.0026	(±0.0008)
		0.1917		0.0038		0.0056		0.0015	
2	1, 1, 1.2, 1	0.0219		0.0031		0.0012		0.0015	
	(3, 3, 4, 3)	0.0167	(±0.0026)	0.0039	(±0.0006)	0.0021	(±0.0006)	0.0012	(±0.0003)
		0.0184		0.0043		0.0023		0.0019	
[c] 3	.3, .3, .3, .3	0.0140		0.0067		0.0053		0.0029	
	(1, 1, 1, 1)	0.0154	(±0.0011)	0.0080	(±0.0007)	0.0034	(±0.0011)	0.0026	(±0.0005)
		0.0163		0.0077		0.0054		0.0019	
3	9.1, 3, 2.7, 3	0.0004		0.0001		0.0047		0.0009	
	(30, 10, 9, 10)	–	(±0.0001)	0.0001	(±0.0003)	–	(±0.0009)	0.0009	(±0.0000)
		0.0006		0.0006		0.0034		0.0008	
[c] 9	.3, .3, .3, .3	0.1166		0.0073		0.0048		0.0015	
	(1, 1, 1, 1)	0.0041	(±0.0795)	0.0073	(±0.0010)	0.0042	(±0.0005)	0.0020	(±0.0003)
		–		0.0090		0.0037		–	
9	2.4, 1.5, 1.5, 2.1	0.0006		0.0092		0.0027		0.0010	
	(8, 5, 5, 7)	0.0004	(±0.0025)	0.0064	(±0.0020)	0.0026	(±0.0004)	0.0023	(±0.0008)
		0.0009		–		0.0033		0.0007	
[c]12	.3, .3, .3, .3	0.1555		0.0073		0.0046		0.0024	
	(1, 1, 1, 1)	0.1570	(±0.0061)	0.0060	(±0.0007)	0.0034	(±0.0009)	0.0029	(±0.0003)
		0.1458		0.0073		0.0029		–	
12	9.1, 3.1, 5, 1.2	0.0004		0.0008		0.0016		0.0024	
	(30, 10, 5, 4)	0.0000	(±0.0002)	0.0001	(±0.0004)	0.0021	(±0.0003)	0.0015	(±0.0005)
		0.0000		0.0001		0.0016		0.0014	

[a] Treated with DMA at 22.5 kg/ha, ae (20 lb/acre, ae)
[b] Within treated plot sampling site.
[c] Outside of treated plot sampling site.
[d] The first depth was the depth from which the 1-day residual samples were taken; the second depth was for the 4-day residual samples; the third depth was the 7-day residual samples; and the fourth depth for the 14-day residual samples.
[e] Pretreatment (0 day) residue level 0.0000 mg/L.
[f] Hyphen indicates no sample was taken or the sample was lost during handling.

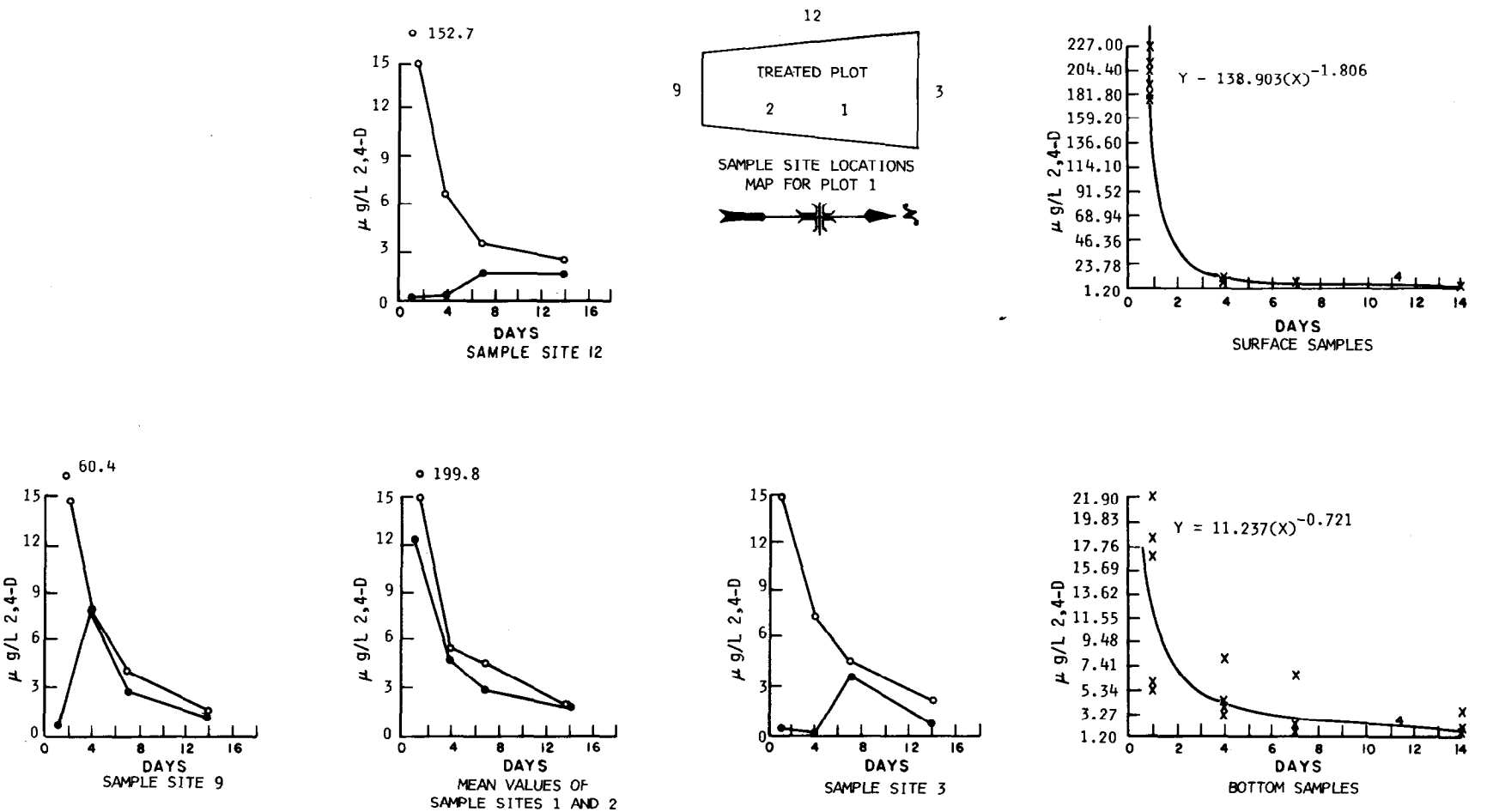


Figure 3.—Banks Lake 2,4-D dissipation in water, plot 1.

Dissipation of 22.5 kg/ha (20 lb/acre) DMA, in water over a 14-day period following treatment.
o = *water sampled near the surface.* • = *water sampled near the bottom.*

Table 3.—*Banks Lake, Washington, Plot 2[a], 2,4-D residue levels (mg/L) 1-, 4-, 7-, and 14-days, posttreatment.*

Site	Depth[d] m (ft)	Residue levels							
		1 day[e]		4 day		7 day		14 day	
[b] 1	.3, .3, .3, .3	0.0874		0.0049		0.0032		0.0019	
	(1, 1, 1, 1)	0.0970	(±0.0054)	0.0095	(±0.0023)	0.0060	(±0.0015)	0.0027	(±0.0005)
		0.0880		0.0076		0.0037		0.0027	
1	1, 1.5, 1.5, 1.2	0.0550		0.0031		0.0000		0.0027	
	(3, 5, 5, 4)	0.0455	(±0.0142)	0.0045	(±0.0014)	0.0028	(±0.0014)	0.0016	(±0.0010)
		0.0271		0.0017		0.0011		0.0036	
[b] 2	.3, .3, .3, .3	0.1135		0.0063		0.0050		0.0028	
	(1, 1, 1, 1)	0.1020	(±0.0010)	0.0063	(±0.0003)	0.0033	(±0.0008)	0.0026	(±0.0001)
		0.0941		0.0068		0.0043		0.0028	
2	1.2, 1.2, 1.2, 1.2	0.0047		0.0028		0.0028		0.0021	
	(4, 4, 4, 4)	0.0040	(±0.0034)	0.0031	(±0.0018)	0.0008	(±0.0011)	0.0026	(±0.0004)
		0.0102		0.0061		0.0009		0.0018	
[c] 3	.3, .3, .3, .3	0.0842		0.0042		0.0033		0.0033	
	(1, 1, 1, 1)	0.0860	(±0.0058)	0.0050	(±0.0005)	0.0036	(±0.0010)	0.0028	(±0.0004)
		0.0751		0.0041		0.0050		0.0025	
3	1.2, 1.5, 1.2, 1.5	0.0641		0.0016		0.0009		0.0013	
	(4, 5, 4, 5)	0.0332	(±0.0261)	0.0026	(±0.0006)	0.0019	(±0.0006)	0.0030	(±0.0012)
		0.0122		0.0028		0.0020		–	
[c] 9	.3, .3, .3, .3	0.1106		0.0067		0.0050		0.0035	
	(1, 1, 1, 1)	0.0498	(±0.0320)	0.0065	(±0.0020)	0.0040	(±0.0005)	0.0028	(±0.0005)
		0.0978		0.0101		0.0048		–	
9	2.0, 1.5, 1.2, 1.5	0.0011		–[f]		0.0013		0.0030	
	(6.5, 5, 4, 5)	0.0025	(±0.0023)	0.0008	(±0.0008)	0.0016	(±0.0003)	0.0018	(±0.0006)
		0.0056		0.0020		0.0020		0.0023	
[c] 12	.3, .3, .3, .3	0.0716		0.0054		0.0037		0.0020	
	(1, 1, 1, 1)	0.0646	(±0.0056)	0.0038	(±0.0008)	0.0038	(±0.0003)	0.0028	(±0.0006)
		0.0757		0.0049		0.0033		0.0031	
12	3, 3, 3.4, 3	0.0013		0.0014		0.0006		0.0010	
	(10, 10, 11, 10)	0.0032	(±0.0010)	0.0012	(±0.0003)	0.0017	(±0.0007)	0.0006	(±0.0002)
		0.0025		0.0018		0.0020		0.0010	

[a] Treated with DMA at 45 kg/ha, ae (40 lb/acre, ae)
[b] Within treated plot sampling site.
[c] Outside of treated plot sampling site.
[d] The first depth was the depth from which the 1-day residual samples were taken; the second depth was for the 4-day residual samples; the third depth was the 7-day residual samples; and the fourth depth for the 14-day residual samples.
[e] Pretreatment (0 day) residue level 0.000 mg/L.
[f] Hyphen indicates no sample was taken or the sample was lost during handling.

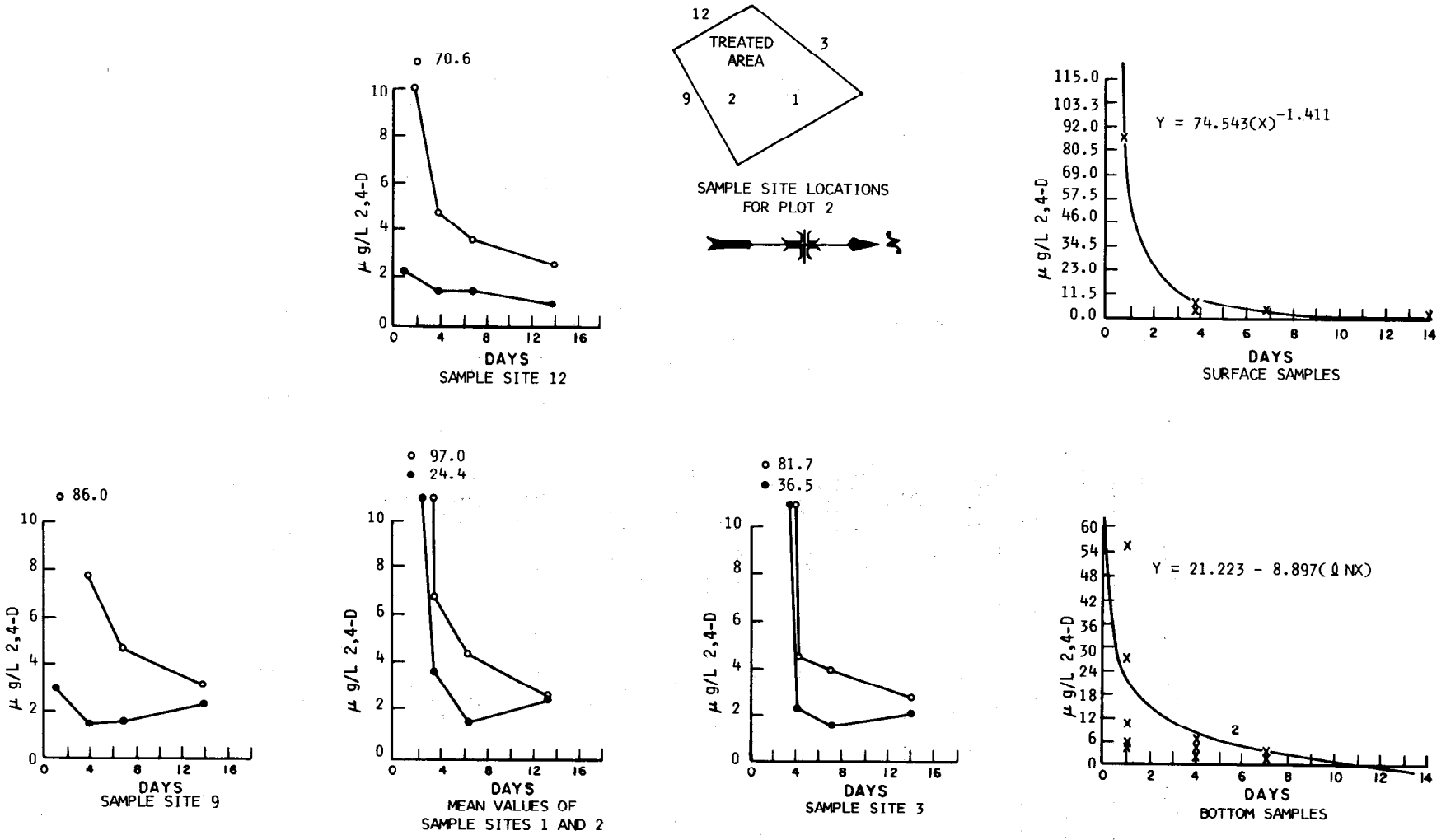


Figure 4.—Banks Lake 2,4-D dissipation in water, plot 2.

Dissipation of 45 kg/ha (40 lb/acre) DMA, in water over a 14-day period following treatment. o = water sampled near the surface. • = water sampled near the bottom.

R^2 = 0.932 in the surface waters. The bottom water dissipation curves are less well correlated with an R^2 = 0.338 because of a slight increase in residue levels between 7- and 14-days posttreatment. However, both statistical treatments suggest residue levels near or approaching zero at 14 days posttreatment. These regressions are similar to those found in the 22.5-kg/ha rate treatment.

BANKS LAKE, plot 3, 45 kg/ha, ae (40 lb/acre, ae), BEE (table 4, fig. 5).—Residue levels resulting from this ester formulation treatment exhibited a rapid decline through a 4 day period in both surface and bottom waters. A maximum individual sample value of 137.5 μg/L was observed with an average of 76.9 μg/L within the treated plot at the 1-day posttreatment sampling. The bottom sample residues showed an increase ranging from a mean 4.6 μg/L level at 7 days to an average of 10.8 μg/L at 14 days. This upward trend then reversed, with a decline to 1μg/L at 28- and 56-days after treatment. This upsurge of concentration seemed to occur in several of the BEE plots near the 7-day posttreatment sampling and could be related to herbicide release characteristics of the granular formulation. This is discussed further in the soil residue section of this report.

Sample sites 3, 9 and 12 demonstrated an outward diffusion pattern similar to the treated plot area, both at the surface and the bottom.

Statistical computation of surface water residue levels demonstrated a decay curve similar to plots 1 and 2 with residues declining to near zero at 14 days. This correlation was R^2 = 0.775. Bottom water samples were less well time correlated with residues of near 7 μg/L at 14 days and an R^2 = 0.429. Plot 3 was one of the shallower plots with depths ranging from 0.6- to 1.2-m (2- to 4-ft). This shallow depth provided less water in the treated column and, therefore, higher residue levels than those found in deeper water. Possible anomalies in the uniform decay curve might be expected as a result. However, the total residue level at any one time was not greatly different than that found in DMA-treated plots.

BANKS LAKE, plot 4, 22.5 kg/ha, ae, (20 lb/acre, ae), BEE (table 5, fig. 6).—Residue levels in this plot treated with 2,4-D BEE exhibited a somewhat less rapid decline in residue from 1 to 4 days after treatment than occurred with the 45 kg/ha rate in plot 3. However, the maximum concentration found 1-day posttreatment was 28 μg/L which was significantly less than the 137.5 μg/L found in the higher rate BEE plot. Seven-day samples showed a pronounced increase in residue levels, as was found in plot 3. The residue decreased rapidly to 3.7 μg/L at the 14-day sampling and continued to decline to 1 μg/L at 56 days. The bottom samples did not reflect a 7-day increase in residue, but declined steadily from a 1-day posttreatment mean level of 25.4 to 8.0 μg/L at 14 days followed by a 28- and 56-day level of 1 μg/L.

Sampling sites outside the treated plot demonstrated considerable variability from the residue dissipation pattern in the treated plot sites 1 and 2. Trends in upstream site 3 were similar to the treated plots, but downstream sample site 9 and offshore site 12 showed very low concentrations at 1 day, increasing at 7 days, followed by declines in surface water residues and increases in bottom water residues. These characteristics may reflect a concentration of this granular BEE formulation at the reservoir bottom followed by some outward dispersion from the treated area. All values found in DMA and BEE dissipation curves from Banks Lake are of a level that would not be expected to adversely affect nontarget organisms and fall well within established tolerances for 2,4-D in multiple-use waters.

The regression curves calculated for surface water show a less sharp drop from 1 through 7 days and more of a steady decay curve. Statistical curve fit for this regression was R^2 = 0.803, not greatly different than the Number 3 plot R^2 of 0.775. The bottom water statistical curve fit was similar in nature to the surface with an R^2 of 0.703.

FORT COBB, plot 1, 22.5 kg/ha, ae, (20 lb/acre, ae), BEE (table 6, fig. 7).—This ester formulation showed a more rapid decline in residues both at the surface and the bottom than did the similar treatment at Banks Lake. Peak concentrations of any one replication of surface water were 39.8 μg/L while the bottom was 64.8 μg/L. The mean combined values of sample sites 1 and 2 were 18.0 and 27.4 μg/L for the surface and bottom, respectively. These values declined to a level near 2.0 μg/L at 4 days' posttreatment, followed by a slight increase to approximately 5 μg/L at the 7-day posttreatment interval. This low residue level could, in part, be attributed to some herbicide absorption by a heavy eurasian watermilfoil infestation in this plot and not dispersion alone.

The outside sampling areas of sites 1:00, 3:00, and 5:00 were characterized by uniform dissipation loss with minor 7-day increases.

Table 4.—*Banks Lake, Washington, Plot 3[a], 2,4-D residue levels (mg/L) 1-, 4-, 7-, and 14-days, posttreatment.*

Site	Depth[d] m (ft)	Residue levels							
		1 day[e]		4 day		7 day		14 day	
[b] 1	.3, .3, .3, .3 (1, 1, 1, 1)	0.0431 0.0550 0.0560	(±0.0072)	0.0015 0.0026 0.0050	(±0.0018)	0.0057 0.0045 0.0060	(±0.0008)	0.0049 0.0026 0.0039	(±0.0011)
1	.6, 1.2, .6, .6 (2, 4, 2, 2)	0.0180 0.0176 0.0253	(±0.0043)	0.0204 0.2000 0.0181	(±0.0012)	0.0024 0.0036 0.0042	(±0.0010)	0.0103 0.0063 0.0088	(±0.0020)
[b] 2	.3, .3, .3, .3 (1, 1, 1, 1)	0.1375 0.1302 0.0398	(±0.0544)	0.0060 0.0052 0.0067	(±0.0007)	0.0059 0.0057 0.0064	(±0.0004)	0.0025 0.0015 0.0018	(±0.0005)
2	.6, .6 .6, 1 (2, 2, 2, 3)	0.0270 0.0232 –[f]	(±0.0027)	0.0068 0.0062 0.0052	(±0.0008)	– 0.0067 0.0040	(±0.0020)	0.0139 0.0119 0.0136	(±0.0011)
[c] 3	.3, .3, .3, .3 (1, 1, 1, 1)	0.1945 0.1704 0.1855	(±0.0122)	0.0060 0.0068 0.0068	(±0.0005)	0.0070 0.0056 0.0066	(±0.0007)	0.0017 0.0029 0.0029	(±0.0007)
3	2.4, 3, 1, 1.2 (8, 10, 3, 4)	0.0120 0.0102 0.0126	(±0.0012)	0.0047 0.0009 0.0036	(±0.0019)	0.0029 0.0030 0.0042	(±0.0007)	0.0027 0.0032 0.0039	(±0.0006)
[c] 9	.3, .3, .3, .3 (1, 1, 1, 1)	0.0283 0.0292 0.0313	(±0.0016)	0.0018 0.0027 0.0028	(±0.0005)	0.0031 0.0039 0.0033	(±0.0004)	0.0034 0.0027 0.0036	(±0.0005)
9	1.2, .6, 1.5, .6 (4, 2, 5, 2)	0.0010 0.0009 0.0001	(±0.0005)	0.0042 0.0017 0.0030	(±0.0012)	0.0026 0.0020 0.0021	(±0.0003)	0.0053 0.0070 0.0077	(±0.0012)
[c]12	.3, .3, .3, .3 (1, 1, 1, 1)	0.1092 0.1090 0.1055	(±0.0021)	0.0040 0.0054 0.0055	(±0.0008)	0.0046 0.0046 0.0046	(±0.0000)	0.0025 0.0019 0.0032	(±0.0006)
12	3, 3, 3, 3 (10, 10, 10, 10)	0.0003 0.0004 –	(±0.0001)	0.0005 0.0000 0.0001	(±0.0003)	0.0014 0.0013 0.0012	(±0.0001)	0.0011 0.0010 0.0016	(±0.0003)

[a] Treated with DMA at 45 kg/ha, ae (40 lb/acre, ae)
[b] Within treated plot sampling site.
[c] Outside of treated plot sampling site.
[d] The first depth was the depth from which the 1-day residual samples were taken; the second depth was for the 4-day residual samples; the third depth was the 7-day residual samples; and the fourth depth for the 14-day residual samples.
[e] Pretreatment (0 day) residue level 0.0000 mg/L.
[f] Hyphen indicates no sample was taken or the sample was lost during handling.

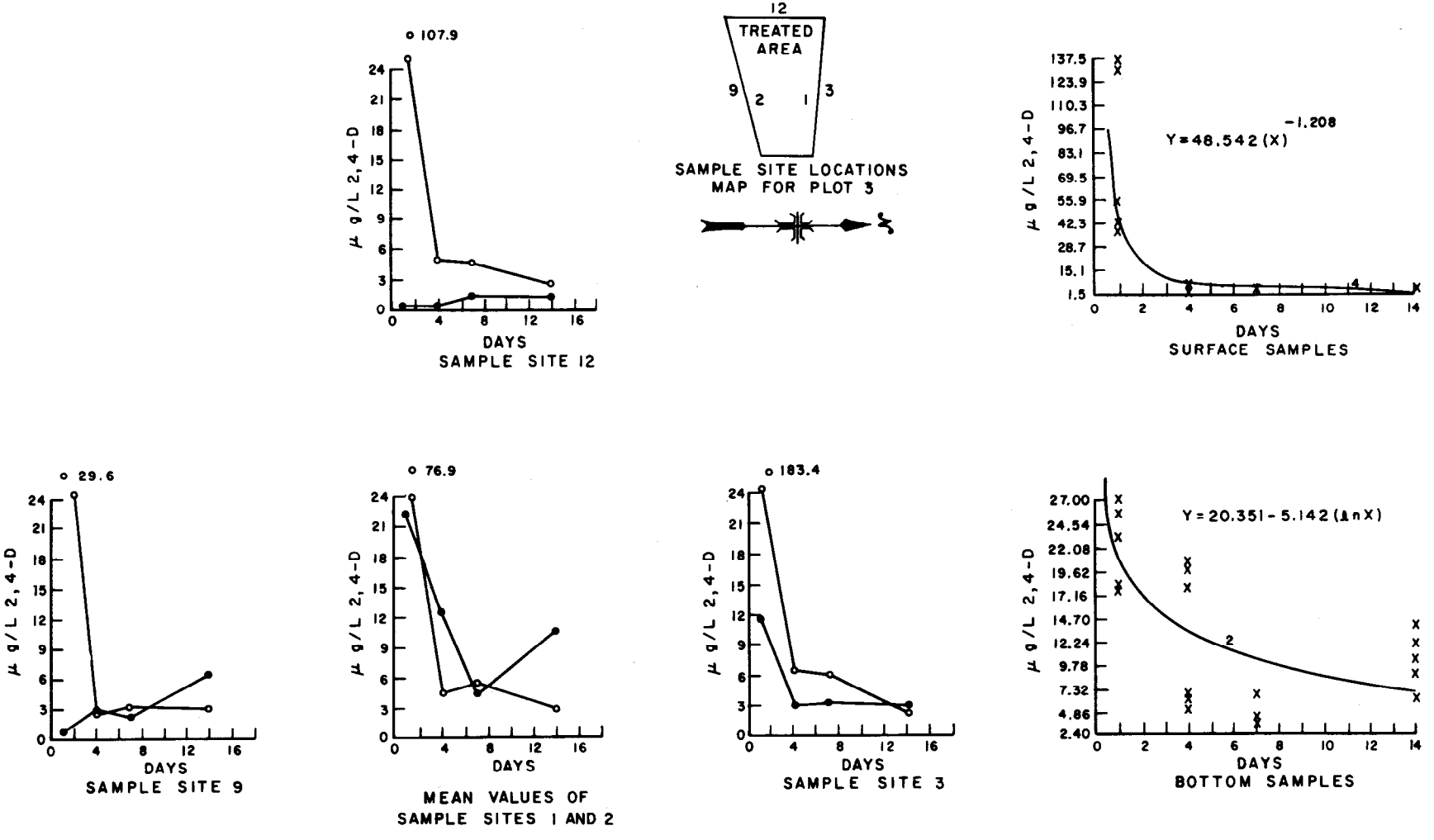


Figure 5.—Banks Lake 2,4-D dissipation in water, plot 3.

Dissipation of 45 kg/ha (40 lb/acre) BEE, in water over a 14-day period following treatment.
o = water sampled near the surface. • = water sampled near the bottom.

Statistical handling of the data in combined plots 1 and 2 produced a decay curve showing rapid decreases from 1 to 4 days' posttreatment declining to near zero by 14 days. The correlation R^2 for this regression was 0.579. This was not as good a fit to the curve as with some other treatments at Banks Lake due, in part, to the apparent 7-day rerelease of herbicide from the granular formulation.

FORT COBB, plot 2, 45 kg/ha, ae, (40 lb/acre, ae), BEE (table 7, fig. 8).—Comparison of replicates from sample sites 1 and 2 produced a mean value of 46.6 µg/L at the surface and 35.6 µg/L near the obttom. Seven-day posttreatment residues approached the zero level followed by an apparent herbicide rerelease with resultant increases to 10 and 5 µg/L at the surface and bottom, respectively. These herbicide concentrations did, however, again decline to levels of 3 and 1 µg/L at the surface andbottom by 28 days after treatment. Again, this formulation reflected the previously noted characteristic of a 7-day increase in residue level following a rapid decline 1 day following the application.

The regression of concentration plotted over the 14-day reporting period showed a less rapid decline over the 7-day period than was observed with the 22.5 kg/ha (20-lb/acre) rate in plot 1, but reached the zero point at 10-day posttreatment. The R^2 of the calculation was only 0.430, not a significant indicator for predictive purposes. As expected, residue levels outside the treated areas seem to follow the treated-plot pattern.

FORT COBB, plot 3, 45 kg/ha, ae, (40 lb/acre, ae), DMA (table 8, fig. 9).—The character of the herbicide dissipation curve observed with this DMA treatment at Fort Cobb is similar to that from Banks Lake, although maximum values differed at 1 day. At Banks Lake, the observed maximum was 90.8 µg/L at the surface and at Fort Cobb it was 164.9 µg/L at the surface. The 1-day bottom samples at Banks Lake were much different at 1 day with mean surface values of 24.7 µg/L while Fort Cobb bottom values were 176.2 µg/L. At Fort Cobb, both surface and bottom concentrations followed similar residue level decay patterns followed by decreases to near zero at 14 days after treatment. Banks Lake values did not reach the zero level but showed some residue remaining with approximately 2 µg/L at 56 days. The statistical calculation of combined sample sites 1 and 2 at Fort Cobb showed a steady uniform decrease in residue concentration to zero in approximately 11 days. The R^2 of the regression was 0.896 which is a very significant fit and suggests some predictive utility in routine field treatments. The observed fit of the data is quite similar to that of the 45-kg/ha (40-lb/acre) DMA treatment at Banks Lake where the R^2 was 0.932. These data suggest that the DMA treatments performed similarly with regard to dispersion, except that the treatment did not move downward in the water column as rapidly at Banks Lake as it did at Fort Cobb.

The sampling sites outside of the Fort Cobb plot 3 treated area followed a pattern very similar to the treated bay area, suggesting very uniform outward movment toward the main reservoir body. Peak values at 1 day from these outside sampling sites were lower than within the treated plots but, at 7 and 14 days, were not greatly different.

FORT COBB, plot 4, 22.5 kg/ha, ae, (20 lb/acre, ae), DMA (table 9, fig. 10).—The 1-day posttreatment samples collected contained very low residue levels with mean values in sites 1 and 2 all below 16 µg/L. The residue levels were very erratic in this treatment with an indication of more rapid setting of the herbicide in the treated water column than has been evident in other DMA-treated plots. The maximum single replicate residue level found was 8.7 µg/L in surface water and 7.1 µg/L in bottom water. The high variability found in plot 4 may have been due to a combination of factors: (1) the treatment was distributed over a 2-day period because of application equipment failure; and (2) this was a small plot of only 18.9 acres, quite shallow, and heavily infested with eurasian watermilfoil. The residue data through 7 days were erratic, but dissipation to low or near zero levels occurred by 14 days' posttreatment.

Statistical analysis of data from the treated plot indicates the lack of correlation between concentration and time after treatment, $R^2 = 0.242$. This computed decay curve, although similar to others, lacks confidence as a predictive tool, unlike many of the other treatments made in these studies. Fortunately, the overall levels of residue found were well below established 2,4-D tolerances or acceptable levels in potable water, irrigation water, and edible fish flesh.

Dissipation of 2,4-D residues outside the treated plots into sample sites 6:00, 8:00, and 10:00 showed a general trend of significant decline through 7 days. Some increases were noted from 7 to 14 days, but the levels were below 10 µg/L.

Table 5.—*Banks Lake, Washington, Plot 4[a], 2,4-D residue levels (mg/L) 1-, 4-, 7-, and 14-days, posttreatment.*

Site	Depth[d] m (ft)	Residue levels							
		1 day[e]		4 day		7 day		14 day	
[b] 1	.3, .3, .3, .3	0.0252		0.0100		0.0123		0.0030	
	(1, 1, 1, 1)	0.0230	(±0.0025)	0.0105	(±0.0004)	0.0195	(±0.0040)	0.0033	(±0.0002)
		0.0280		0.0108		0.0130		0.0030	
1	.6, .6, .6, .6	0.0284		0.0065		0.0124		0.0106	
	(2, 2, 2, 2)	0.0238	(±0.0024)	0.0086	(±0.0018)	0.0175	(±0.0028)	0.0100	(±0.0011)
		0.0250		0.0100		0.0131		0.0085	
[b] 2	.3, .3, .3, .3	0.0217		0.0100		0.0166		0.0042	
	(1, 1, 1, 1)	0.0214	(±0.0001)	0.0100	(±0.0000)	0.0149	(±0.0009)	0.0047	(±0.0004)
		0.0215		0.0099		0.0152		0.0040	
2	.6, .6, .6, .6	0.0234		0.0125		0.0038		0.0057	
	(2, 2, 2, 2)	0.0266	(±0.0023)	0.0112	(±0.0008)	0.0030	(±0.0022)	0.0064	(±0.0007)
		–		0.0110		0.0071		0.0072	
[c] 3	.3, .3, .3, .3	0.0126		0.0037		0.0064		0.0030	
	(1, 1, 1, 1)	0.0124	(±0.0010)	0.0049	(±0.0009)	0.0048	(±0.0008)	0.0027	(±0.0003)
		0.0107		0.0055		0.0054		0.0024	
3	.6, .6, 1.8, .6	0.0094		0.0045		0.0033		0.0066	
	(2, 2, 6, 2)	0.0120	(±0.0013)	0.0058	(±0.0013)	0.0032	(±0.0009)	0.0053	(±0.0008)
		0.0103		0.0071		0.0017		0.0052	
[c] 9	.3, .3, .3, .3	0.0063		0.0064		0.0161		0.0025	
	(1, 1, 1, 1)	0.0033	(±0.0015)	0.0073	(±0.0005)	0.0155	(±0.0052)	0.0028	(±0.0001)
		0.0042		0.0071		0.0068		0.0027	
9	1.5, 1, 1.2, 1.2	0.0008		0.0077		–		0.0074	
	(5, 3, 4, 4)	0.0001	(±0.0004)	0.0082	(±0.0003)	0.0049	(±0.0014)	0.0054	(±0.0014)
		0.0000		0.0083		0.0029		0.0082	
[c]12	.3, .3, .3, .3	0.0020		0.0027		0.0048		0.0044	
	(1, 1, 1, 1)	0.0036	(±0.0008)	0.0031	(±0.0003)	0.0040	(±0.0004)	0.0029	(±0.0008)
		0.0024		0.0024		0.0041		0.0030	
12	2.1, 1, 1.8, 1	0.0007		0.0020		0.0021		0.0051	
	(7, 3, 6, 3)	0.0000	(±0.0007)	0.0019	(±0.0003)	0.0020	(±0.0006)	0.0096	(±0.0023)
		0.0014		0.0014		0.0010		0.0080	

[a] Treated with BEE at 22.5 kg/ha, ae (20 lb/acre, ae)
[b] Within treated plot sampling site.
[c] Outside of treated plot sampling site.
[d] The first depth was the depth from which the 1-day residual samples were taken; the second depth was for the 4-day residual samples; the third depth was the 7-day residual samples; and the fourth depth for the 14-day residual samples.
[e] Pretreatment (0 day) residue level 0.000 mg/L.
[f] Hyphen indicates no sample was taken or the sample was lost during handling.

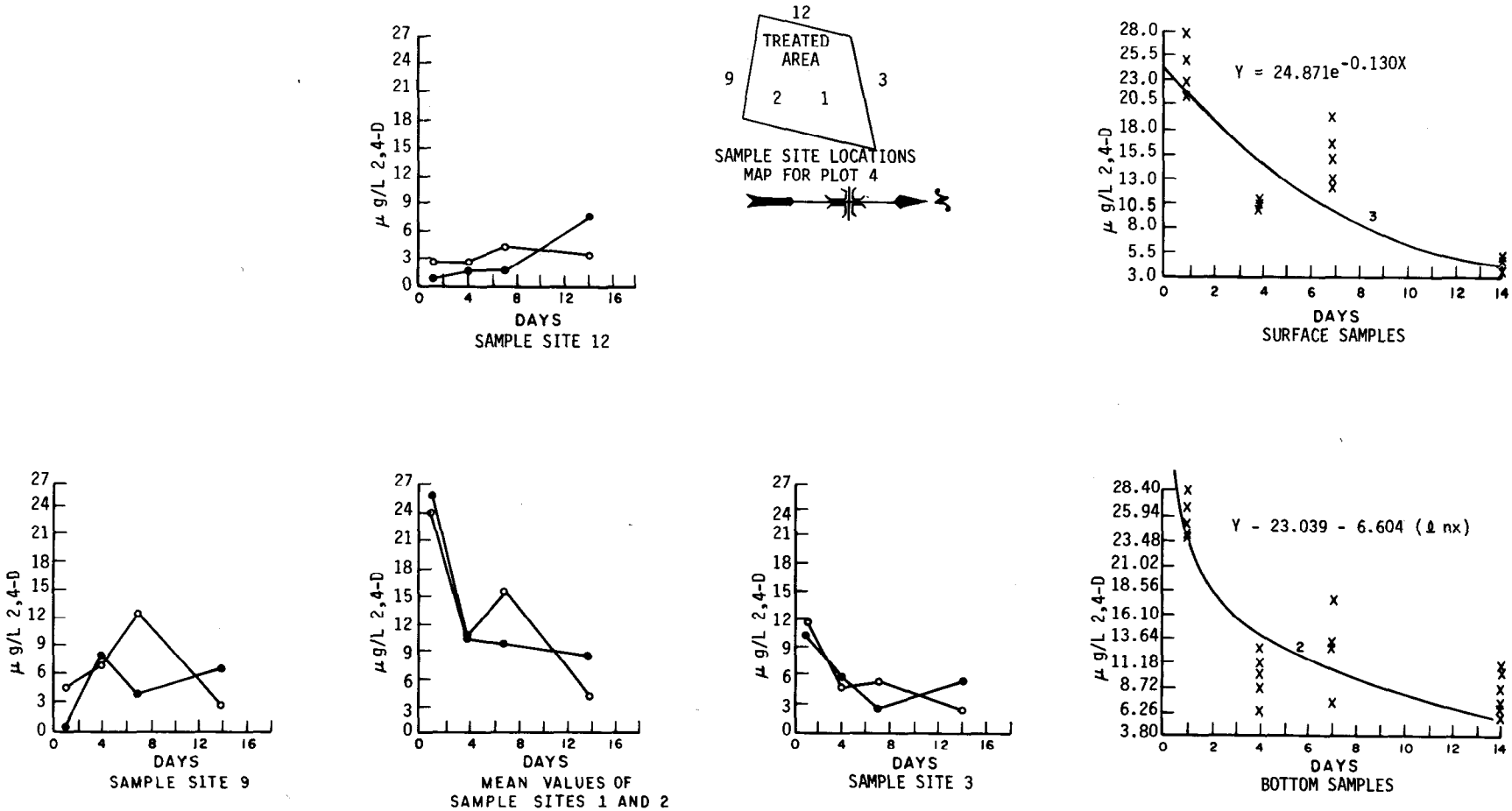


Figure 6.—Banks Lake 2,4-D dissipation in water, plot 4.

Dissipation of 22.5 kg/ha (20 lb/acre) BEE, in water over a 14-day period following treatment.
o = water sampled near the surface. • = water sampled near the bottom.

Table 6.—*Fort Cobb, Oklahoma, Plot 1[a], 2,4-D residue levels (mg/L) 1-, 4-, 7-, and 14-days, posttreatment.*

Site	Depth	Residue levels							
		1 day[e]		4 day		7 day		14 day	
[b] 1	Surface	0.0101 0.0141 0.0046 0.0079	(±0.0040)	0.0026 0.0015 0.0020	(±0.0005)	0.0163 0.0024 0.0099	(±0.0069)	0.0013 0.0003 0.0001	(±0.0006)
1	Bottom	0.0129 0.0318 0.0196	(±0.0096)	0.0007 0.0014 0.0020	(±0.0006)	0.0179 0.0038 0.0036	(±0.0082)	0.0003 0.0000 0.0001	(±0.0001)
[b] 2	Surface	0.0145 0.0275 0.0249 0.0398	(±0.0069)	0.0723 0.0008 0.0025	(±0.0408)	0.0002 0.0003 0.0001	(±0.0001)	0.0001 0.0001 0.0015	(±0.0008)
2	Bottom	0.0319 0.0645 0.0042	(±0.0302)	0.0014 0.0026 0.0042	(±0.0014)	0.0015 0.0027 0.0006	(±0.0010)	0.0000 0.0001 0.0002	(±0.0001)
[c] 1:00	Surface	0.0344 0.0826 0.0125 0.0313	(±0.0298)	0.0009 0.0009 -[d]	(±0.0000)	0.0034 0.0000 0.0001	(±0.0019)	0.0000 0.0004 0.0001	(±0.0002)
1:00	Bottom	0.0091 0.0160 0.0067 0.0117	(±0.0035)	0.0008 0.0000 0.0020	(±0.0010)	0.0000 0.0001 0.0000	(±0.0000)	0.0000 0.0000 0.0000	-
[c] 3:00	Surface	0.0063 0.0063 0.0290	(0.0131)	-[d] 0.0012 0.0028	(±0.0011)	0.0000 0.0031 0.0087	(±0.0044)	0.0000 0.0000 0.0000	-
3:00	Bottom	0.0072 0.0123 0.0144 0.0219	(±0.0061)	0.0021 0.0115 0.0004	(±0.0060)	0.0049 0.0046 0.0000	(±0.0027)	0.0000 0.0000 0.0000	-
[c] 5:00	Surface	0.0234 0.0139 0.0744 0.0779	(±0.0334)	0.0186 0.0008 0.0073	(±0.0090)	0.0001 0.0090 0.0004	(±0.0050)	0.0001 0.0001 0.0012	(±0.0006)
5:00	Bottom	0.0345 0.0268 0.0330	(±0.0041)	0.0001 -[d] 0.0005	(±0.0003)	0.0002 0.0000 0.0000	(±0.0001)	0.0002 0.0001 0.0001	-

[a] Treated with BEE at 22.5 kg/ha, ae (20 lb/acre, ae)
[b] Within treated plot sampling site.
[c] Outside of treated plot sampling site.
[d] Hyphen indicates no sample was taken or the sample was lost during handling.
[e] Pretreatment (0 day) residue level 0.0000 mg/L.

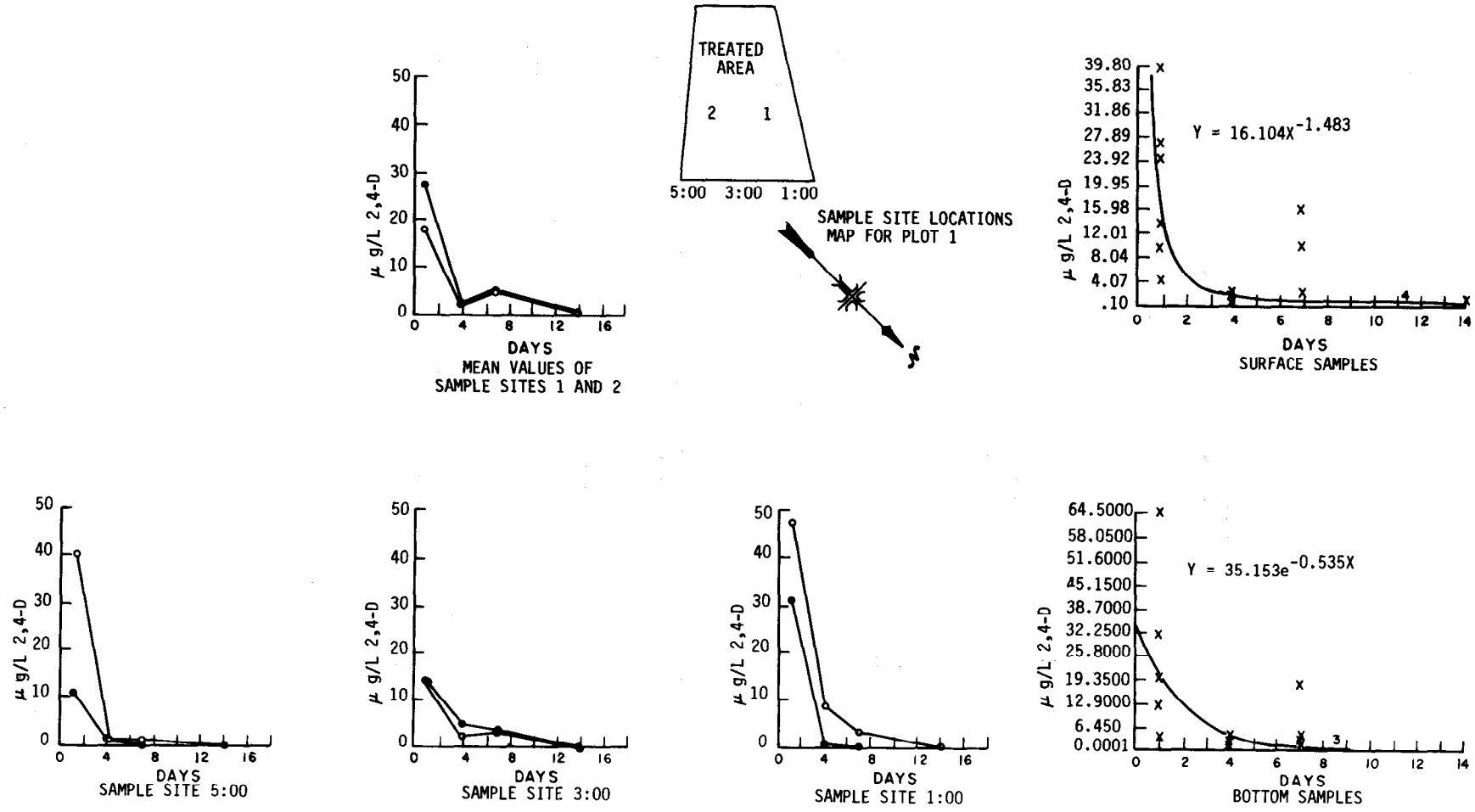


Figure 7 .—Fort Cobb 2,4-D dissipation in water, plot 1.

Dissipation of 22.5 kg/ha (20 lb/acre) BEE, in water over a 14-day period following treatment.
o = water sampled near the surface. • = water sampled near the bottom.

Table 7.—*Fort Cobb, Oklahoma, Plot 2[a], 2,4-D residue levels (mg/L) 1-, 4-, 7-, and 14-days, posttreatment.*

Site	Depth	Residue levels							
		1 day[e]		4 day		7 day		14 day	
[b] 1	Surface	0.0350		0.0005		0.0000		0.0264	
		0.0332	(±0.0011)	0.0009	(±0.0003)	0.0000	-	0.0211	(±0.0067)
		0.0351		0.0012		0.0000		0.0131	
1	Bottom	-[d]		0.0004		0.0000		0.0118	
		0.0074	(±0.0039)	0.0005	(±0.0003)	0.0000	-	0.0124	(±0.0043)
		0.0129		0.0000		0.0000		0.0047	
[b] 2	Surface	0.0552		0.0004		0.0000		0.0000	
		0.0585	(±0.0036)	0.0025	(±0.0012)	0.0000	-	0.0000	-
		0.0624		0.0005		0.0000		0.0000	
2	Bottom	0.0568		0.0004		0.0013		0.0000	
		0.0607	(±0.0044)	0.0008	(±0.0003)	0.0000	(±0.0007)	0.0000	-
		0.0656		0.0009		0.0000		0.0000	
[c] 1:00	Surface	0.0067		0.0011		0.0010		0.0000	
		0.0153	(±0.0088)	0.0009	(±0.0004)	0.0000	(±0.0006)	0.0000	-
		0.0243		0.0016		0.0010		0.0000	
1:00	Bottom	0.0080		0.0013		0.0233		0.0000	
		0.0136	(±0.0029)	0.0010	(±0.0003)	0.0077	(±0.0118)	0.0000	-
		0.0097		0.0006		0.0001		0.0000	
[c] 3:00	Surface	0.0545		0.0011		0.0006		0.0000	
		0.0606	(±0.0032)	0.0011	(±0.0005)	0.0005	(±0.0019)	0.0011	(±0.0008)
		0.0591		0.0020		0.0039		-	
3:00	Bottom	0.0231		0.0000		-		0.0032	
		0.0351	(±0.0125)	0.0003	(±0.0002)	0.0000	(±0.0012)	0.0000	(±0.0017)
		0.0100		0.0003		0.0017		0.0028	
[c] 5:00	Surface	0.0460		0.0007		0.0032		0.0000	
		0.0602	(±0.0150)	0.0018	(±0.0006)	0.0033	(±0.0002)	0.0000	-
		0.0301		0.0017		0.0029		-	
5:00	Bottom	0.0274		0.0008		0.0036		-	
		0.0575	(±0.0167)	0.0000	(±0.0004)	0.0021	(±0.0011)	0.0000	-
		0.0297		0.0001		-		0.0000	

[a] Treated with BEE at 45 kg/ha, ae (40 lb/acre, ae)
[b] Within treated plot sampling site.
[c] Outside of treated plot sampling site.
[d] Hyphen indicates no sample was taken or the sample was lost during handling.
[e] Pretreatment (0 day) residue level 0.0000 mg/L.

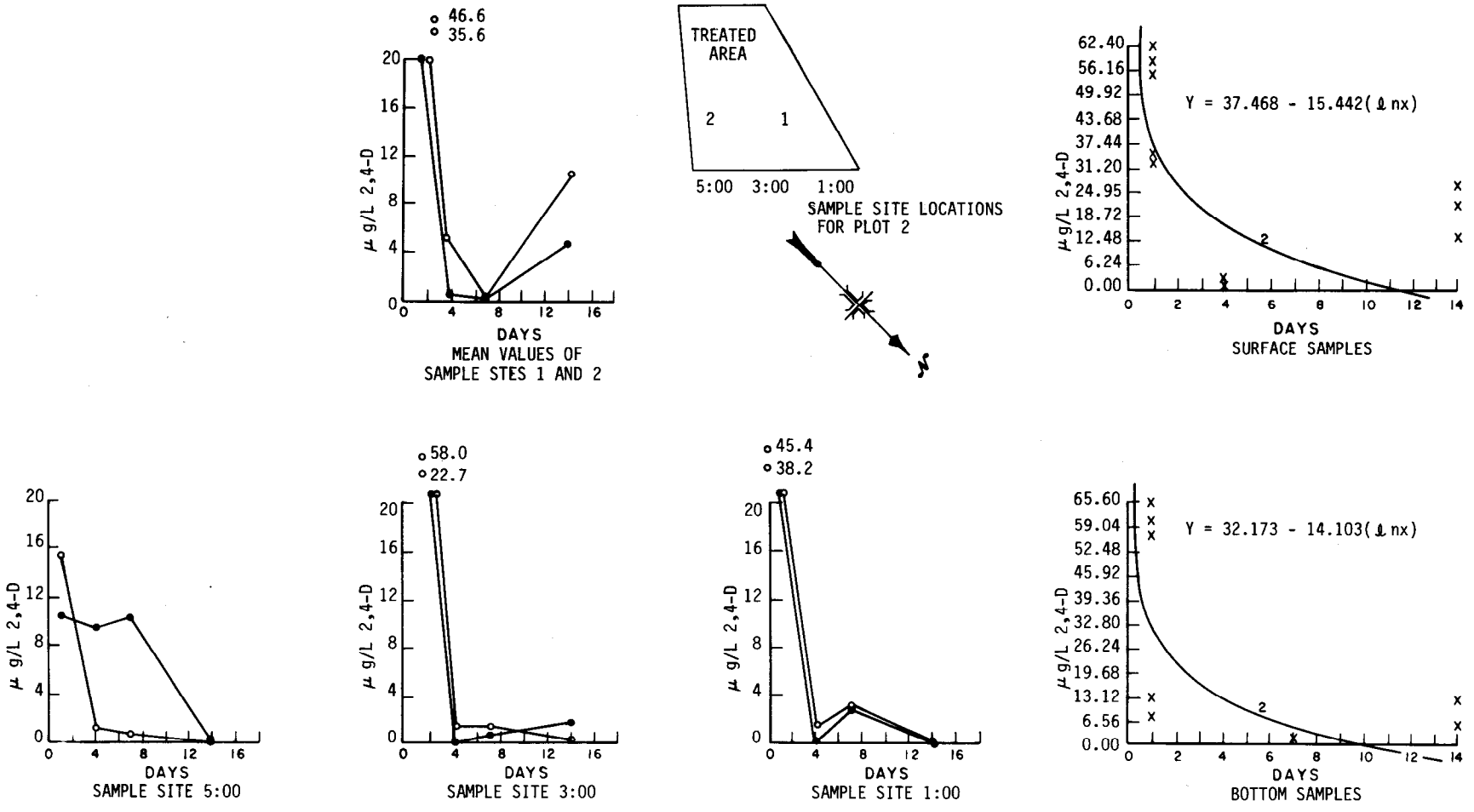


Figure 8.—Fort Cobb 2,4-D dissipation in water, plot 2.

Dissipation of 45 kg/ha (40 lb/acre) BEE, in water over a 14-day period following treatment.
o = water sampled near the surface. • = water sampled near the bottom.

Table 8.—*Fort Cobb, Oklahoma, Plot 3[a], 2,4-D residue levels (mg/L) 1-, 4-, 7-, and 14-days, posttreatment.*

Site	Depth	Residue levels							
		1 day[e]		4 day		7 day		14 day	
[b] 1	Surface	0.1438 0.1437 0.1114	(±0.0187)	0.0882 0.0427 0.0752	(±0.0234)	0.0225 0.0160 0.0183	(±0.0033)	0.0015 0.0002 0.0008	(±0.0006)
1	Bottom	0.1453 0.1916 0.1896	(±0.0262)	0.0447 0.0474 0.0536	(±0.0046)	0.0370 0.0292 0.0248	(±0.0062)	0.0012 0.0012 0.0000	(±0.0006)
[b] 2	Surface	0.1945 0.1894 0.2069	(±0.0090)	0.0420 0.0472 0.0181	(±0.0155)	0.0523 0.0317 0.0285	(±0.0129)	0.0005 0.0002 0.0009	(±0.0003)
2	Bottom	0.1453 0.1674 0.2179	(±0.0372)	0.0451 0.0335 0.0336	(±0.0066)	0.0175 0.0111 0.0177	(±0.0037)	0.0009 0.0000 0.0004	(±0.0004)
[c] 10:00	Surface	0.1201 0.0699[d] 0.0357 0.1038	(±0.0375)	0.0201 0.0226 0.0200	(±0.0015)	0.0160 0.0172 0.0122	(±0.0026)	0.0043 0.0020 0.0022	(±0.0013)
10:00	Bottom	0.0994 0.1240 0.1399	(±0.0204)	0.0279 0.0299 0.0272	(±0.0014)	0.0083 0.0067 0.0141	(±0.0039)	0.0007 0.0011 0.0014	(±0.0003)
[c] 8:00	Surface	0.1139 0.0501[d] 0.0463 0.0372	(±0.0351)	0.0175 0.0452 0.0106	(±0.0183)	0.0132 –[f] 0.0132	-	0.0008 0.0020 0.0014	(±0.0006)
8:00	Bottom	0.0422 0.1231 0.0869	(±0.0405)	0.0308 0.0224 0.0286	(±0.0043)	0.0117 0.0132 0.0113	(±0.0010)	0.0006 0.0010 0.0012	(±0.0003)
[c] 6:00	Surface	0.0712[d] 0.0484 0.1118 0.0655	(±0.0269)	0.0330 0.0179 0.0306	(±0.0081)	0.0199 0.0146 0.0117	(±0.0041)	0.0009 0.0029 0.0019	(±0.0010)
6:00	Bottom	0.0783 0.0950 0.0549 0.0722[d]	(±0.0165)	0.0246 0.0247 0.0311	(±0.0037)	0.0124 0.0153 0.0100	(±0.0026)	0.0017 0.0005 0.0006	(±0.0007)

[a] Treated with DMA at 45 kg/ha, ae (40 lb/acre, ae)
[b] Within treated plot sampling site.
[c] Outside of treated plot sampling site.
[d] Sample reanalyzed to confirm
[e] Pretreatment (0 day) residue level 0.0000 mg/L.
[f] Hyphen indicates no sample was taken or the sample was lost during handling.

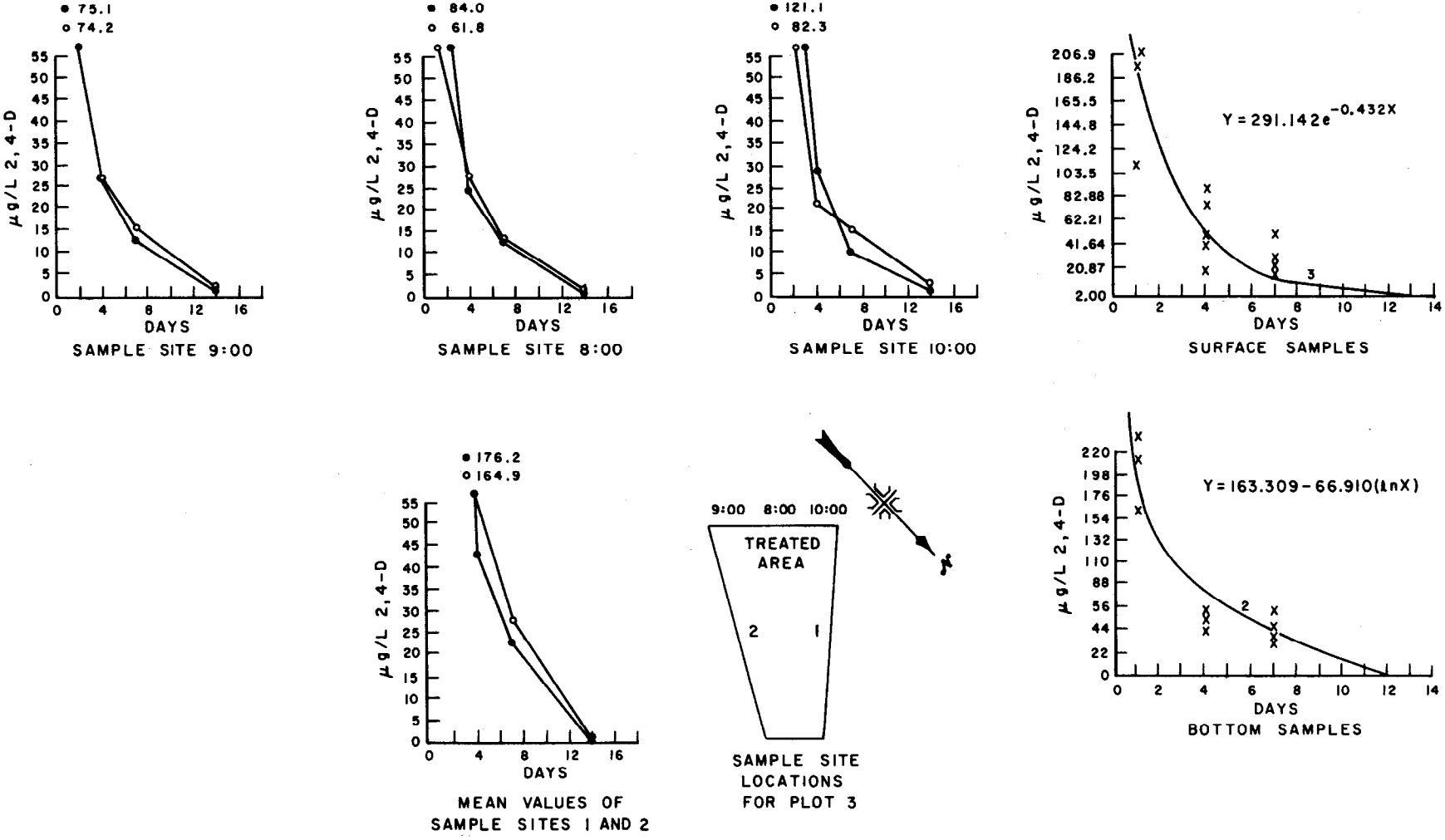


Figure 9.—Fort Cobb 2,4-D dissipation in water, plot 3.

Dissipation of 45 kg/ha (40 lb/acre) DMA, in water over a 14-day period following treatment.
○ = water sampled near the surface. • = water sampled near the bottom.

Table 9.—*Fort Cobb, Oklahoma, Plot 4[a], 2,4-D residue levels (mg/L) 1-, 4-, 7-, and 14-days, posttreatment.*

Site	Depth	Residue levels							
		1 day[e]		4 day		7 day		14 day	
[b] 1	Surface	0.0064 0.0038 0.0054	(±0.0013)	0.0117 0.0123 0.0041	(±0.0044)	0.0060 0.0112 0.0072	(±0.0027)	0.0002 0.0000 0.0027	(±0.0015)
1	Bottom	0.0048 0.0071[d] 0.0235 0.0348	(±0.0142)	0.0045 0.0137 0.0057	(±0.0050)	0.0000 0.0010 0.0000	(±0.0006)	0.0004 0.0000 0.0007	(±0.0003)
[b] 2	Surface	0.0087 0.0067 0.0022	(±0.0033)	0.0227 0.0168 0.0091	(±0.0068)	0.0167 –[f] 0.0228	(±0.0043)	0.0033 0.0039 0.0011	(±0.0015)
2	Bottom	0.0180 0.0015 0.0040[d] 0.0060 0.0053[d]	(±0.0064)	0.0052 0.0030 0.0047	(±0.0011)	0.0018 0.0067 0.0003	(±0.0033)	0.0001 0.0008 0.0000	(±0.0004)
[c] 10:00	Surface	0.0126 0.0067 0.0141	(±0.0039)	0.0026 0.0050 0.0037	(±0.0012)	0.0035 0.0067 0.0047	(±0.0016)	0.0015 0.0101 0.0064	(±0.0043)
10:00	Bottom	0.0059 0.0007 0.0033	(±0.0026)	0.0031 0.0068 0.0070	(±0.0022)	0.0005 0.0019 0.0000	(±0.0010)	0.0102 0.0077 0.0036	(±0.0033)
[c] 8:00	Surface	0.0502 0.0372 0.0078	(±0.0217)	0.0037 0.0025 0.0046	(±0.0010)	0.0065 0.0071 0.0033	(±0.0020)	0.0036 0.0029 0.0041	(±0.0006)
8:00	Bottom	0.0055[d] 0.0030 0.0122 0.0103	(±0.0047)	0.0055 0.0104 0.0087	(±0.0025)	0.0018 0.0016 0.0001	(±0.0010)	0.0024 0.0021 0.0000	(±0.0013)
[c] 6:00	Surface	0.0195 0.0164 0.0107 0.0122[d]	(±0.0040)	0.0039 0.0027 0.0035	(±0.0006)	0.0091 0.0097 0.0062	(±0.0016)	0.0006 0.0017 0.0024	(±0.0010)
6:00	Bottom	0.0048 0.0050 0.0035	(±0.0008)	0.0096 0.0061 –[f]	(±0.0025)	0.0016 0.0012 0.0004	(±0.0006)	0.0009 0.0010 0.0040	(±0.0018)

[a] Treated with DMA at 22.5 kg/ha, ae (20 lb/acre, ae)
[b] Within treated plot sampling site.
[c] Outside of treated plot sampling site.
[d] Sample reanalyzed to confirm.
[e] Pretreatment (0 day) residue level 0.0000 mg/L.
[f] Hyphen indicates no sample was taken or the sample was lost during handling.

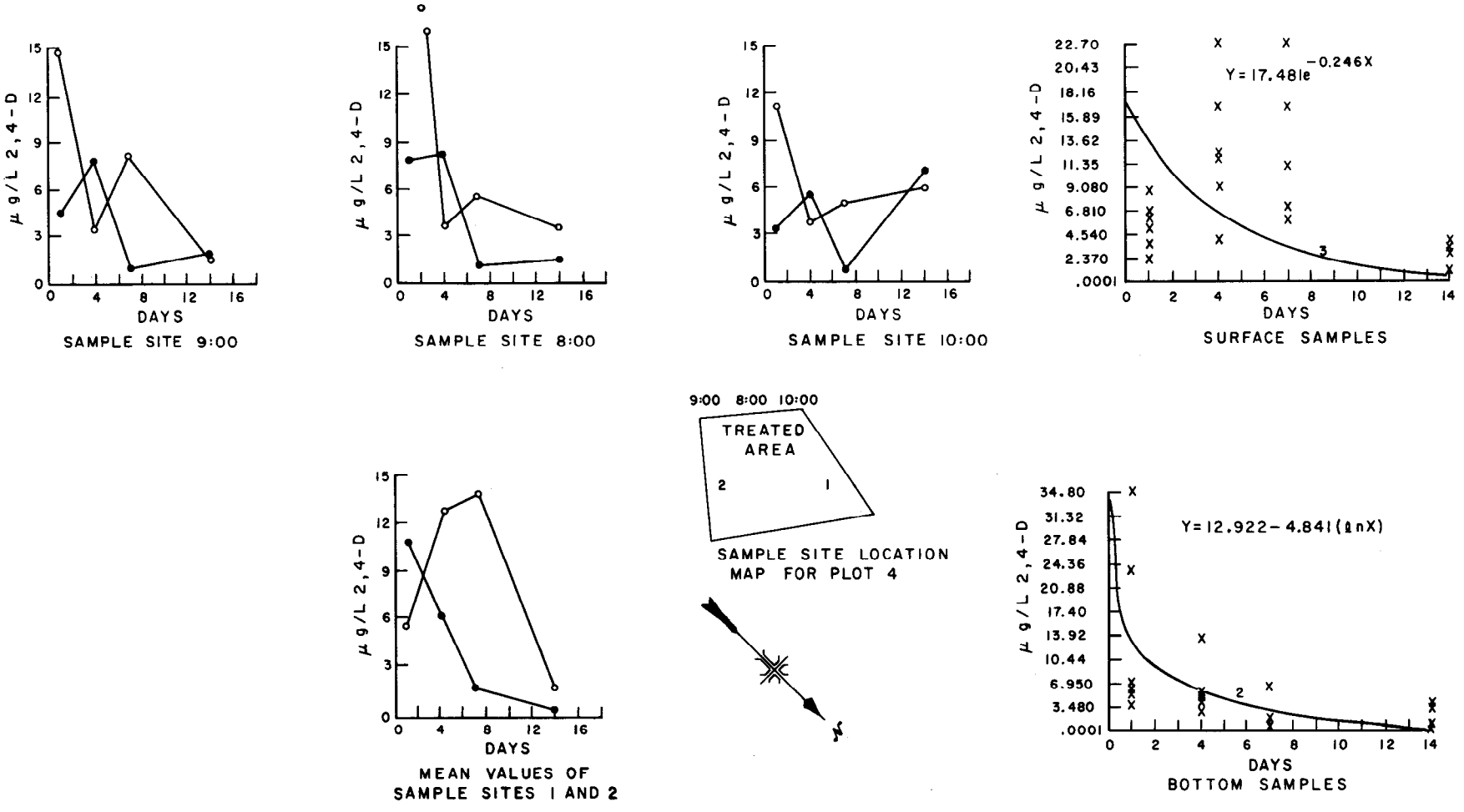


Figure 10.—Fort Cobb 2,4-D dissipation in water, plot 4.

Dissipation of 22.5 kg/ha (20 lb/acre) DMA, in water over a 14-day period following treatment.
o = water sampled near the surface. • = water sampled near the bottom.

Herbicide Residues at Reservoir Outlets

The 2,4-D residue data in table 10 detail one of the most important aspects of this study. This table lists the residue levels found at the reservoir outlet works delivering water for multiple uses in these two geographical locations.

The distribution of the three replications for sampling dates 1 through 14 days was very close. The highest outlet level found at Banks Lake was 7.6 μg/L, a level which is not considered hazardous for any proposed irrigation or potable water use. This is well below the established experimental tolerance for experimental permits granted for this study [6] as well as established tolerances for 2,4-D in the Tennessee Valley River system for control of eurasian watermilfoil [7] and for use of DMA in control of ditchbank weeds in Western United States irrigation systems [8] [9].

The two outlets at Fort Cobb, Chickasha and Anadarko municipal and industrial use, were similar to those found at Banks Lake. The maximum value found there was 14.9 μg/L at the Chickasha outlet 4 days' posttreatment.

The State of Oklahoma Department of Health also monitored the potable water from Fort Cobb discharges in cooperation with the Bureau of Reclamation and the Fort Cobb Master Water Conservancy District. This was done to ensure safe chemical concentrations in this water during the study period. These data, given in table 11, are included to provide additional confirmation of the residue levels at the study site. One value of 62 μg/L was found, but this was suspected of being a contaminated sample. Most of the other values fell well within the ranges found by our investigations.

The Oklahoma State Department of Health data support the hypothesis that residue levels from these experimental treatments did not approach established experimental tolerance for 2,4-D and safety for multiple use of these treated waters was demonstrated.

Results of these studies demonstrate that when DMA or BEE formulations are applied no closer than 0.75 km (one-half mile) from a reservoir or lake outlet, the residue levels would not be hazardous for multiple water use. This distance from an outlet works appears to provide a safety factor for sufficient dissipation of the herbicide through a combination of dilution, absorption, hydrolysis, or other decomposition and time for these factors to function. The authors hypothesize that in smaller water bodies where these distances from outlets works cannot be attained, subdividing the reservoir areas into a number of sections (i.e. 4 to 8) and waiting at least 4 days between herbicide treatments would provide sufficient opportunity for dissipation of the herbicide to levels acceptable for multiple use.

2,4-D Residues in Hydrosoil

Determinations of 2,4-D residues in hydrosoil were completed through 56 days' posttreatment. Residue data resulting from analysis of hydrosoil samples are presented in tables 12 and 13 and figures 11 through 18. Hydrosoil samples were collected only within the treated plot at sampling sites 1 and 2. For discussion purposes, within plot residues determined from these two sites were averaged. However, in figures 11 through 18, they were graphed separately.

Pretreatment hydrosoil samples collected from Banks Lake indicated no detectable 2,4-D residue. However, as was observed with pretreatment water samples from Fort Cobb Reservoir, trace 2,4-D residues were detected in some Fort Cobb Reservoir pretreatment hydrosoil samples. A possible explanation is the proximity of Fort Cobb Reservoir to areas of intensive agricultural activity with the related possibility for surface drainage contamination.

As with the water residue discussion, herbicide formulations and rates of application are discussed individually in chronological order of plot number and geographic location.

BANKS LAKE, plot 1, 22.5 kg/ha, ae (20 lb/acre, ae), DMA (table 12, fig. 11).—The highest average residue level detected in plot 1 was 0.0709 μg/g at 14 days' posttreatment. Residues increased from 1 to 4 days' posttreatment followed by a decline to 7 days (fig. 11). A residue increase from 7 to 14 days occurred, followed by another decline to 56 days' posttreatment. Residue values were less than 0.10 μg/L throughout the entire sampling period. The low hydrosoil residues provided further confirmation of the uniform dispersion throughout the water column.

BANKS LAKE, plot 2, 45 kg/ha, ae, (40 lb/acre, ae), DMA (table 12, fig. 12).—The maximum observed residue in plot 2 was 0.1723 μg/g at 28 days' posttreatment. A residue increase from 1 to 4 days occurred, followed by a decline to 7 days' posttreatment. An increase in 2,4-D residue levels was observed during the 7- to 28-day

Table 10.—*Residue Levels at Outlets, Banks Lake, Washington, and Fort Cobb Reservoir, Oklahoma.*

Site	Depth	Residue levels (mg/L) 1 day		4 day		7 day		14 day	
Banks Lake									
—	4 m	0.0000		0.0062		0.0076		0.0028	
		0.0003	(±0.0005)	0.0076	(±0.0008)	0.0054	(±0.0015)	0.0026	(±0.0007)
		0.0011		0.0062		0.0047		0.0039	
7-8-81 pretreatment		0.0000	–						
Fort Cobb Reservoir									
Chickasha									
outlet	Mid	0.0000		0.0099		0.0000			
		0.0002	(±0.0001)	0.0124	(±0.0025)	0.0000	–		
		–[a]		0.0149					
Anardarko									
outlet	Mid	–		0.0084		–		–	
		–		0.0073	(±0.0043)	–		–	
		–		0.0004		–		–	
8-18-81 pretreatment		0.0030	–						

[a] Hyphen indicates no samples were taken.
All values in ppm (mg/L).

Table 11.—*2,4-D Residue Levels at Fort Cobb Reservoir, August 1981*[1].

Date	Fort Cobb Midpoint	Anardarko Intake	Anardarko Finished	Chickasha Intake	Chickasha Finished	Comments
18 Aug 81	* 0.500	0.630	* 0.500	* 0.500	* 0.500	Prior to application
19 Aug 81	1.690	* 0.500	* 0.500	* 0.500	* 0.500	
20 Aug 81	* 0.500	4.200	* 0.500	62.000	1.000	First application
21 Aug 81	1.600	11.500	* 0.500	1.430	0.580	
22 Aug 81	1.000	1.300	* 0.500	1.000	0.530	
23 Aug 81	0.680	1.300	* 0.500	* 0.500	1.970	
25 Aug 81	2.600	7.000	4.000	11.300	1.030	Second application
26 Aug 81	7.900	3.700	1.830	5.000	1.000	
27 Aug 81	1.860	4.630	4.340	3.380	1.460	
28 Aug 81	* 0.500	1.630	1.250	1.050	0.750	
31 Aug 81	0.600	* 0.500	* 0.500	1.190	* 0.500	
2 Sept 81	* 0.500	* 0.500	* 0.500	* 0.500	* 0.500	

All values in ppb(μg/L); 2,4-D Standard = ppb.
* = less than detection limit of analysis.
[1] Data on this table were produced by the Oklahoma State Department of Health, 1000 Northeast 10th Street, PO Box 5351, Oklahoma City, Oklahoma 73152

interval followed by a decline to 56 days' posttreatment. The hydrosoil dissipation pattern in plot 2 was very similar to that observed in plot 1 with the exception of the slight residue increase of the 14- and 28-day sampling intervals. Average within-plot residue levels were less than 0.20 μg/g for the entire 56-day sampling period. Again, low residue levels are probably attributable to uniform dispersion throughout the water column.

BANKS LAKE, plot 3, 45 kg/ha, ae, (40 lb/acre, ae), BEE (table 12, fig. 13).—Plot 3 at Banks Lake showed a maximum hydrosoil residue level of 25.526 μg/g at the 7-day posttreatment sampling. Residue levels decreased from 1 to 4 days but increased substantially from 4 to 7 days' posttreatment (fig. 13). From this 7-day peak, levels decreased through 56 days. The 7-day posttreatment peak is of particular interest in view of the fact that a similar occurrence was noted in water samples from BEE formulation treated plots. It is most likely attributable to a rerelease from the herbicide pellet.

BANKS LAKE, plot 4, 22.5 kg/ha, ae, (20 lb/acre, ae) BEE (table 12, fig. 14).—Banks Lake, plot 4 was treated with BEE. The highest observed hydrosoil residue levels occurred at 7 days' posttreatment (fig. 14) where a within-treated plot average of 11.224 μg/g was noted. Residue levels in the hydrosoil increased from 1 to 7 days posttreatment followed by a gradual decline to 56 days' posttreatment. The overall observed hydrosoil dissipation rates were similar to those observed in plot 3 with indication of the same 7-day additional release of herbicide from the granular formulation.

FORT COBB RESERVOIR, plot 1, 22.5 kg/ha, ae, (20 lb/acre, ae), BEE (table 13, fig. 15).—Plot 1 at Fort Cobb Reservoir, Oklahoma, was treated with the granular 2,4-D BEE formulation and the maximum hydrosoil residue levels occurred at 4 days' posttreatment when a within-plot average of 1.433 μg/g was noted (fig. 15). The overall dissipation pattern was similar to that observed with plot 4 at Banks Lake except that the rerelease peak occurred at 4 days' posttreatment rather than 7 days. Overall, hydrosoil residue levels were lower at Fort Cobb Reservoir than at Banks Lake for the entire 56-day sampling period. Numerous factors might have contributed to this observed contrast, but a heavier eurasian watermilfoil infestation with accompanying absorption of 2,4-D by the plant material is probably one of the most significant ones, as discussed by Gangstad in the article on dissipation of 2,4-D in large reservoirs [10]. Also, water temperatures at Fort Cobb were higher, as were conductivity values. The water quality parameters, both physical and chemical, are discussed in the Environmental Effects of Herbicide Treatment section.

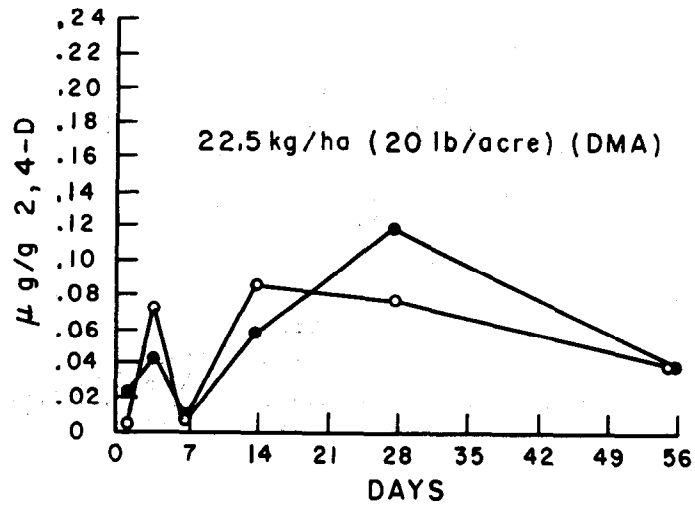
FORT COBB RESERVOIR, plot 2, 45 kg/ha, ae, (40 lb/acre, ae), BEE (table 13, fig. 16).—The maximum 2,4-D hydrosoil residue level detected in Fort Cobb, plot 2 was 3.942 μg/g at 1-day posttreatment. Residues decreased from 1- to 4-day posttreatment, then increased from 4 to 14 days. Residue levels decined from 14 to 56 days. Comparison of figures 13 and 16 reveals that the residue dissipation trend was similar to that of Banks Lake, plot 3, except that Fort Cobb residue levels were lower. This lower residue level is attributable at least in part to the heavier infestations of eurasian watermilfoil at Fort Cobb. The apparent rerelease from the granular BEE formulation observed at Banks Lake also occurred at Fort Cobb between 4- and 14-days' posttreatment.

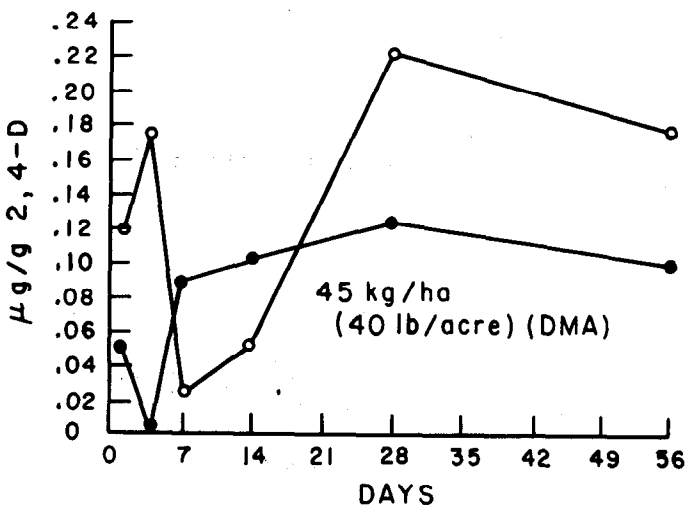
FORT COBB RESERVOIR, plot 3, 45 kg/ha, ae, (40 lb/acre, ae), DMA (table 13, fig. 17).—Plot 3 at Fort Cobb Reservoir had a maximum average hydrosoil residue of 0.172 μg/g at 7-days' posttreatment. Residues increased from 1- to 7-days' posttreatment, followed by a decrease through 56 days (fig. 17). This prolonged increase in residues through 7-days posttreatment could be attributable to an initial uptake of 2,4-D by aquatic plant populations followed by a subsequent release of adsorbed herbicide upon onset of vegetation decomposition. Throughout the 56-day sampling period, residue values were less than 0.20 μg/g. Comparison of figures 11 and 12, Banks Lake, plots 1 and 2, with figure 17, shows that the same uniform dispersion throughout the water column was in evidence at both geographic locations.

FORT COBB RESERVOIR, plot 4, 22.5 kg/ha, ae, (20 lb/acre, ae), DMA (table 13, fig. 18).—The highest average residue level found in hydrosoil from within plot 4 was 0.133 μg/g at 7 days' posttreatment. Residues increased from 1 to 7 days' posttreatment, at which time a decline from 7 to 56 days' posttreatment occurred. Hydrosoil 2,4-D residues were less than 0.15 μg/g throughout the 56-day sampling period. The 2,4-D water residues were quite variable in Fort Cobb, plot 4, while the hydrosoil residue for plots 3 and 4 (figs. 17 and 18) show a similar pattern of a 7-day residue peak, followed by a sharp decline from 7 to 14 days' posttreatment with a more gradual decline to 56 days.

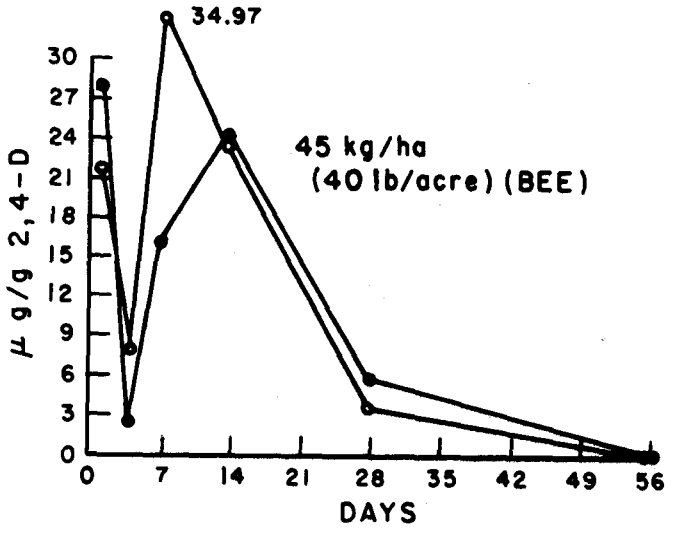
Table 12.—*Banks Lake 2,4-D hydrosoil residues.*

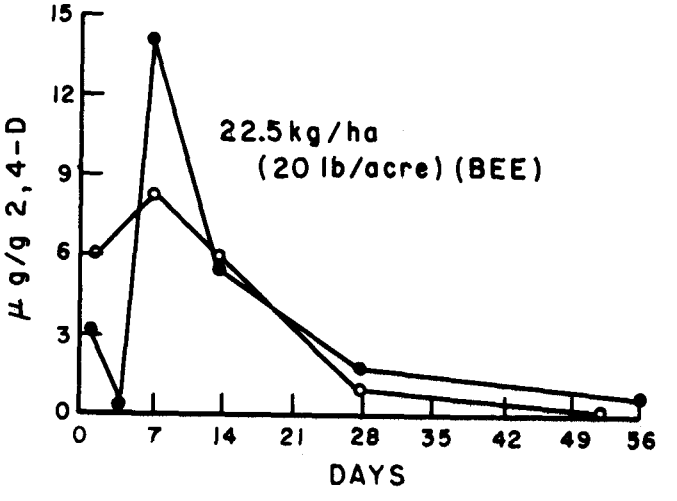
Posttreatment sampling day	Site No.	Rep. No.	Plot 1 22.5 kg/ha (20 lb/acre) DMA (μg/g)	Plot 2 45 kg/ha (40 lb/acre) DMA (μg/g)	Plot 3 45 kg/ha (40 lb/acre) BEE (μg/g)	Plot 4 22.5 kg/ha (20 lb/acre) BEE (μg/g)
1 day	1	1	0.0078	0.0000	31.5334	5.6603
	1	2	0.0000	0.2318	24.7367	6.3544
	2	1	0.0000	0.0468	23.3904	0.9366
	2	2	0.0424	0.0548	19.3058	5.4292
4 days	1	1	0.0000	0.1654	–	7.4653
	1	2	0.1428	0.1846	5.2934	5.9574
	2	1	0.0000	0.0000	6.0405	0.3248
	2	2	0.0831	0.0000	9.7605	–
7 days	1	1	0.0000	0.0000	20.3921	8.8453
	1	2	0.0000	0.0463	11.7604	7.8418
	2	1	0.0146	0.1038	32.8690	16.4253
	2	2	0.0000	0.0739	37.0826	11.7851
14 days	1	1	0.0744	0.0686	19.1702	5.4735
	1	2	0.0961	0.0351	29.4324	6.3728
	2	1	0.0325	0.1376	10.4282	3.6608
	2	2	0.0808	0.0664	35.7335	7.1751
28 days	1	1	0.0726	0.2011	2.1777	0.9064
	1	2	0.0803	0.2412	9.5725	1.1085
	2	1	0.1627	0.1479	3.0993	1.3704
	2	2	0.0785	0.0993	4.1169	1.9750
56 days	1	1	0.0244	0.2117	0.0006	0.0000
	1	2	0.0542	0.1414	0.0004	0.0040
	2	1	0.0401	0.0678	0.0004	0.6812
	2	2	0.0323	0.1294	0.0009	0.6096





Figures 11 and 12.—Banks Lake 2,4-D dissipation in hydrosoil over a 56-day period following treatment. o = hydrosoil sampled in site 1. • = hydrosoil sampled in site 2.

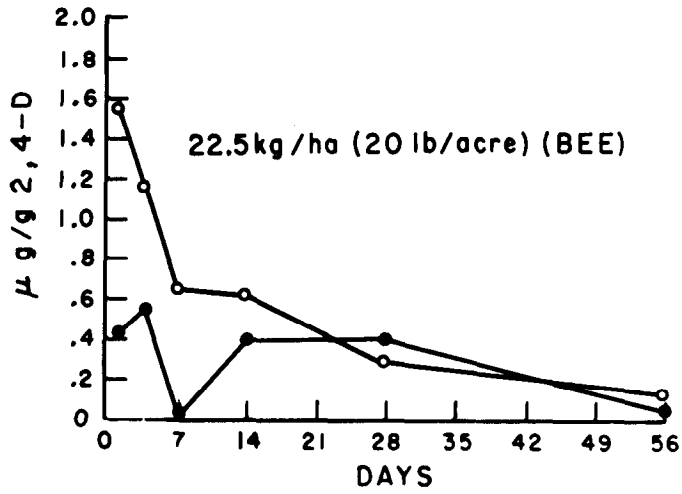


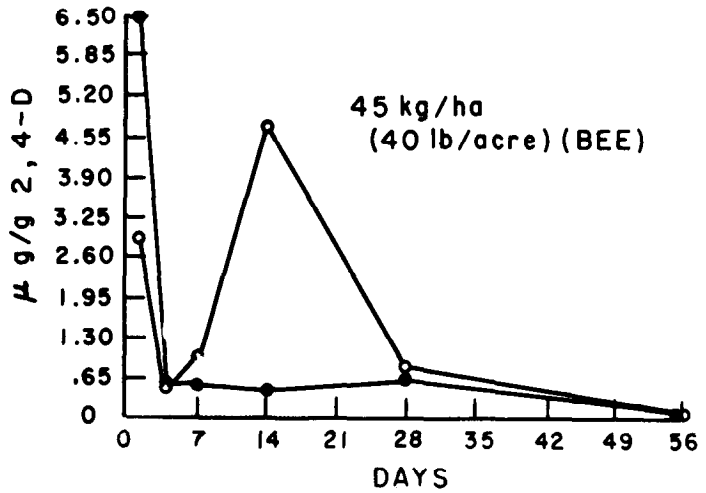


Figures 13 and 14.—Banks Lake 2,4-D dissipation in hydrosoil over a 56-day period following treatment. o = hydrosoil sampled in site 1. • = hydrosoil sampled in site 2.

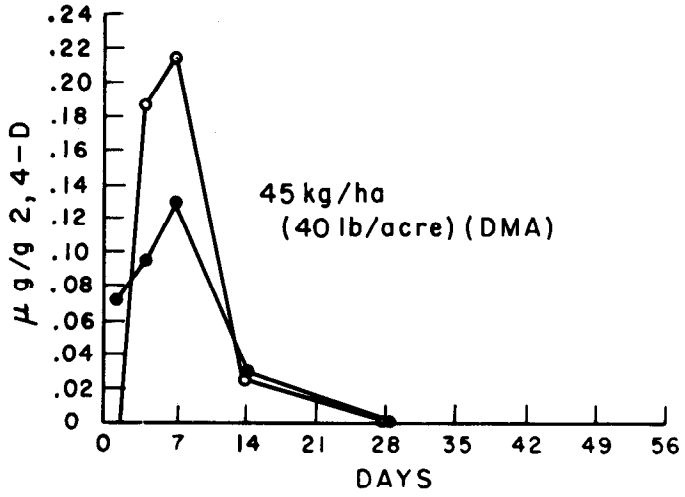
Table 13.—*Fort Cobb Reservoir 2,4-D hydrosoil residues.*

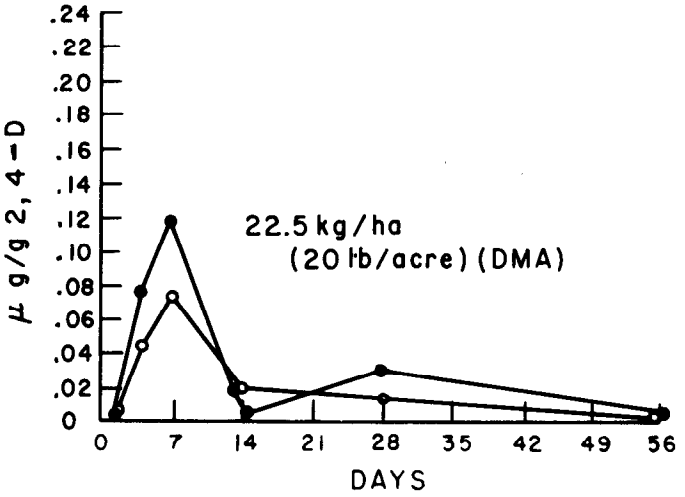
Posttreatment sampling day	Site No.	Rep. No.	Plot 1 22.5 kg/ha (20 lb/acre) BEE (μg/g)	Plot 2 45 kg/ha (40 lb/acre) BEE (μg/g)	Plot 3 45 kg/ha (40 lb/acre) DMA (μg/g)	Plot 4 22.5 kg/ha (20 lb/acre) DMA (μg/g)
1 day	1	1	0.1430			
	1	1	1.1760	0.6866	0.0000	0.0000
	1	2	3.3540	2.2261	0.0000	0.0000
	2	1	0.2901	6.3603	0.0000	0.0000
	2	2	0.5950	6.5938	0.1433	–
4 days	1	1	2.3520	0.4666	0.0971	0.0893
	1	2	–	0.4853	0.2758	–
	2	1	0.5021	0.4497	0.0825	0.1518
	2	2	0.6093	0.4824	0.1103	–
	2				0.0928	
7 days	1	1	0.6447	1.0344	0.3166	0.0000
	1	2	0.6829	0.9063	0.1136	0.1495
	2	1	–	0.0841	0.1943	0.1410
	2	2	–	1.2967	0.0641	0.0943
		1		0.2840		
14 days	1	1	0.7585	1.8230	0.0252	0.0334
	1	2	1.0968	7.6420	0.0232	–
	1	–	0.0401			
	2	1	0.4244	0.1420	–	–
	2	2	0.3819	0.8137	0.0566	0.0081
28 days	1	1	0.2714	0.8561	0.0000	0.0135
	1	2	0.3067	0.8302	–	0.0164
	2	1	0.4281	0.8546	–	0.0000
	2	2	0.3863	0.4759	0.0000	0.0602
56 days	1	1	0.0967	0.1304	–	0.0000
	1	2	0.1191	0.0992	–	0.0000
	1	–	0.1546			
	2	1	–	0.0179	0.0000	0.0000
	2	2	0.1262	0.0764	0.0000	0.0024





Figures 15 and 16.—Fort Cobb 2,4-D dissipation in hydrosoil over a 56-day period following treatment. o = hydrosoil sampled in site 1. • = hydrosoil sampled in site 2.





Figures 17 and 18.—Fort Cobb 2,4-D dissipation in hydrosoil over a 56-day period following treatment. ○ = hydrosoil sampled in site 1. • = hydrosoil sampled in site 2.

Decomposition Products of 2,4-D

Banks Lake, 2,4-Dichlorophenol (table 14).—No 2,4-D Dichlorophenol was found in Banks Lake pretreatment hydrosoil samples from any of the four plots. Hydrosoil residues throughout the 56- day sampling period were less than 0.3000 μg/g, with one exception. A residue of 4.5460 μg/g was detected in plot 3 treated at the 45 kg/ha (40-lb/acre) rate with BEE at 1-day posttreatment.

Residues within plots 1 and 2 treated at 22.5 and 45 kg/ha (20 and 40 lb/acre) with DMA, respectively, throughout the 56-day sampling period were less than 0.0500 μg/g.

Residues within plots 3 and 4 treated at 45 and 22.5 kg/ha (40 and 20 lb/acre), respectively, with BEE were distributed throughout the 14-through 56-day posttreatment interval.

Banks Lake dimethylnitrosamines (table 15).—Trace levels of dimethylnitrosamines were found in Banks Lake pretreat water samples collected in plots 1, 2, 3, and at the outlet. Residue levels in pretreatment samples were less than 1.0 μg/L.

Naturally occurring amines and nitrogen in water could form dimethylnitrosamines resulting in trace levels as observed in pretreatment samples.

In water samples analyzed from 1- day to 14-days posttreatment, only three dimethylnitrosamine residues were observed. Plot 1 was treated at 22.5 kg/ha (20 lb/acre) with DMA residues of 0.2000 and 0.3000 μg/L were observed within the plot and at the outlet, respectively. At 7-days posttreatment, a residue of 0.1000 μg/L was observed in plot 4, which had been treated at 22.5 kg/ha (20 lb/acre) with BEE.

Fort Cobb Reservoir 2,4-Dichlorophenol (table 16).—Trace levels of 2,4-Dichlorophenol were found in Fort Cobb Reservoir pretreatment hydrosoil samples in plots 1 and 2. Residue levels in plots 1 and 2 were 0.0234 and 0.0114 μg/g, respectively.

As with Banks Lake, hydrosoil residues throughout the 56-day sampling period were less than 0.3000 μg/g. Phenol residues found in plots 1 and 2, treated at 22.5 and 45 kg/ha (20 and 40 lb/acre) with BEE, respectively, were uniformly distributed throughout the 56-day sampling period. The maximum phenol residue found in BEE treated plots was 0.2133 μg/g found in plot 1 at 56 days' posttreatment.

Fort Cobb Reservoir dimethylnitrosamine (table 17).—Trace levels of dimethylnitrosamines were found in Fort Cobb Reservoir pretreatment water samples in plot 4 and at the outlet. Pretreatment residues in plot 4 and at the outlet were 0.2000 and 1.000 μg/L, respectively.

As found at Banks Lake, naturally occurring amines and nitrogen in water could form dimethylnitrosamines resulting in trace levels as observed in some pretreatment samples. Dimethylnitrosamine residues were generally low and did not exceed 2.0 μg/L over the 14-day posttreatment sampling. At 4-days posttreatment, residues were the highest for all four plots. In plots 1 and 2, treated at 22.5 and 45 kg/ha (20 and 40 lb/acre) with BEE, residues were 1.2000 and 1.6000 μg/L, respectively, while plots 3 and 4—treated at 45 and 22.5 kg/ha (40 and 20 lb/acre) with DMA— had residues of 1.6000 and 1.000 μg/L, respectively. In plot 1 at 14-days posttreatment, a residue of 0.2000 μg/L was detected.

Environmental Effects of Herbicide Treatments

2,4-D Residues in Fish Flesh

The fish flesh 2,4-D residue study was designed to be conducted concurrently with the water dissipation evaluation as an aid in supporting the proposed tolerance for 2,4-D use in western reservoirs. Determinations of residues in fish flesh resulting from the application of DMA and BEE formulations applied for eurasian watermilfoil control in Western United States reservoirs are required. The United States Environmental Protection Agency established a temporary tolerance of 1 mg/L for residues of the herbicide 2,4-D applied as either the dimethylamine salt or the butoxyl-ethanol ester formulation in edible flesh and in or on various crop commodity groupings [6]. These temporary tolerances, in addition to one of 0.1 mg/L in potable water, were terminated February 28, 1982, on expiration of the Experimental Use Permits.

In terrestrial chemical weed control studies on standing crops, possible damage is generally reflected in stand or yield reduction, both of which are directly measurable. Evaluation of the effects of aquatic chemical applications on nontarget mobile organisms such as fish and benthos is much more difficult. In this study, it was determined that the most valid fish flesh residue data could be obtained by sampling resident populations. The desired sample was specified in the protocol as being at least two species at

Table 14.—*Banks Lake 2,4-D Dichlorophenol hydrosoil residue.*

Posttreatment sampling day	Site No.	Rep. No.	Plot 1 22.5 kg/ha (20 lb/acre) DMA (μg/g)	Plot 2 45 kg/ha (40 lb/acre) DMA (μg/g)	Plot 3 45 kg/ha (40 lb/acre) BEE (μg/g)	Plot 4 22.5 kg/ha (20 lb/acre) BEE (μg/g)
Pretreat			0.0000	0.0000	0.0000	0.0000
1 day	1	1	0.0078	0.0000	0.0023	0.0000
	1	2	0.0000	0.0000	4.5460	0.0000
	2	1	0.0000	0.0000	0.0000	0.0000
	2	2	0.0424	0.0000	0.0000	0.0000
4 days	1	1	0.0000	0.0000	–	0.0000
	1	2	0.0000	0.0000	0.0000	0.0173
	2	1	0.0000	0.0000	0.0000	0.0000
	2	2	0.0000	0.0000	0.0000	–
7 days	1	1	0.0000	0.0000	0.0000	0.0000
	1	2	0.0000	0.0000	0.0000	0.0000
	2	1	0.0217	0.0000	0.0000	0.0000
	2	2	0.0000	0.0000	0.0000	0.0000
14 days	1	1	0.0000	0.0014	0.2746	0.0000
	1	2	0.0108	0.0000	0.0000	0.0136
	2	1	0.0000	0.0014	0.0000	0.0062
	2	2	0.0113	0.0000	0.0592	0.0989
28 days	1	1	0.0000	0.0000	0.0629	0.0090
	1	2	0.0000	0.0000	0.1008	0.0041
	2	1	0.0000	0.0000	0.0199	0.0624
	2	2	0.0000	0.0000	0.0104	0.0113
56 days	1	1	0.0000	0.0000	0.0931	0.0017
	1	2	0.0054	0.0000	0.0000	0.0003
	2	1	0.0000	0.0000	0.0004	0.0000
	2	2	0.0000	0.0070	0.0959	0.0000

Table 15.—*Banks Lake dimethylnitrosamine residues in water.*

Posttreatment sampling day	Plot 1 22.5 kg/ha (20 lb/acre) DMA (μg/L)	Plot 2 45 kg/ha (40 lb/acre) DMA (μg/L)	Plot 3 45 kg/ha (40 lb/acre) BEE (μg/L)	Plot 4 22.5 kg/ha (20 lb/acre) BEE (μg/L)	Outlet (μg/L)
Pretreat	0.4000	0.3000	0.6000	–	0.4000
1 day	0.0000	0.0000	0.0000	0.0000	0.0000
4 days	0.2000	0.0000	0.0000	–	0.3000
7 days	0.0000	0.0000	0.0000	0.1000	0.0000
14 days	0.0000	–	–	0.0000	0.0000

– not determined.

Table 16.—*Fort Cobb Reservoir 2,4-D dichlorophenol hydrosoil residue.*

Posttreatment sampling day	Site No.	Rep. No.	Plot 1 22.5 kg/ha (20 lb/acre) BEE (μg/g)	Plot 2 45 kg/ha (40 lb/acre) BEE (μg/g)	Plot 3 45 kg/ha (40 lb/acre) DMA (μg/g)	Plot 4 22.5 kg/ha (20 lb/acre) DMA (μg/g)
Pretreat			0.0234	0.0000	0.0000	0.0114
1 day	1	1	0.0000			
	1	1	0.0242	0.0050	0.0000	0.0000
	1	2	0.0335	0.0323	0.0000	0.0000
	2	1	0.0000	0.0612	0.0000	0.0000
	2	2	0.0000	0.0000	0.0000	-
4 days	1	1	0.0878	0.0000	0.0000	0.0686
	1	2	-	0.0000	0.0000	-
	2	1	0.0000	0.0000	0.0000	0.0000
	2	2	0.0000	0.0591	0.0000	-
	2				0.0000	
7 days	1	1	0.0000	0.0912	0.0155	0.0000
	1	2	0.0000	0.0000	0.0000	0.0000
	2	1	-	0.0534	0.0259	0.0000
	2	2	-	0.0893	0.0000	0.0000
	-[a]	1		0.0000		
14 days	1	1	0.0000	0.1135	0.0000	0.0000
	1	2	0.0000	0.0618	0.0000	-
	1	-	0.0000			
	2	1	0.0000	0.0000	-	-
	2	2	0.0000	0.0069	0.0694	0.0000
28 days	1	1	0.1078	0.0000	0.0000	0.0000
	1	2	0.0000	0.0286	-	0.0000
	2	1	0.0000	0.0000	-	0.0000
	2	2	0.0000	0.0000	0.0036	0.0000
56 days	1	1	0.0000	0.0000	-	0.0000
	1	2	0.0000	0.0675	-	0.0000
	1	-	0.2133			
	2	1	-	0.0000	0.0000	0.0000
	2	2	0.0000	0.0000	0.0000	0.0000

[a] Site number data missing from sample.
- Not determined.

Table 17.—*Fort Cobb dimethylnitrosamine residues in water.*

Posttreatment sampling day	Plot 1 22.5 kg/ha (20 lb/acre) BEE (μg/L)	Plot 2 45 kg/ha (40 lb/acre) BEE (μg/L)	Plot 3 45 kg/ha (40 lb/acre) DMA (μg/L)	Plot 4 22.5 kg/ha (20 lb/acre) DMA (μg/L)	Outlet (μg/L)
Pretreat	0.0000	0.0000	0.0000	0.2000	1.1000
1 day	0.0000	1.6000	0.0000	0.0000	-
4 days	1.2000	1.6000	1.6000	1.0000	-
7 days	0.0000	0.0000	0.0000	0.0000	0.0000
14 days	0.2000	0.0000	0.0000	0.0000	-

- Not determined.

two trophic levels in the 178- to 254-mm (7- to 10-in) sizes. Both electrofishing and gill net sampling techniques were employed. This method of sampling was used instead of caged fish to more closely simulate an actual exposure. Fish can readily move out of treatment areas and, thus, minimize the probability of continuous exposure.

Fish flesh residue from Banks Lake, plots 1 and 2, 22.5 and 45 kg/ha, ae (20 and 40 lb/acre, ae), DMA (table 18).—The dominant Banks Lake resident fish species used for chemical analysis were white sucker (*Catostomus commersoni*), whitefish (*Coregonus lupeaformis*), and carp (*Cyprinus carpio*). Length of fish averaged 343 mm (13.5 in) for white sucker, 317 mm (12.5 in) for whitefish and 409 mm (16.1 in) for carp.

No trace levels of DMA were found in pretreat fish samples collected on July 8, 1981, from plots 1 and 2. This confirms pretreatment water and hydrosoil data which was free of background 2,4-D.

Table 18.—*Banks Lake DMA residues in fish (individual and average).*

Sampling date	Plot	Rep. No.	Fish type	µg/g	Average µg/g (± S.D.[2])
Pretreat	1	1	Carp	0.0000	0.0000 (±0.0000)
	2	1	Carp	0.0000	
	2	2	Carp	0.0000	
	2	3	Carp	0.0000	0.0000
	2	4	Carp	0.0000	(±0.0000)
	2	5	Carp	0.0000	
	2	6	Carp	0.0000	
1 day	1	1	Carp	0.1938	
	1	2	Carp	0.0000	0.646
	1	3	Carp	0.0000	(±0.1119)
	2	1	Whitefish	0.0000	
	2	2	White sucker	0.0000	0.0000
	2	3	White sucker	0.0000	0.0000
	2	4	White sucker	0.0000	
7 days	1	1	Carp	0.0495	0.0495
	1	2	Carp	–[1]	(±0.0000)
	2	1	Carp	0.0000	
	2	2	Carp	0.1053	0.512
	2	3	Carp	0.0484	(±0.0527)
14 days	1	1	Carp	0.0000	
	1	2	Carp	0.0000	0.0405
	1	3	Carp	0.1217	(±0.0702)
	2	1	Carp	0.2592	
	2	2	Carp	0.0000	0.0864
	2	3	Carp	0.0000	(±0.1496)

[1] Sample not analyzed.
[2] Standard deviation of mean.

Over the 14-day sampling period, little or no DMA residues were found in fish collected in plots 1 and 2 which were treated at 22.5 and 45 kg/ha (20 and 40 lb/acre), respectively (table 18). Residues throughout the 14-day sampling period never exceeded 0.3000 µg/g. The lack of 2,4-D residues in the majority of fish analyzed up to 14-days' posttreatment could be attributed to minimal exposure time within the treated area due to movement in and out of these areas.

Fish flesh residue from Banks Lake, plots 3 and 4, 45 and 22.5 kg/ha, ae (40 and 20 lb/acre, ae), BEE (table 19).—The dominant Banks Lake resident fish species used for chemical analysis was common carp (*Cyprinus carpio*). Carp ranged from 200- to 550-mm (8- to 22-in) length.

No trace levels of BEE were found in pretreat fish samples collected on July 8, 1981, from plots 3 and 4. This confirms pretreatment water and hydrosoil data which were free of background 2,4-D.

Table 19.—*Banks Lake BEE residues in fish (individual and average).*

Sampling date	Plot	Fish type	µg/g	Ave. µg/g
Pretreat	3	Carp	0.0000	0.0000
	3	Carp	0.0000	
	3	Carp	0.0000	
1 day	3	Carp	0.0000	0.0000
	Outlet	Sucker	0.0000	0.0000
4 days	3	Carp	0.0000	0.0000
	3	Carp	0.0000	
	4	Carp	0.0000	0.0000
	4	Carp	0.0000	
	4	Carp	0.0000	
	4	Carp	0.0000	
7 days	3	Carp	0.0000	0.0000
	3	Carp	0.0000	
	3	Carp	0.0000	
	3	Carp	0.0000	
	3	Carp	0.0000	
	4	Carp	0.0000	0.0000
	4	Carp	0.0000	
	4	Carp	0.0000	
	4	Carp	0.0000	
	4	Carp	0.0000	
	4	Carp	0.0000	
14 days	3	Carp	0.0072	0.0072
	4	Carp	0.0000	0.0000
	4	Carp	0.0000	

Over the 14-day sampling period, little or no BEE residues were found in fish collected in plots 3 and 4 which were treated at 45 and 22.5 kg/ha (40 and 20 lb/acre), respectively (table 19). The lack of 2,4-D residues in the majority of fish analyzed up to 14 days' posttreatment could be attributed to minimal exposure time within the treated areas due to movement in and out of these areas.

Fish population in Banks Lake was extremely sparse in the study site area during 1981. This was caused by two factors: (1) 2 previous years of drawdown to attempt to control eurasian watermilfoil influenced the population and, (2) the shallow areas selected for the study site probably did not attract any species but the common carp.

Fish flesh residues from Fort Cobb Reservoir, plots 1 and 2, 22.5 and 45 kg/ha, ae (20 and 40 lb/acre, ae) BEE (table 20).—The dominant resident fish species selected for chemical analysis were common carp (*Cyprinus carpio*) and largemouth bass (*Micropterus salmoides*). Occasionally, bluegill (*Lepomis macrochirus*) was used to substitute for lack of largemouth bass samples. Largemouth bass averaged approximately 300-mm (12-in) length while carp averaged 350-mm (14-in) length.

Table 20 illustrates the fact that trace levels of BEE were found in pretreatment fish samples taken on August 18, 1981, from plot 1. This confirms that trace amounts of 2,4-D were present in the pretreated water and hydrosoil samples. Pretreatment residue levels in carp were 0.0083 μg/g and 0.0003 μg/g in largemouth bass.

Fish samples were analyzed up to 14 days' posttreatment in plots 1 and 2 which were treated at 22.5 and 45 kg/ha (20 and 40 lb/acre) with BEE, respectively. Residue levels over the 14-day sampling period indicate higher levels in carp than in largemouth bass. Residues in carp and largemouth bass were higher in plot 1 treated at 22.5 kg/ha (20 lb/acre) with BEE than in plot 2 treated at 45 kg/ha (40 lb/acre) with BEE throughout the 14-day sampling period. With one exception, loss of 2,4-D in fish in plots 1 and 2 occurred in both species over the 14-day sampling period. In plot 1, residues in carp increased from 0.0216 μg/g on 1-day posttreatment to 0.0417 μg/g on 14-day posttreatment. Overall BEE residues were less than 0.05 μg/g in both plots 1 and 2 over the 14-day sampling period.

Higher residues in carp occur because the species is a bottom feeder (detritus) and could ingest granular ester pellets from the hydrosoil. Lower residues in largemouth bass may occur because the species is a fish predator (piscivorous), not feeding on fish at the hydrosoil level. Apparently no bioaccumulation occurred.

Table 20.—*Fort Cobb Reservoir BEE residues in fish (individual and average).*

Sampling date	Plot	Rep. No.	Fish type	μg/g	Average μg/g (± S.D.)*
Pretreats	1	3	LMB**	0.0000	
	1	2	LMB	0.0000	0.0003
	1	1	LMB	0.0009	(±0.0005)
	1	1	Carp	0.0062	0.0083
	1	2	Carp	0.0103	(±0.0028)
	1	2	LMB	0.0000	0.0265
	1	3	LMB	0.0531	(±0.0376)
1 day	1	1	Carp	0.0666	
	1	2	Carp	0.0025	0.0216
	1	3	Carp	0.0207	(±0.0329)
	2	1	LMB	0.0055	
	2	2	LMB	0.0025	0.0027
	2	3	LMB	0.0009	(±0.0023)
	2	1	Carp	0.0138	
	2	2	Carp	0.0266	0.0233
	2	3	Carp	0.0543	(±0.0207)
7 days	1	1	LMB	0.0083	
	1	2	LMB	0.0066	0.0066
	1	3	LMB	0.0059	(±0.0012)
	1	1	Carp	0.0469	0.0301
	1	2	Carp	0.0297	(±0.0120)
	2		Bluegill	0.0004	0.0000
	2	1	Carp	0.0000	(±0.0000)
	2	2	Carp	0.0051	0.0063
	2	3	Carp	0.0385	(±0.0210)
14 days	1	1	LMB	0.0018	
	1	2	LMB	0.0027	0.0012
	1	3	Bluegill	0.0000	(±0.0014)
	1	1	Carp	0.0000	
	1	2	Carp	0.0002	0.0417
	1	3	Carp	0.1498	(±0.0864)
	2	1	LMB	0.0000	0.0005
	2	2	LMB	0.0016	(±0.0011)
	2	1	Carp	0.0059	
	2	2	Carp	0.0000	0.0000
	2	3	Carp	0.0000	

* Average μg/g = values with background subtracted.
** LMB = largemouth bass.
SD = Standard deviation of mean.

Fish flesh residue from Fort Cobb Reservoir, plots 3 and 4, 45 and 22.5 kg/ha, ae (40 and 20 lb/acre, ae), DMA (table 21).—The dominant resident fish species selected for chemical analyses were common carp (*Cyprinus carpio*) and largemouth bass (*Micropterus salmoides*). Carp averaged approximately 380-mm (15-in) in

length while largemouth bass averaged 300-mm (12-in) in length.

Examination of table 21 indicates a trace level of 0.0084 μg/g DMA was observed in one pretreat carp specimen taken from plot 4. This is a confirmation that trace amounts of 2,4-D are present in pretreatment water, and that water hydrosoil samples contained trace levels of 2,4-D.

Fish samples were analyzed up to 14-days' posttreatment in plots 3 and 4 treated at 45 and 22 kg/ha (40 and 20 lb/acre) with DMA, respectively. Residue levels at 1-day posttreatment indicate that residues in plot 3 for carp and largemouth bass were higher than plot 4 for both fish species.

At 4-days' posttreatment, residues were observed only in largemouth bass samples collected in plot 4. Residues at 14-days posttreatment were higher in carp samples taken from plot 4 than in carp and largemouth bass samples in plot 3. Overall, DMA residues were less than 0.35 μg/g in plots 3 and 4 over the 14-day sampling period and did not exceed the food additive tolerance of 1.0 mg/L (1.0 p/m).

Residues in both species can be attributed to the uniform distribution of DMA throughout the water column and longer exposure times due to treatments being in sheltered bays.

Effects of Herbicide Residue on Plankton Populations

Plankton samples collected at the two reservoir sites throughout the study period were processed at the Denver laboratory using three replicated subsample volumes of 1.0 mL each to determine cell numbers and cell identifications. Sedgewick-Rafter or Palmer counting cells were utilized for microscopic examination. The resulting plankton data were analyzed and compared with water and hydrosoil 2,4-D residue data.

Banks Lake plankton.—Plankton identified from this study site are identified in table 22.

The dominant algae during the study was the filamentous green algae *Ulothrix* sp. Zooplankton populations were dominated by copepods (fig.19).

Abundance of zooplankton and phytoplankton in shown in figure 20. The total numbers of planktors showed some seasonal increase throughout the 56-day study period. This may be related to normal seasonal increases in populations resulting from an increase in temperature, light, and environmental factors other than herbicide.

No changes in population distribution or abundance of plankton in Banks Lake occurred within

Table 21.—*Fort Cobb Reservoir DMA residues in fish (individual and average).*

Sampling date	Plot	Rep. No.	Fish type	μg/g	Average μg/g (± S.D.)*
Pretreats	4	1	LMB*	0.0000	
	4	2	LMB	0.0000	0.0000
	4	3	LMB	0.0000	(±0.0000)
	4	1	Carp	0.0084	
	4	2	Carp	0.0000	0.0028
	4	3	Carp	0.0000	(±0.0048)
1 day	3	1	LMB	0.0584	0.0663
	3	2	LMB	0.0742	(±0.0111)
	3	3	LMB	-	
	3	1	Carp	0.0316	
	3	2	Carp	0.0000	0.0105
	3	3	Carp	0.0000	(±0.0182)
	4	1	LMB	0.0000	
	4	2	LMB	0.0088	0.0001
	4	3	LMB	0.0000	(±0.0022)
	4	1	Carp	0.0000	
	4	2	Carp	0.0000	0.0031
	4	3	Carp	0.0179	(±0.0075)
4 days	3	1	LMB	0.0000	
	3	2	LMB	-**	0.0000
	3	3	LMB	0.0000	(±0.0000)
	3	1	Carp	0.0000	
	3	2	Carp	0.0000	0.0000
	3	3	Carp	0.0000	(±0.0000)
	4	1	LMB	0.0084	
	4	2	LMB	0.0000	0.0140
	4	3	LMB	0.0421	(±0.0195)
	4	1	Carp	0.0000	
	4	2	Carp	0.0000	0.0000
	4	3	Carp	0.0000	(±0.0000)
	4	4	Carp	0.0000	
14 days	3	1	LMB	0.0268	
	3	2	LMB	0.0000	0.0143
	3	3	LMB	0.0162	(±0.0135)
	3	1	Carp	0.3266	
	3	2	Carp	0.0000	0.1088
	3	3	Carp	0.0000	(±0.1885)
	4	1	LMB	0.0000	
	4	2	LMB	-	0.0000
	4	3	LMB	-	(±0.0000)
	4	1	Carp	0.0989	
	4	2	Carp	-	0.0914
	4	3	Carp	0.0895	(±0.0094)

* LMB = largemouth bass.

** - = sample not analyzed.

SD = Standard deviation of mean.

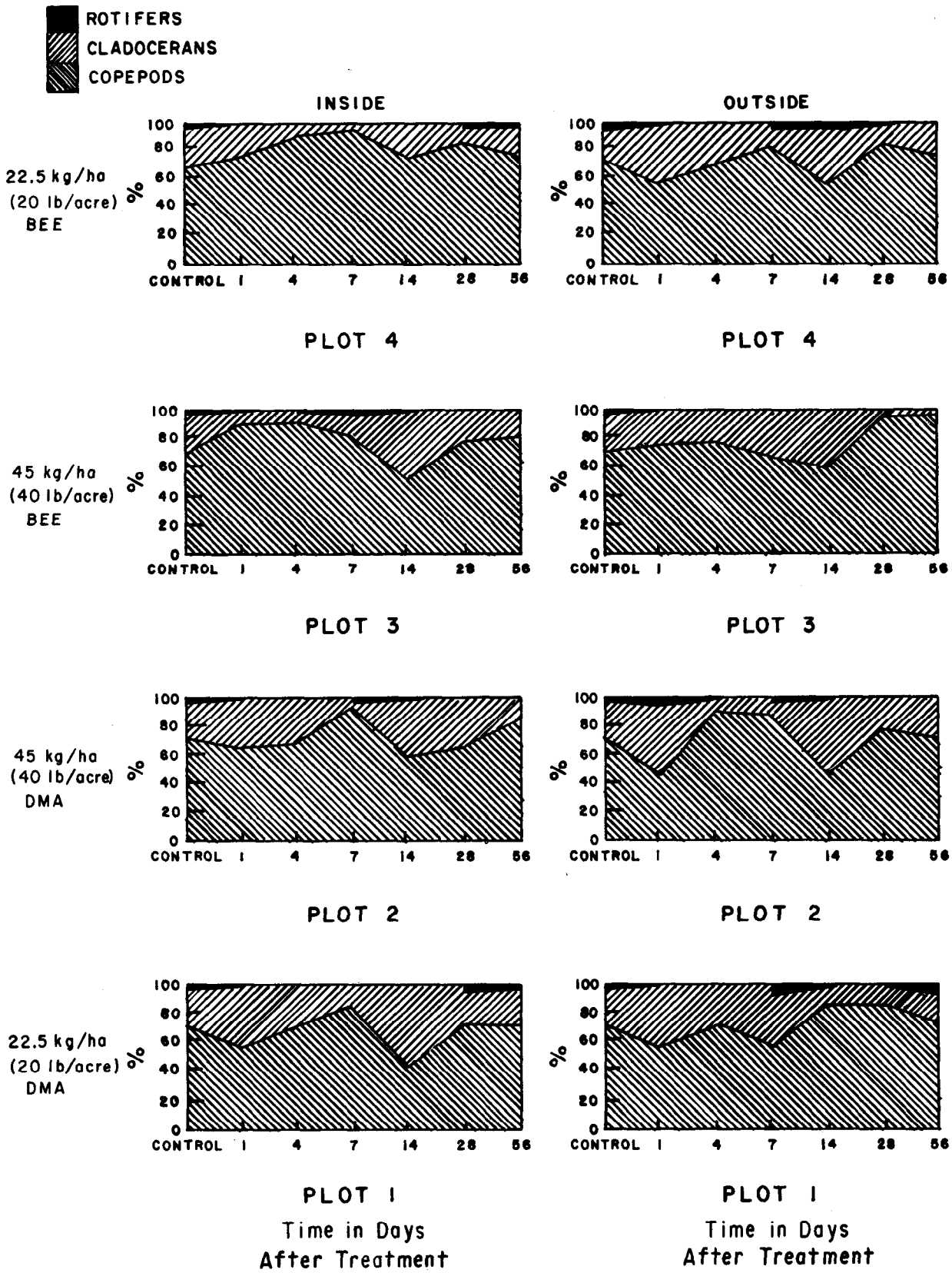


Figure 19.—Banks Lake zooplankton composition.

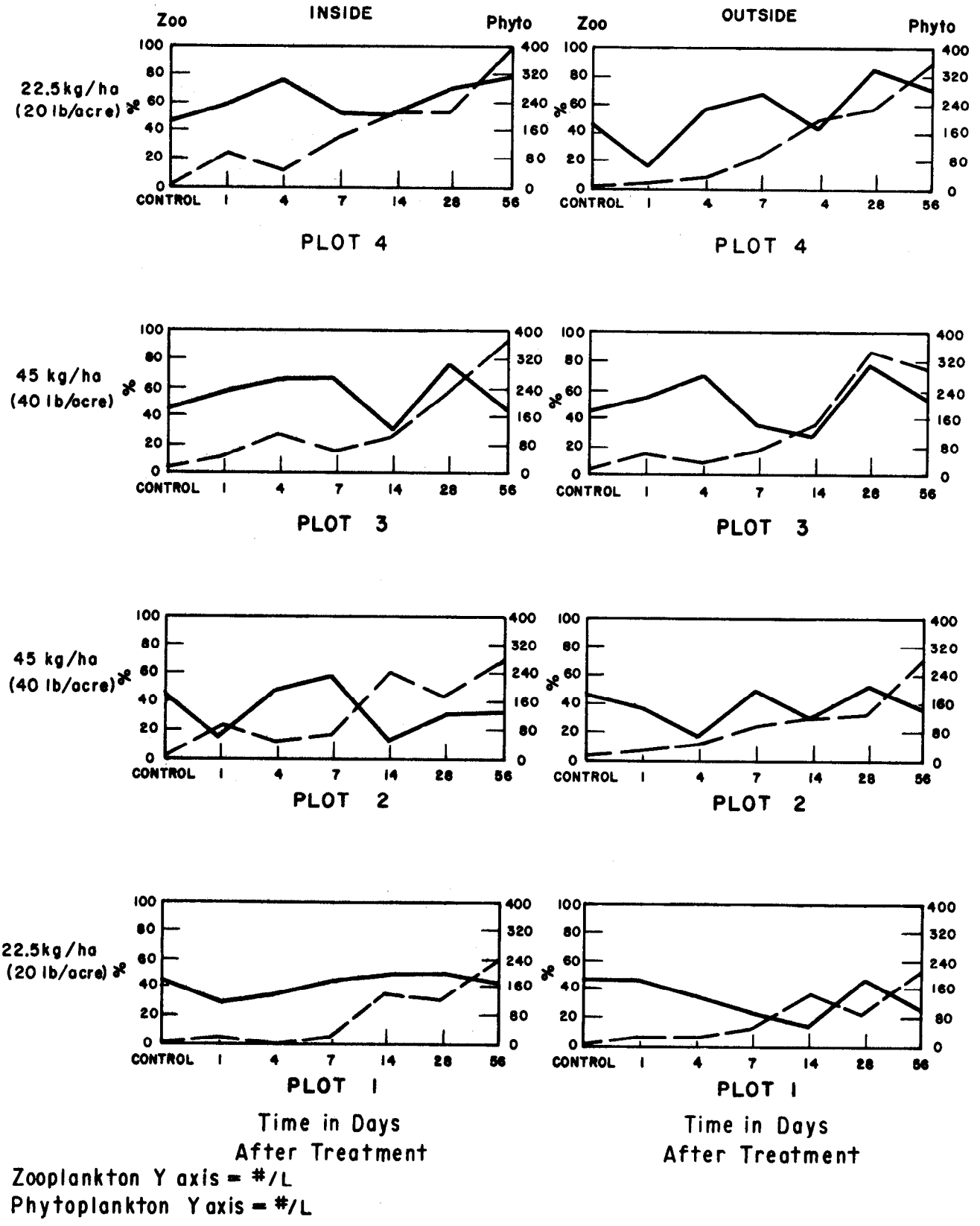


Figure 20.—Banks Lake zooplankton-phytoplankton abundance.

Table 22.—*Banks Lake Plankton genera.*

Zooplankton			Phytoplankton			
Copepods	Cladocerans	Rotifers	Green algae	Blue-green algae	Diatoms	Dinoflagellates
Diaptomus	*Daphnia*	*Keratella*	*Ulothrix*	*Anabaena*	*Fragilaria*	*Dinobryon*
Cyclops	*Sida*	*Kellicottia*	*Sphaerocystis*			*Ceratium*
	Bosmina	*Asplanchna*	*Spyrogyra*			
	Leptodora					

the study period which could be directly related to determined herbicide residues. The populations within and outside of treated plots were found to be quite similar, and the slight fluctuations of populations noted are not easily attributed to any determined effects from the herbicides.

The resulting diversity indices were plotted from samples taken inside and outside the treated plots (fig. 21). This figure shows that very little difference can be seen between the herbicide treated areas and those outside the treatment plots.

Fort Cobb plankton.—The dominant algae in Fort Cobb Reservoir was the blue-green algae *Aphanizomenon.* Zooplankton populations were predominantly cladocerans. Table 23 lists plankton genera found at the study site.

Figure 22 shows the composition of the zooplankton community throughout the study. There appeared to be no significant change throughout the study period in the plankton composition either inside or outside the herbicide treated areas.

Plankton population abundance was much higher than that found in Banks Lake (fig. 23). Some differences in the zooplankton, and particularly phytoplankton, populations appeared to occur when comparing samples from inside and outside of the treated plots. These population abundances do not seem to show much relationship to herbicide residues found in the water during the course of the study. Efforts were made to treat the data statistically, but no significant relationships emerged that might be related to herbicide exposures. These observed variations may well be microenvironmental effects but, more likely, are experimental error related to field or laboratory sampling. Since this portion of the overall study was designed to demonstrate any major responses of these aquatic organisms to herbicide exposure, only minimal replication of field sampling was conducted.

The diversity indices calculated for the plankton samples collected from Fort Cobb Reservoir are shown in figure 24. Similar to Banks Lake, the diversity of species found within and outside the treated plot areas shows very little difference.

In conclusion, analyses of plankton populations as possible indicators of environmental effects resulting from the experimental herbicide plot treatments showed no readily discernible adverse effects. This was especially evident at Banks Lake. If any adverse effects occurred during the study, they did not correlate well with measured 2,4-D water residues and were of very short duration. The general conclusion from this portion of the study is that no significant changes in the plankton populations occurred as the result of experimental herbicide treatments.

Table 23.—*Fort Cobb Reservoir plankton species (observed in samples during 1981).*

Zooplankton			Phytoplankton	
Copepods	Cladocerans	Rotifers	Green algae	Blue-green algae
Cyclops	Daphnia	Asplanchna	Ulothrix	Anabaena
Diaptomus	Bosmina	Polyarthra		Aphanizomenon
	Sida			

Environmental Effects on Water Quality

One-liter water samples were collected at mid-depth both within each of the treated plots and at the outlets of each reservoir throughout the period of study. These samples were preserved by freezing and transported to the laboratory for analysis.

In addition, in situ physical-chemical water quality parameters were collected periodically throughout the study in treated and nontreated areas utilizing a Model 4000 Hydrolab Corporation electronic multiparameter instrument.

The resulting physical-chemical water measurements collected from 0- through 56-days, post-treatment, are given for Banks Lake (table 24) and Fort Cobb Reservoir (table 25).

Comparing temperature, pH, DO (dissolved oxygen), and conductivity between the treated

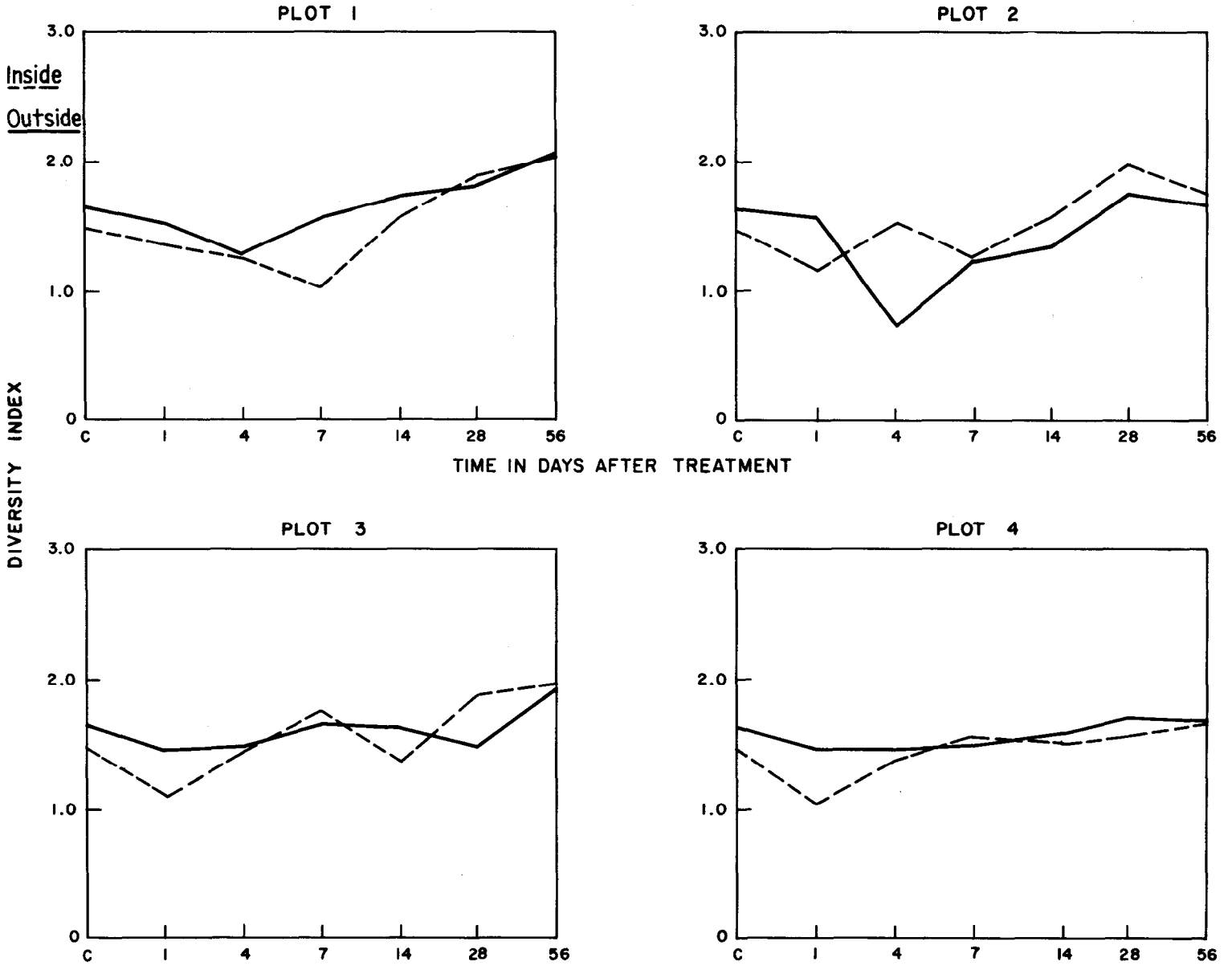


Figure 21.—Banks Lake zooplankton diversity.

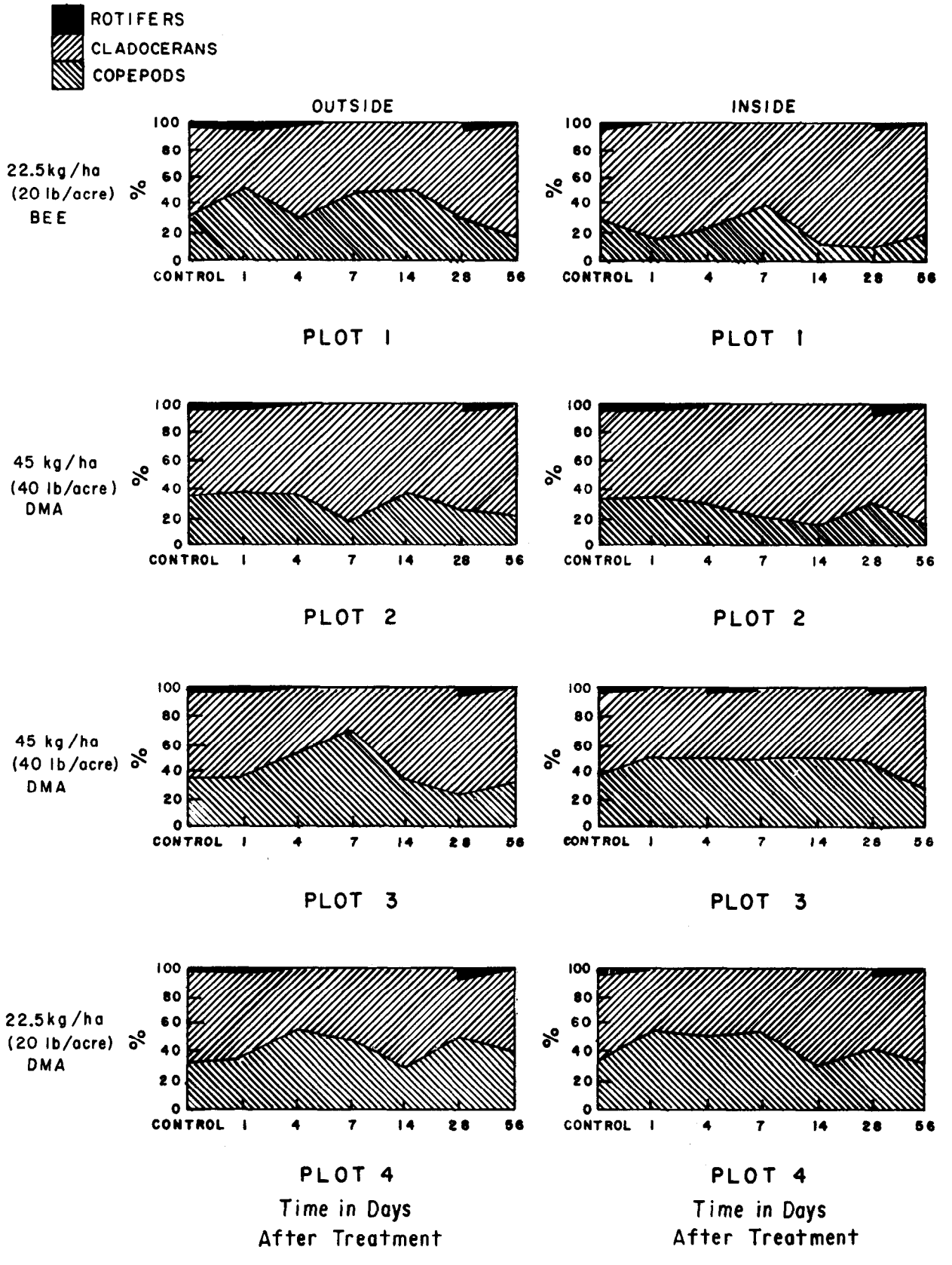


Figure 22.—Fort Cobb zooplankton composition.

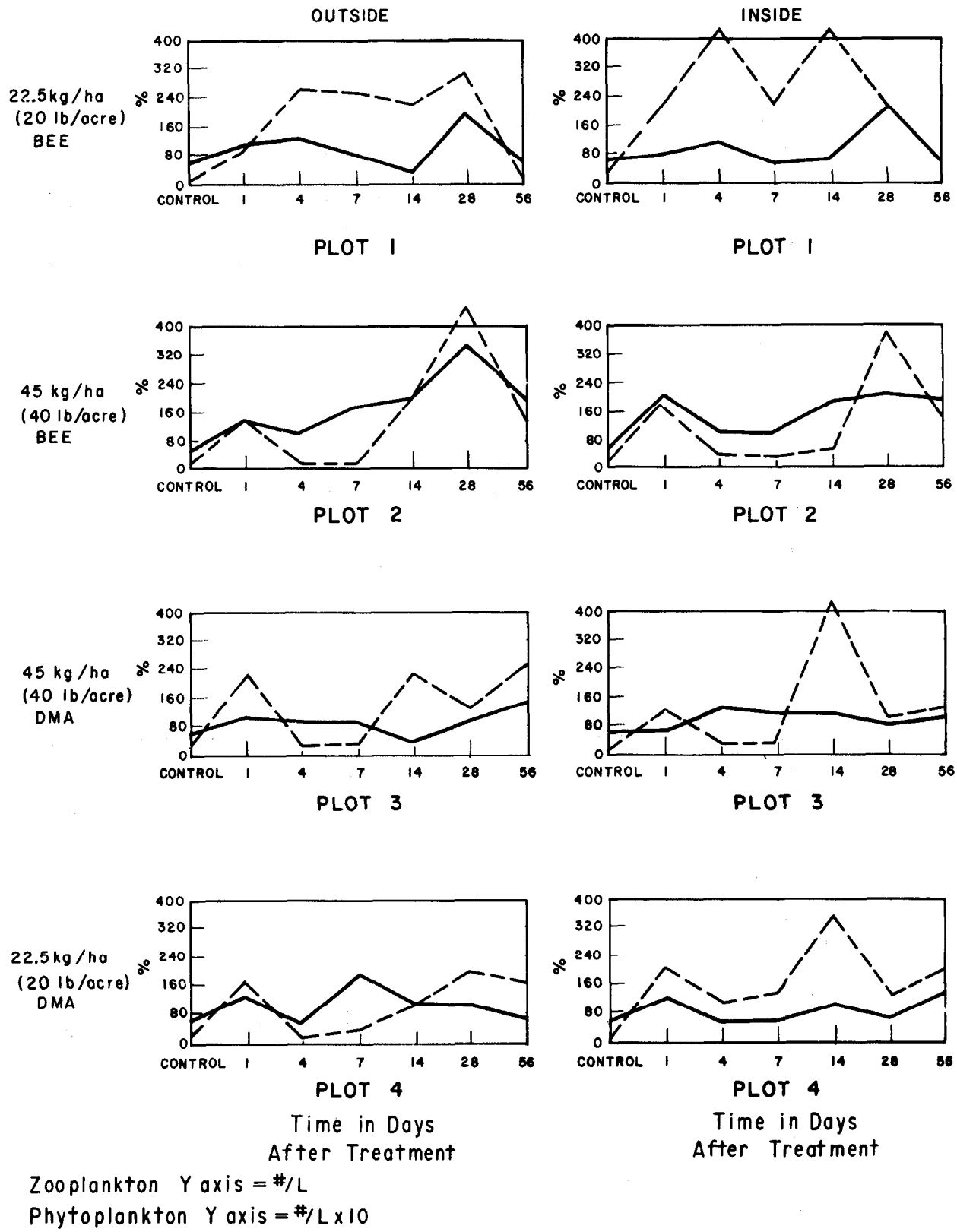


Figure 23.—Fort Cobb Reservoir zooplankton-phytoplankton abundance.

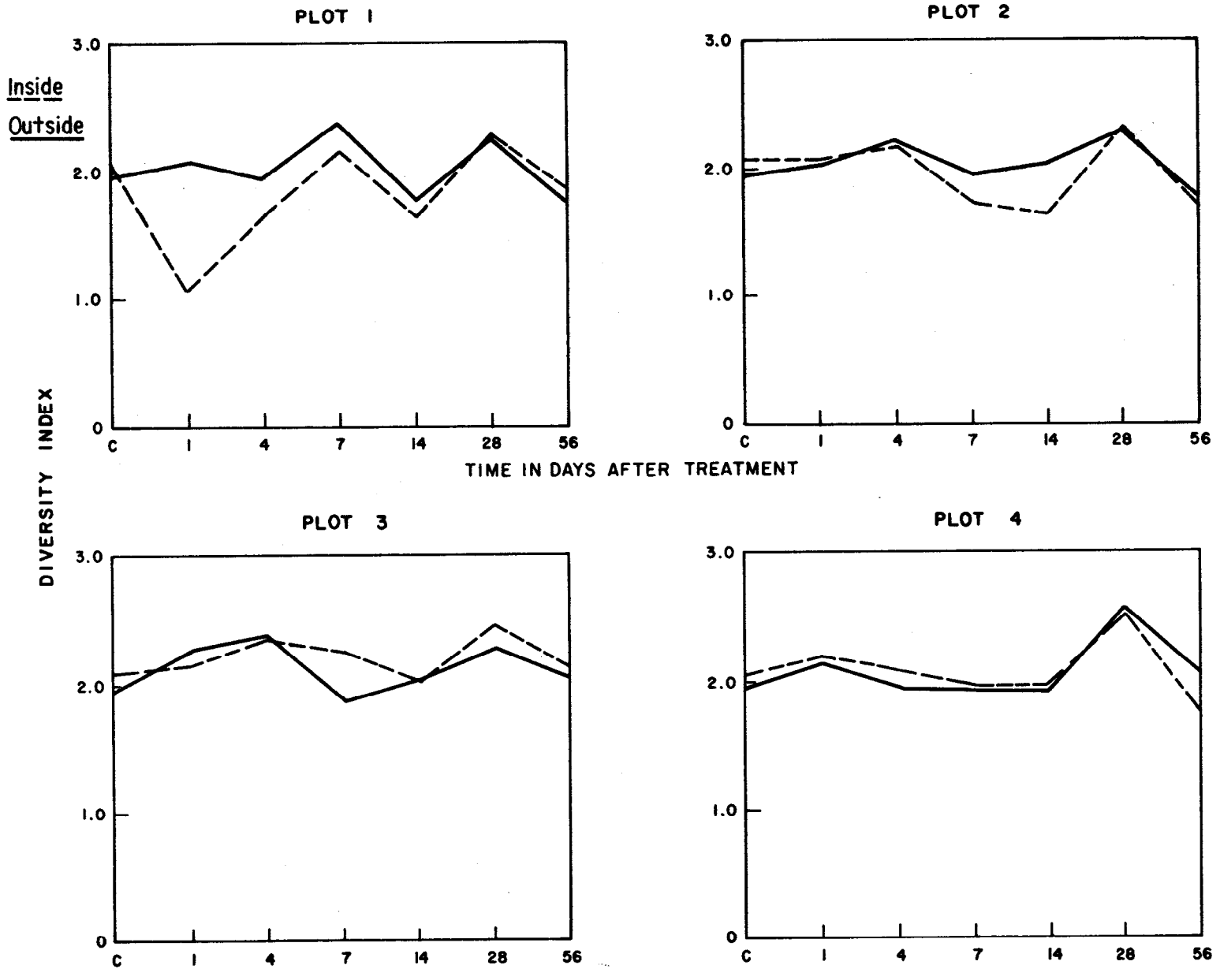


Figure 24.—Fort Cobb zooplankton diversity.

Table 24.—*Banks Lake in situ*[1] *physical-chemical water quality parameters.*

Chemical/physical parameters	Days, posttreatment						
	0	1	4	7	14	28	56
		Herbicide DMA application rate 22.5 kg/ha (20 lb/acre), ae Herbicide plot No. 1, site 1, within treated plot					
Temperature, °C	–	16.1	16.7	17.0	19.5	19.6	18.4
pH	–	7.8	7.8	7.7	7.3	7.1	7.6
Dissolved oxygen, mg/L	–	9.8	10.0	9.4	9.1	9.5	7.5
Conductivity, μS/cm	–	150	150	139	148	153	125
		Herbicide DMA application rate 45 kg/ha (40 lb/acre), ae Herbicide plot No. 2, site 1, within treated plot					
Temperature, °C	15.7	16.5	16.7	17.0	19.5	19.2	18.4
pH	7.8	7.8	7.7	7.7	7.7	7.2	7.6
Dissolved oxygen, mg/L	9.4	10.0	9.7	9.3	9.2	9.3	7.7
Conductivity, μS/cm	147	150	149	135	144	159	141
		Herbicide BEE application rate 45 kg/ha (40 lb/acre), ae Herbicide plot No. 3, site 2, within treated plot					
Temperature, °C	–	16.1	16.0	17.4	20.4	18.5	18.3
pH	–	7.7	7.6	7.8	7.7	7.1	7.7
Dissolved oxygen, mg/L	–	9.6	9.6	9.4	9.1	9.2	7.0
Conductivity, μS/cm	–	147	148	136	149	156	139
		Herbicide BEE application rate 22.5 kg/ha (20 lb/acre), ae Herbicide plot No. 4, site 2, within treated plot					
Temperature, °C	–	16.7	16.4	17.3	21.7	18.9	18.2
pH	–	7.8	7.7	7.7	7.7	7.1	7.5
Dissolved oxygen, mg/L	–	9.8	9.8	9.5	8.7	9.4	7.4
Conductivity, μS/cm	–	145	152	140	145	158	140
		Water sample site: Near reservoir outlet					
Temperature, °C	–	15.8	16.6	–	19.4	20.1	18.3
pH	–	7.7	7.8	–	7.8	7.3	7.5
Dissolved oxygen, mg/L	–	9.8	10.0	–	9.2	9.3	7.5
Conductivity, μS/cm	–	151	153	–	144	162	146

[1] In situ data obtained by use of Model 4000 Hydrolab.

Table 25.—*Fort Cobb Reservoir in situ*[1] *physical-chemical water quality parameters.*

Chemical/physical parameters	Days, posttreatment						
	0	1	4	7	14	28	56
	Herbicide BEE application rate 22.5 kg/ha (20 lb/acre), ae Herbicide plot No. 1, site 2, within treated plot						
Temperature, °C	25.6	–	–	–	–	22.1	20.7
pH	8.3	–	–	–	–	8.3	8.0
Dissolved oxygen, mg/L	3.1	–	–	–	–	7.2	6.6
Conductivity, μS/cm	601	–	–	–	–	263	550
	Herbicide BEE application rate 45 kg/ha (40 lb/acre), ae Herbicide plot No. 2, site 2, within treated plot						
Temperature, °C	25.6	–	–	–	–	20.7	19.5
pH	7.5	–	–	–	–	8.1	8.1
Dissolved oxygen, mg/L	3.1	–	–	–	–	6.2	6.6
Conductivity, μS/cm	604	–	–	–	–	549	510
	Herbicide DMA application rate 45 kg/ha (40 lb/acre), ae Herbicide plot No. 3, site 2, within treated plot						
Temperature, °C	25.5	–	–	–	–	21.7	19.3
pH	7.4	–	–	–	–	7.7	7.3
Dissolved oxygen, mg/L	3.0	–	–	–	–	5.4	6.0
Conductivity, μS/cm	598	–	–	–	–	508	410
	Herbicide DMA application rate 22.5 kg/ha (20 lb/acre), ae Herbicide plot No. 4, site 2, within treated plot						
Temperature, °C	25.5	26.0	–	–	–	23.2	19.5
pH	7.9	7.6	–	–	–	8.1	8.0
Dissolved oxygen, mg/L	3.2	3.8	–	–	–	5.6	6.1
Conductivity, μS/cm	608	590	–	–	–	558	560
	Water sample site: Near Chickasha Reservoir outlet						
Temperature, °C	–	–	–	–	–	23.7	19.4
pH	–	–	–	–	–	8.6	7.9
Dissolved oxygen, mg/L	–	–	–	–	–	7.0	5.8
Conductivity, μS/cm	–	–	–	–	–	558	560
	Water sample site: Near Anadarko Reservoir outlet						
Temperature, °C	–	–	–	–	–	24.0	–
pH	–	–	–	–	–	8.6	–
Dissolved oxygen, mg/L	–	–	–	–	–	7.1	–
Conductivity, μS/cm	–	–	–	–	–	558	–

[1] In situ data obtained by use of Model 4000 Hydrolab.

plots and untreated area at the reservoir outlet showed few significant differences occurring between the sample site areas. Unfortunately, data were not available at the Fort Cobb site 1-through 14-days' posttreatment because of equipment failure. However, the differences between pretreatment and 28-and 56-day posttreatment data at Fort Cobb were minimal.

The overall physical-chemical environmental conditions at the two reservoirs were quite different during the study. Banks Lake was treated earlier in the year than Fort Cobb Reservoir and, therefore, water temperatures were much cooler. Banks Lake functions as a water equalizing reservoir and, thus, has more flow-through characteristics than the Oklahoma site which stores mainly summer surface runoff precipitation. This flushing would cause mineral content to be lower at Banks Lake. Hydrogen ion (pH) characteristics follow conductivity, resulting in Banks Lake having a lower pH than Fort Cobb. Low dissolved oxygen is one of the indicators of environmental stress which could result from the herbicidal treatments. Decomposing macrophytes killed by 2,4-D might be expected to reduce DO values, thus causing some undesirable effects on aquatic animals. DO was at or above saturation in Banks Lake. Aquatic weed growths at this study site were minimal. This fact coupled with cooler water temperatures and a continuous flow-through pattern of water being delivered for irrigation purposes would have contributed to this condition.

Fort Cobb Reservoir treatments were made in shallow back bay areas which may have had some influence in producing lower DO values shown at the 0-day sample point. Lack of data during the more critical periods of 1-through 14-days' posttreatment prevented an assessment of any adverse influence of herbicidal treatment on this parameter. Observations of heavy eurasian watermilfoil infestations made during and after the treatments did not indicate any undesirable effects on the fish populations of Fort Cobb. Also, plankton data reported previously did not indicate any adverse conditions and it is unlikely that DO stress occurred.

The results of water chemical analyses are summarized for the study period at Banks Lake (table 26). Individual data collected at each herbicide treatment site and at the outlet throughout each sampling time are presented in the appendix of this report for each geographical site. These data would illustrate any change during the study period occurring from the treatment as well as normal seasonal shifts. Examination of these data throughout the study at both geographical locations did not indicate any significant changes in total water chemistry or ionic composition which could be interpreted as being directly related to herbicidal treatment of test plots.

Water quality data obtained during these studies at both Banks Lake and Fort Cobb Reservoir do not indicate that any undesirable effects occurred as a result of the experimental herbicide treatments. The treatments made on plots at Banks Lake would not have been expected to have any significant influence on the water quality because of their exposure to the general flow patterns and being in the general open body of the main reservoir. At Fort Cobb Reservoir, treatment of cove areas not as well exposed to the main body of the reservoir and effects of herbicidal treatments on heavy weed growth observed might indicate some environmental stress on the chemical-physical environment. However, measurements did not indicate that this occurred. These studies suggest that 2,4-D treatments of this size and nature did not have any measurable undesirable effects on water quality through the 28- to 56-day period. Pesticides are biological poisons and materials like 2,4-D additions to an aquatic ecosystem must have had some influence on that system. Obviously, destruction of the macrophyte population is going to have some immediate adverse effect; however, this study of water quality did not indicate any significant influence on chemical-physical characteristics of water quality. The amount of organic additions (2,4-D formulations) and the herbicide's characteristics apparently was not of sufficient magnitude or toxicity to overcome the reservoir's ecosystem resilience for absorbing and stabilizing such materials.

SUMMARY AND CONCLUSIONS

2,4-D Residues in Water

The temporary potable water tolerances of 0.1 mg/L (p/m) established under the EUP was exceeded in only a few of individual water samples during the course of these studies. Data from the studies support the previously established EPA potable water tolerances of 0.1 mg/L 2,4-D and water quality criteria [8, 11] and the raw agricultural commodities tolerance of 0.1 mg/L 2,4-D [7] resulting from ditchbank weed control herbicide applications. Residues in this study were also well below the

Table 26.—*Chemical analyses of water samples collected at Banks Lake, Washington—summary of replicated pretreatment and posttreatment samples from 0- through 28-days.*

Water quality parameter	Sample locations				
	Outlet	Plot 1	Plot 2	Plot 3	Plot 4
Conductivity, μm/c	126.1	129.2	115.2	113.6	113.2
pH	8.3	8.3	8.3	8.2	8.4
Total dissolved solids, 105 °C	92.0	64.1	79.7	59.3	68.1
Calcium, mg/L	16.5	17.7	18.6	15.6	18.0
Magnesium, mg/L	3.1	3.9	2.0	4.2	2.3
Sodium, mg/L	2.4	2.3	2.2	2.1	2.1
Potassium, mg/L	0.9	0.8	0.8	0.8	0.9
Carbonate, mg/L	0.0	0.2	1.8	0.8	2.7
Bicarbonate, mg/L	46.0	57.2	48.4	51.2	45.1
Sulfate, mg/L	17.5	17.1	16.2	12.0	16.8
Chloride, mg/L	0.7	0.7	0.7	0.7	0.7
Anion and cation, mg/L	89.0	100.0	91.1	91.5	88.7

established tolerances for raw agricultural commodities when residues result from water hyacinth control and eurasion watermilfoil control [7, 12, 13]. The three instances that exceeded the 0.1 mg/L value occurred during the 1-day posttreatment sampling, where an average water surface value of 0.199 mg/L was determined within the 22.5 kg/ha (20-lb/ acre) DMA plot at Banks Lake, while 0.183 and 0.197 mg/L average surface levels were found outside the 45 kg/ha (40-lb/acre) BEE plot at Banks Lake and within the 45 kg/ha (40-lb/ acre) DMA plot at Fort Cobb Reservoir, respectively. However, it should be emphasized that this occurred in 3 of 1680 water samples analyzed or 0.18 percent of the total. All herbicidal treatments at both reservoirs exhibited rapid 1- to 14-day dissipation rates with trace levels of 2 to 3 μg/L (p/b) being reached by 14 days' posttreatment.

A comparison of surface to near bottom collected water samples showed similar trends in herbicide residue levels, but with more variability being seen in the near bottom samples. The dimethylamine formulation showed some tendency to sink more slowly throughout the water column than did the ester formulation.

Statistical computation of water residue data disclosed a smooth decay curve from the 1-through 14-day posttreatment period. Concentrations of 2,4-D were generally zero or of only trace quantities after 14 days. Tests for curve fits resulted in good correlation with R^2 values ranging from 0.49 to 0.96. Most of the coefficients were in the 0.80 to 0.90 range, suggesting that these data might be useful for herbicide dissipation predictions. Dispersion throughout the water column seemed to be the predominant factor in herbicide dissipation. Outward movement from the treated plot also seemed to be a result of local dispersion factors. Convection, reservoir turnover, or wind currents seemed to have little or no influence in these studies.

One of the more critical findings in the study of herbicide residues in water was reflected in sampling at the reservoir outlets, which in all instances were over 0.75 km (0.50 mi) from any treated area. Only trace amounts of 2,4-D were found at either reservoir's outlet works. The greatest concentrations found in our studies were 7.6 μg/L at Banks Lake and 9.9 μg/L at Fort Cobb Reservoir. All the values are one magnitude below the 0.1 mg/L temporary tolerance.

Because water from Fort Cobb Reservoir was being routinely used for potable purposes, the Oklahoma Health Department continually monitored outlet residue levels. The only value to cause any concern was 62.0 μg/L. Health department officials felt that this was very likely an anomaly resulting from contamination during sampling or handling.

Herbicide Residues in the Hydrosoil

Generally, low residue values were found in the hydrosoils. In DMA-treated areas, concentrations of 2,4-D ranged from 0 to 0.241 μg/g at Banks Lake and 0 to 0.316 μg/g at Fort Cobb. The maximum values were generally 1 day after treatment. The ester formulation produced the highest residue, as reflected in a value of 37 μg/g resulting from a 45-kg/ha (40-lb/acre) application at Banks Lake. Fort Cobb Reservoir residue levels were routinely much lower with a maximum of 7.6 μg/g

resulting from a 45-kg/ha BEE application. It is likely that the more eutrophic soils, heavier infestations of eurasian watermilfoil, and warmer temperatures at Fort Cobb provided more absorption sites and an environment for more rapid microbiological decomposition than occurred at Banks Lake.

Soil residue dissipation curves in a few instances showed some concentrations increasing from 0- to 28-days following herbicide application, followed by a decline to trace or zero amounts by 28- to 56-days, depending on site and rate of application. There was some suggestion of a 7-day posttreatment herbicide residue level increase possibly resulting from a rerelease of herbicide from the BEE pelleted formulation. This could have been due to a delayed physical decomposition of the clay carrier or from the pellet surface. These 7-day increases were not reflected in the water residue data.

2,4-D Decomposition Product Monitoring

Trace amounts of 2,4-D dichlorophenol were occasionally found in hydrosoils, predominantly in plots treated with the ester formulation. These residues were in the μg/L range. Some pretreatment soil samples were found to contain traces of the dichlorophenols from a contamination source other than these herbicide treatments. No dichlorophenols were found in water samples, so they were likely bound to soil colloids.

Dimethylnitrosamine in the μg/L range was found in a few water samples from both sites. Pretreatment samples also were found to have nitrosamines. It is not likely that these trace levels were related to herbicide treatments, but were naturally occurring from organic sources other than the herbicides.

None of the decomposition products was of other than a trace amount and would not be considered of significant environmental concern. They are, in all probability, not the result of 2,4-D and its formulation decomposition.

Fish Flesh Residues of 2,4-D

Residues found in fish fillet samples, considered to be the portion most representative of food for human consumption, were well within the established food additive tolerance of 1.0 p/m [7, 13, 14]. No evidence of bioaccumulation was noted during the course of these studies. This was confirmed by similar observations by Tennessee Valley Authority in their eurasian watermilfoil control program [16].

Effects of 2,4-D on Plankton Populations

Plankton populations representing both phytoplankton and invertebrate organism populations showed little or no discernible adverse effect from exposure to the 2,4-D treatments. Measurements of organism abundance, composition, and species diversity in the water columns treated with herbicide were not significantly different from nontreated check areas on the reservoirs. Robinson and Morley made similar observations on nontarget organisms including phytoplankton when BEE was used for eurasian watermilfoil control [15].

Effect of Treatments on Water Quality

The two reservoirs were considerably different in their physical-chemical composition. Fort Cobb is more representative of a eutrophic

Table 27.—*Chemical analyses of water samples collected at Fort Cobb Reservoir, Oklahoma—summary of replicated pretreatment and posttreatment samples from 0- through 56-days.*

Water quality parameter	Sample locations				
	Outlet	Plot 1	Plot 2	Plot 3	Plot 4
Conductivity, μm/c	382	424	425	450	366
pH	8.0	8.0	8.0	8.0	8.0
TDS, 105 °C	270	279	292	318	231
Calcium, mg/L	31.5	29.2	33.8	31.3	29.2
Magnesium, mg/L	17.3	19.9	18.5	21.0	18.5
Sodium, mg/L	20.5	30.5	26.5	30.3	27.6
Potassium, mg/L	6.2	8.4	8.3	8.9	7.8
Carbonate, mg/L	0.0	0.0	0.1	0.0	0.0
Bicarbonate, mg/L	81.7	81.6	94.3	99.0	84.3
Sulfate, mg/L	109.3	127.0	121.6	132.0	118.2
Chloride, mg/L	12.4	13.6	12.8	13.9	12.1
Anion and cation, mg/L	279.0	313.0	316.0	336.5	297.0

habitat and Banks Lake an oligotrophic or mesotrophic aquatic environment. The study data associated with these parameters suggest that none of the 2,4-D treatments at either reservoir significantly affected water quality. Both reservoirs showed considerable resilience to the herbicide exposure and conditions remained quite stable during the period of study.

In conclusion, the data resulting from these studies support the potential environmentally safe use of DMA or BEE for controlling eurasian watermilfoil in reservoirs when proper precautions in treatment and usage are employed. The established tolerances for potable water and irrigation water use were not exceeded at the reservoir outlets. No significant amounts of herbicide residue were persistent in the hydrosoils with only trace concentrations being detected. Potential carcinogens such as dichlorophenol or nitrosoamine were found only in extremely low trace concentrations and where they occurred, in pretreatment samples, were probably natural contaminants. The fish flesh residues found were in all cases within the established food additive tolerance. Plankton indicator organisms showed little or no effects from the herbicide exposure. Water quality was not altered to any extent based on the physical-chemical measurements made throughout the study period.

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APPENDIX A

Attachment 1	Protocol for 2,4-D Residue Dissipation Studies for Experimental Use Permits Numbered 11683-EUP-2 and 11683-EUP-3.
Attachment 2	General Protocol for Analysis of DMA (2,4-D Dimethylamine), 2,4-Dichlorophenol, BEE (2,4-D Butoxy Ethanol Ester), and Dimethylnitrosamine.
Attachment 3	Protocol for Extraction and Analysis of DMA (2,4-D Dimethylamine) 2,4-Dichlorophenol, and BEE (2,4-D Butoxy Ethanol Ester) from Water Using HPLC (High Pressure Liquid Chromatography).
Attachment 4	Protocol for Extraction and Analysis of Dimethylnitrosamine from Water Using HPLC (High Pressure Liquid Chromatography)
Attachment 5	Protocol for Extraction and Analysis of DMA (2,4-D Dimethylamine), 2,4-Dichrophenol, and BEE (2,4-D Butoxy Ethanol Ester) from Hydrosoil Using HPLC (High Pressure Liquid Chromatography)
Attachment 6	GC/MS (Gas Chromatography/Mass Spectrometry) Confirmation
Attachment 7	Protocol for Fish Collections for 2,4-D Residue Analyses
Attachment 8	Extraction and Analysis of BEE (2,4-D Butoxy Ethanol Ester) and DMA (2,4-D Dimethylamine) from Fish Tissue by Gas Chromatography
Attachment 9	Protocol for Monitoring Aquatic Invertebrates
Attachment 10	Supplemental Labeling for Experimental Use with Aqua-Kleen^{R}-EPA Registration No. 254-109
Attachment 11	Supplemental Labeling for Experimental Use with Weedar 64^{R} Herbicide-EPA Registration 254-2

Attachment 1

PROTOCOL FOR 2,4-D RESIDUE DISSIPATION STUDIES FOR EXPERIMENTAL USE PERMITS NUMBERED 11683-EUP-2 AND 11683-EUP-3

1. Purpose of Study

Information is needed on the dissipation characteristics of BEE (2,4-D Butoxy Ethanol Ester) and DMA (2,4-D Dimethyamine Salt) when applied for control of eurasian watermilfoil in western reservoirs. This study is designed to obtain pretreatment (baseline) and posttreatment 2,4-D residue levels in treated and untreated portions of water bodies representing typical geographic and climatological conditions in the United States. In addition, responses of existing aquatic weeds (primarily eurasian watermilfoil) will be determined. The following procedures are designed to be applicable to any reservoir or lake. However, specific plans and timetables must be developed for each test location in order to accommodate the size, hydrologic characteristics, water-use and climatological conditions.

Definition of sample collection terminology.

- Geographic Location(s):

  Ft. Cobb Reservoir, Oklahoma (Bureau of Reclamation) Banks Lake, Washington (Bureau of Reclamation) Lake Seminole, Florida-Georgia (Corps of Engineers)

  - Plots: Located within geographic location, and comprised of 10 to 16 ha (25 to 40 acres)

  - Stations: Located within and outside of plots

  - Sites: Sample collection points located around stations

2. Application (Note: All applications will be made under the supervision of a Certified Pesticide Applicator.)

A. Plot Selection

Select suitable plots that are known to support stands of eurasian watermilfoil and mark with anchored, floating buoys. (Note: Size must conform to EPA (US Environmental Protection Agency) regulations for experimental plots or to "conditional label", if appropriate.) Size will range from 10 to 16 ha (25 to 40 acres) in total. If possible, plots must be no less than 0.8 km (one-half mi) from any potable, agricultural or industrial water outlet. Each plot to be treated will vary according to local conditions but preferably select plots with similar weed distribution.

B. Rates/Methods of Application

(1) Two rates of each formulation are to be applied: 22 .5 and 45 kg/ha, ae (20 and 40 lb/acre, ae) providing a total of four treatments per geographic location.

(2) If possible, each plot must be at least 0.8 km (½ mi) from adjacent treated plots.

(3) Follow product label and supplemental directions for method of application.

C. Data to be recorded (See fig. A-1)

(1) Geographic location (reservoir, lake)

(2) Plots (Indicate size and location of plots on a reservoir map)

(3) Date, time, weather conditions at time of treatment, water temperature, air temperature

(4) Herbicide application rates, formulation, manufacturer's batch number, method of application, duration of application.

(5) Distance of sampling stations from treated plots (See fig. A-1)

3. Sampling

A. Sampling Station Locations

(1) Within treated plots: Select and mark with buoys two (2) stations, one in each half of the plot.

(2) Outside treated plots: Select three (3) stations 90 to 180 m (100 to 200 yds) (preferably the more distant) from the midpoint of three sides of each plot and mark with a buoy.

(3) Sample water at reservoir outlets to multiple-use systems to determine background 2,4-D levels. Sample as close to outlets as practical.

a. Triplicate middepth water samples are to be taken.

b. Samples are to be taken on same days as those in treated and outside the treated area.

B. Sampling Method

(1) Water—(Attachments 3 and 4)

a. Triplicate 1-liter samples are to be taken from 0.3 m (1 ft) below the surface and at the bottom for both BEE and DMA treatments using an appropriate water sampling device (Kemmerer/Van Dorn Sampler, 12 volt Jabsco Water Puppy pump, or similar instrument). Sample two stations within the treated plots and three stations adjacent to the treated plots. Triplicate middepth samples shall be taken within 0.8 km (½ mi) of the outlets to multiple-use systems (See fig. A-1).

b. Sampling Schedule—One day pretreatment water samples are to be taken. After treatment, the following schedule is used:

1 day posttreatment
4 day
7 day
14 day
28 day
56 day
More as needed (To determine by residue analysis)

Summary of required number of samples from each BEE plot at each sampling time: 6 samples per station per collection day

6 × 5 stations = 30 samples/plot/collection day
30 × 2 plots = 60 samples/geographic location/collection day
60 × 7 sampling dates = 420 samples/geographic location
420 × 2 geographic locations = 840 samples total

Summary of required samples from each DMA plot at each sampling time: 6 samples per station per collection day

6 × 5 stations = 30 samples/plot/collection day
30 × 2 plots = 60 samples/geographic location/collection day
60 × 7 sampling dates = 420 samples/geographic location
420 × 2 geographic locations = 840 samples total

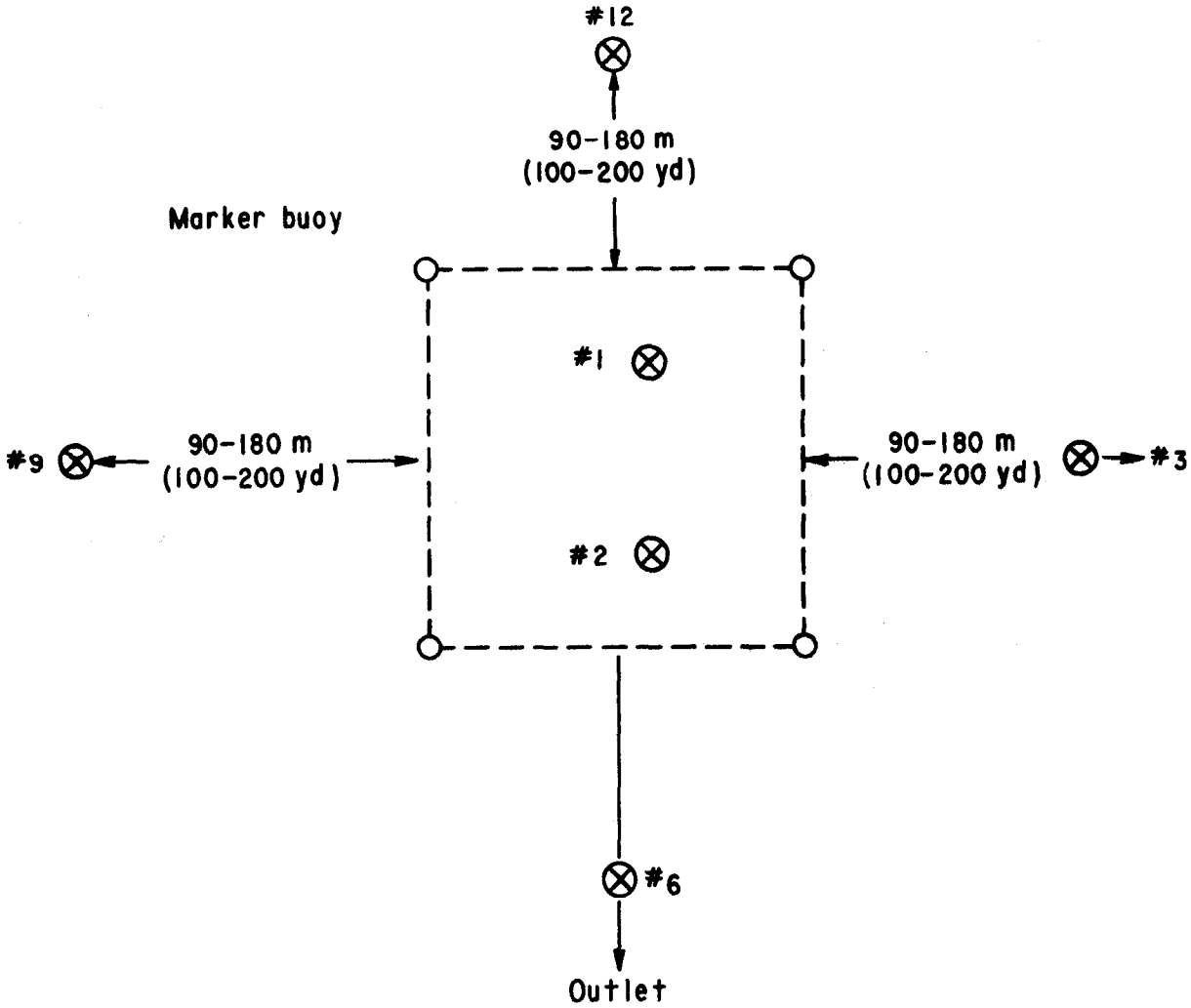


Figure A-1.
2,4-D PLOT DIAGRAM

Water Sampling Identification Codes:

a. Inside stations – Label #1, #2
b. Outside stations – Label #3, #6, #9, #12 in clockwise order, with 6 o'clock oriented toward *nearest water outlet*

Example of Label for Water and Hydrosoil –

Site: Banks Lake
Date treated: 26 June 1979
Rates/Formulation: 22.5 kg/ha (20 lb/acre) BEE
Date Sampled: 27 June 1979
I.D. #1 (within treated plot)

Note: Outside stations at Banks Lake were labeled according to fig. A-1; however, outside stations at Fort Cobb Reservoir were labeled differently due to the size of the plots which encompassed the treated coves. Outside stations at plot 1 and plot 2 at Fort Cobb were labeled 1:00, 3:00, and 5:00 while outside stations at plot 3 and plot 4 were labeled 10:00, 8:00, and 6:00. Stations at 5:00 and 6:00 were oriented closest to nearest water outlet.

Summary of required samples from the outlet sampling stations at each sampling time: 3 samples per station per collection day

3 × 1 station = 3 samples/outlet station/collection day
3 × 7 sampling dates = 21 samples/outlet station
21 × 2 geographic locations = 42 samples total

(2) Hydrosoil—(Attachments 5 and 6)

a. Sampling Sites—Use the two (2) within-plot water sampling stations as reference points. Using a 9- to 18-m (10- to 20-yd) radius from each marker, locate two random sites around each marker.

b. Method—An appropriate sampling device (Ekman dredge, pipe dredge, or similar instrument) will be used to obtain hydrosoil samples. Place in plastic bags and freeze as soon as possible. Ship to analytical lab as with water samples.

c. Sampling Schedule—One day pretreatment sample will be taken, as will 4, 7, 28, 56, and 90 days posttreatment samples.

Summary of required number of hydrosoil samples at each sampling time: 2 samples per station per collection day

2 × 2 stations = 4 samples/plot/collection day
4 × 4 plots = 16 samples/location/collection day
16 × 6 sampling dates = 96 samples/location
96 × 2 geographic locations = 192 samples total

(3) Ecological Effects—(Attachments 7 and 8)

a. Fish Flesh Residue: See attached protocol for fish collection and analysis for DMA and BEE

b. Aquatic Invertebrate Populations: See attachment 9 for study protocol

(4) All water and hydrosoil samples collected by Bureau of Reclamation will be shipped air freight to USBR Denver Lab (use dry ice for all except chemically preserved biological samples.) Shipping containers and sample bottles will be supplied by the Denver Laboratory. Notify the Denver laboratory of shipment date, flight number, and airline. Fish samples will be frozen and shipped air freight to laboratory sites to be determined. Samples collected by Corps of Engineers are to be processed as determined by that agency. Air freight shipment of samples containing dry ice requires appropriate labeling and approval from each airline handling the samples.

4. Efficacy evaluation

A. Pretreatment

(1) After plot is marked, the approximate percent coverage and composition of aquatic weed communities should be determined by visual observation and recorded.

B. Posttreatment

(1) The approximate percent coverage and composition of aquatic weed communities as related to the pretreatment weed population determined as in paragraph IV.A.(1) above, should be evaluated 7 days, 28 days, and 56 days posttreatment.

C. Control (untreated) Sites

An untreated plot should be selected as far from the treated plots as possible (at least 0.4 km (¼ mi) away) with *about the same aquatic plant composition*. Observations of approximate percent coverage and composition of aquatic weed communities should be made as with treated sites, and photos should be taken.

5. Water Quality

Dissolved oxygen, pH, conductivity, hardness, and temperature will be determined at each sampling site and interval with analytical equipment supplied and operated by USBR Denver laboratory personnel. The Denver laboratory will provide guidance on methods and equipment for physical/chemical analyses and will periodically conduct total water quality analysis.

6. Extraction and Analysis of DMA, BEE, 2,4-Dichlorophenol and Dimethylnitrosamine in water and DMA and BEE in hydrosoil and fish flesh. (See attached protocols and example chromatograms.)

7. Example of plot layout and sample label: See Figure A-1.

Attachment 2

GENERAL PROTOCOL FOR ANALYSIS OF DMA, 2,4-DICHLOROPHENOL, BEE, AND DIMETHYLNITROSAMINE

Waters Sep-Pak C_{18}^{R} cartridges will be used for analysis of DMA (2,4-D Dimethylamine) 2,4-Dichlorophenol, BEE (2,4-D Butoxy Ethanol Ester) and dimethylnitrosamine using High Pressure Liquid Chromatography.

Analysis of the four components will be accomplished in two phases:

1. Phase I—Clean up and concentration of DMA, 2,4-Dichlorophenol and BEE.
   A. Trace enrichment from water and elution from precolumn (Sep-Pak C_{18}^{R}) cartridge.
   B. Isocratic elution (accomplished in minutes).
2. Phase II—Partitioning and concentration of dimethylnitrosamine
   A. Partitioning eluant from Phase I or fresh sample.
   B. Concentrate dimethylnitrosamine by evaporation.
   C. Isocratic elution.

Samples will be analyzed utilizing Waters Associates Radial Compression Module with a 10 cm × 8 mm i.d., 10μ, μBondapak C_{18}^{R} column.

Solvent systems will be delivered by two constant flow, nonpulsed, high pressure pumps (Waters Associates Model 6000). A model U6K universal injector with a Waters Model 440 U.V. detector will be employed. Gradient programming will be controlled by a Waters Model 660 solvent programmer. A 280 nm filter will be used for DMA, 2,4-Dichlorophenol, and BEE analysis while a 254-nm filter will be used for the dimethylnitrosamine analysis. Chromatograms will be recorded using a Hewlett-Packard Model 3885 integrator. Gradient programming will be controlled by a Waters 660 solvent programmer.

Attachment 3

PROTOCOL FOR EXTRACTION AND ANALYSIS OF DMA (2,4-D DIMETHYLAMINE), 2,4-DICHLOROPHENOL, AND BEE (2,4-D BUTOXY ETHANOL ESTER) FROM WATER USING HPLC (HIGH PRESSURE LIQUID CHROMATOGRAPHY)

1. Shake samples thoroughly.

2. Pour 250-mL samples into a 500-mL Erlenmeyer flask.

3. Samples taken from plots treated with DMA are treated as follows:

   A. Lower pH to approximately 2.25 to 2.50 with 5N sulfuric acid (H_2SO_4).

   B. Mix well for 1 minute.*

4. Samples taken from plots treated with BEE and outlet samples are treated as follows:

   A. Adjust pH to approximately 12.25 to 12.50 using 10N sodium hydroxide (NaOH).

   B. Stir well and shake for 30 minutes on a mechanical shaker.

   C. Lower pH to approximately 2.20 to 2.50 with 10N hydrochloric acid (HCL).**

5. Filter and concentrate samples through the following described aparatus:

   A. Vacuum filter samples through a Buchner funnel (4.7 cm-diameter) containing a No. 1 Whatman filter. Tapered end of Buchner funnel is passed through a No. 4 rubber stopper which is fitted onto a 30 cm^3 syringe. Luered end of syringe fits into short end of Sep-Pak C_{18}^R cartridge. Long end of Sep-Pak fits into glass tubing extending through a No. 8 stopper fitting onto a 2-liter Erlenmeyer flask.

   B. Sep-Pak C_{18}^R should be preconditioned with 10 mL of acetonitrile (CH_3CN) followed by 10 mL of distilled water.

   C. Vacuum during filtering and concentrating should be approximately 10 to 15 cm Hg for 30 to 40 minutes.

6. 2,4-D is then eluted from Sep-Pak using 2 mL of 100% acetonitrile (CH_3CN) as follows:

   Use 1 mL of 100% acetonitrile to rinse off sides of Buchner funnel and filter paper. Use remaining 1 mL to rinse off sides of glass syringe.

7. Catch 2,4-D in a 15-mL centrifuge tube for HPLC analysis. Final volume should be approximately 2 to 2.5 mL.

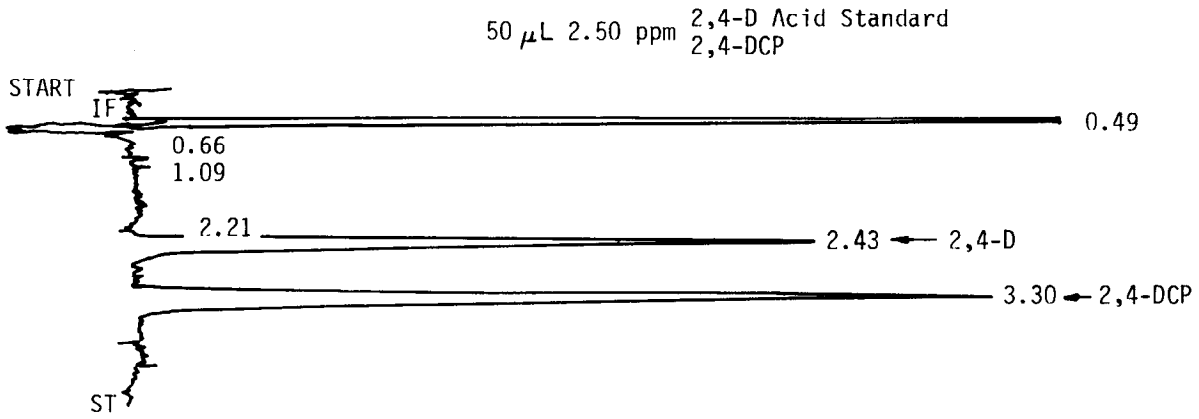
8. Analyze DMA, BEE, and 2,4-Dichlorophenol with HPLC using an isocratic system consisting of: 40% Acetonitrile/60 percent H_2O (1 percent acetic acid) at 2.5 mL/min.

Anticipated Recovery Levels

* Recovery of DMA 90-95 percent

** Recovery of BEE 80-85 percent

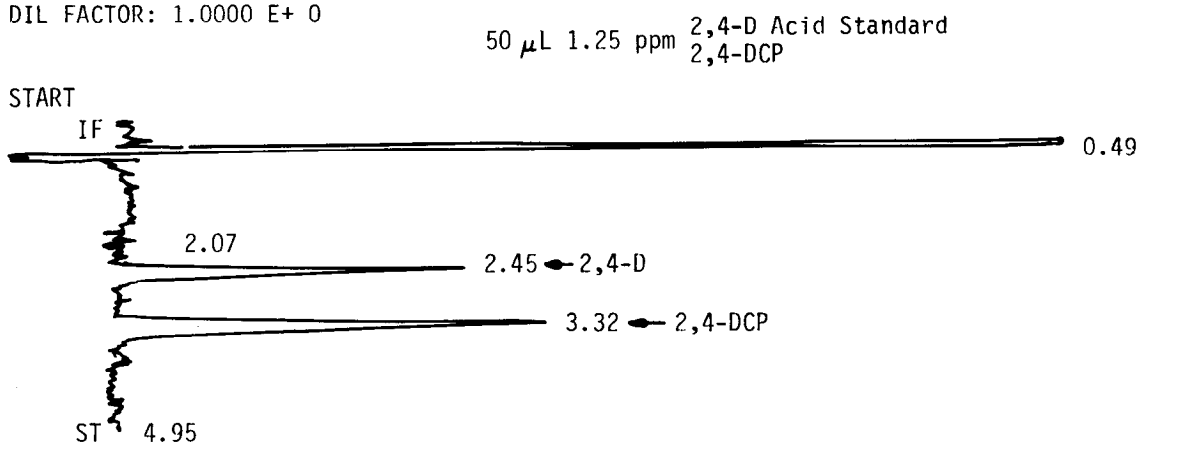
Recovery of 2,4-Dichlorophenol 75 to 80 percent for DMA and BEE samples spiked with 2,4-Dichlorophenol



HP RUN # 21 SEP/08/81 TIME 13:15:51
ID: 40-60-2.5
ESTD

RT	EXP RT	AREA	CAL #	AMT
2.43	2.40	12250	(R) 1	124.390
3.30	3.32	18570	2	129.390

DIL FACTOR: 1.0000 E+ 0



HP RUN # 22 SEP/08/81 TIME 13:25:27
ID: 40-60-2.5
ESTD

RT	EXP RT	AREA	CAL #	AMT
2.45	2.40	5973	(R) 1	60.652
3.32	3.34	9116	2	63.517

Figure A-2.—Standard HPLC.

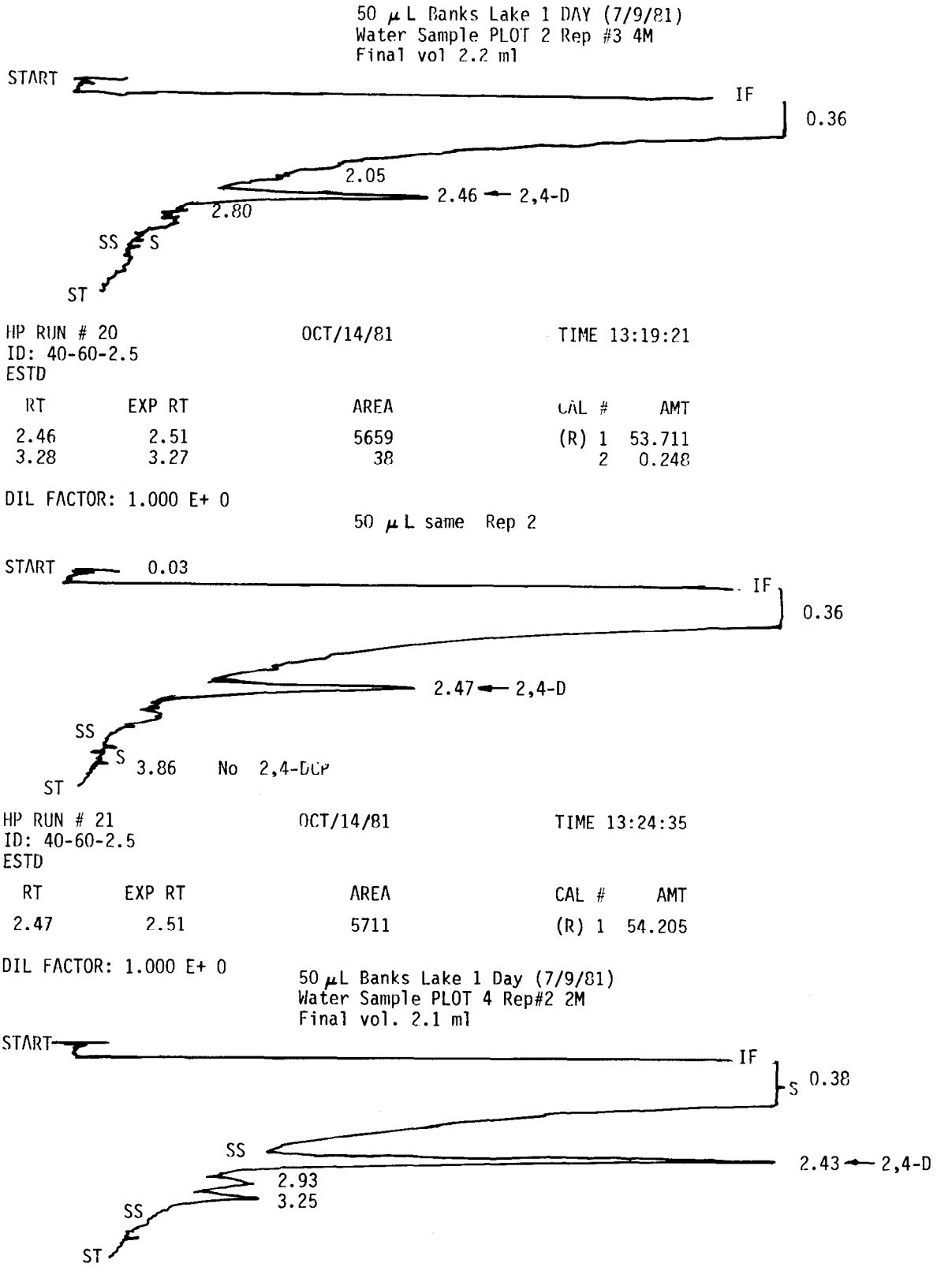


Figure A-3.—Banks Lake Water Sample HPLC.

Attachment 4

PROTOCOL FOR EXTRACTION AND ANALYSIS OF DIMETHYLNITROSAMINE FROM WATER USING HPLC (HIGH PRESSURE LIQUID CHROMATOGRAPHY)

1. Shake sample thoroughly.

2. Pour 250-mL sample into a 500-mL Erlenmeyer flask.

3. Extract water two times with 50 mL of methylene chloride.

4. Pool both methylene chloride extracts into a 150-mL beaker and evaporate down to approximately 5 mL using a warm water bath.

5. Transfer to a 15-mL centrifuge tube using an additional 5 mL of methylene chloride to rinse 150-mL beaker.

6. Evaporate methylene chloride down to approximately 0.5 mL and then add 1 mL of acetonitrile (CH_3CN).

7. Evaporate down to 0.5 mL and then bring up to 2 mL with 50 percent acetonitrile/50 percent distilled water.

8. Analyze for dimethylnitrosamine using an isocratic system consisting of:* 10 percent acetonitrile/90 percent water at 1.0 mL/min.

*Anticipated Recovery levels
40-50 percent Recovery of Dimethylnitrosamine from water.

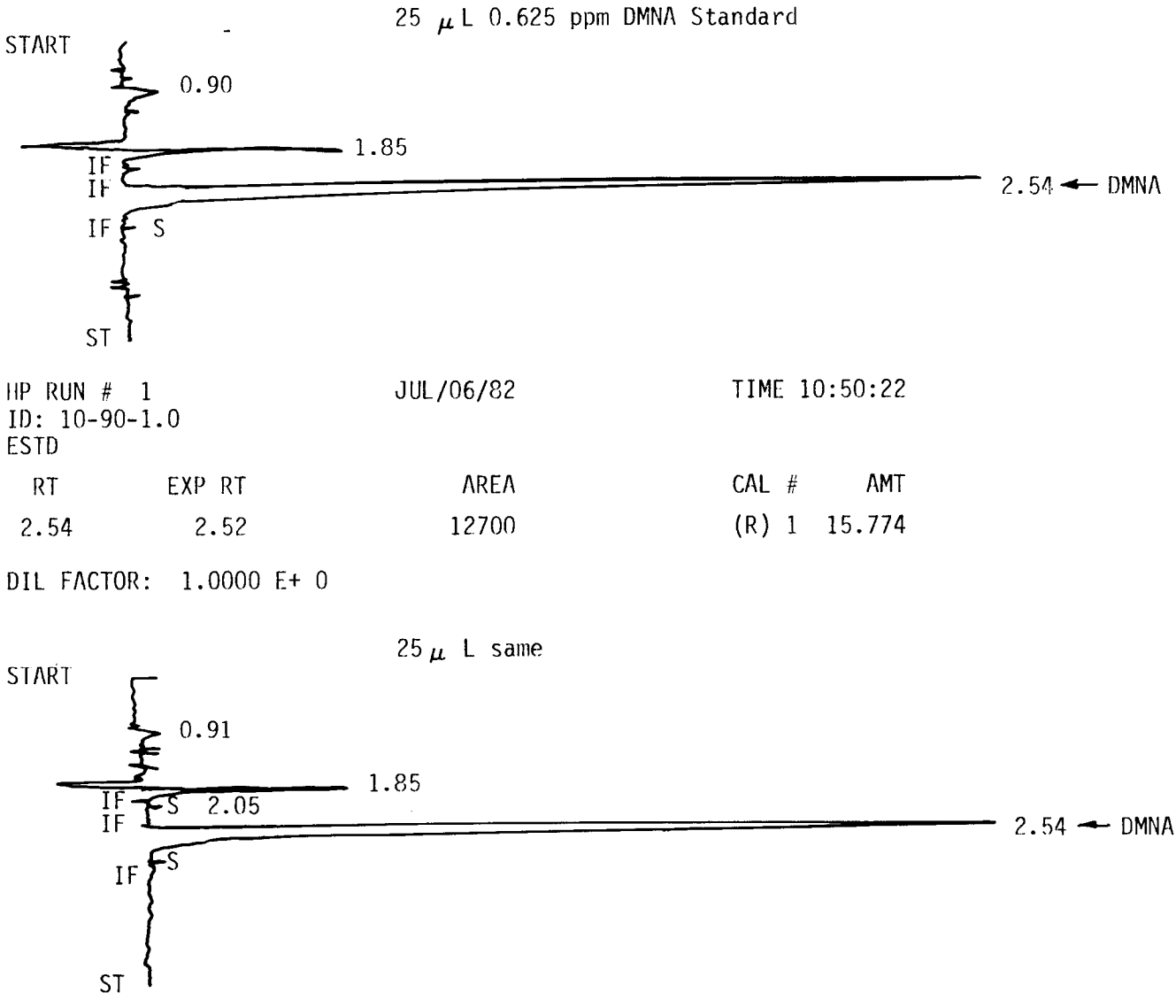
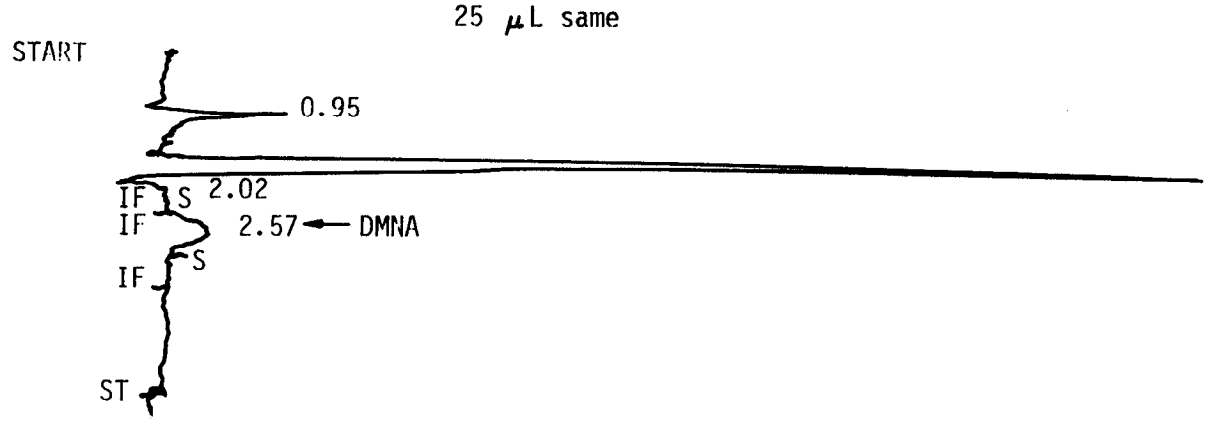


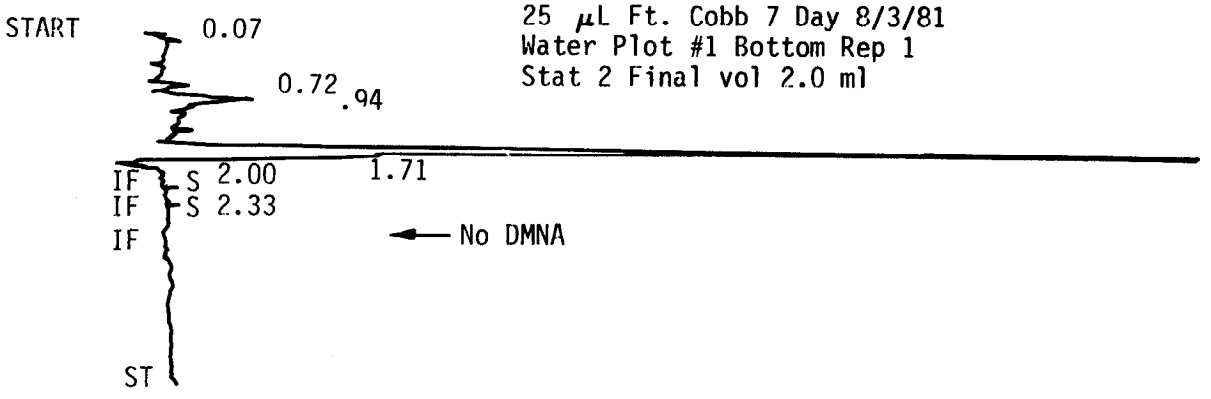
Figure A-4.—Standard HPLC.



HP RUN # 24 JUL/06/82 TIME 14:37:34
ID: 10-90-1.0
ESTD

RT	EXP RT	AREA	CAL #	AMT
2.57	2.54	1313	(R) 1	1.631

DIL FACTOR: 1.000 E+ 0



HP RUN # 25 JUL/06/82 TIME 15:11:55
ID: 10-90-1.0
NO PEAKS IN WDOS

Figure A-5.—Ft. Cobb Res. Water Sample HPLC.

PROTOCOL FOR EXTRACTION AND ANALYSIS OF DMA (2,4-D DIMETHYLAMINE), 2,4-DICHLOROPHENOL, AND BEE (2,4-D BUTOXY ETHANOL ESTER) FROM HYDROSOIL USING HPLC (HIGH PRESSURE LIQUID CHROMATOGRAPHY)

1. Air dry hydrosoil for 72 hours.

2. Grind the soil in a mortar and pestle, throughly mix, screen through a 20-mesh sieve, and weigh out a 20-gram subsample.

3. Add the soil sample to a 100-mL beaker and slurry with 15 mL distilled water and allow to equilibrate for 15 minutes.

4. Extract the sample with 50 mL methanol using an ultrasonic probe and sonify 1 minute at 50 watts.

5. After initial sonification, wait ½ hour for soil to sediment before decanting the solvent and suction filtering into a 1000-mL Erlenmeyer flask.

6. Suction filtrate using a 7-cm Buchner funnel containing a 7-cm No. 50 Whatman filter.

7. Re-extract the remaining sediment with another 50 mL of methanol using the same sonification process. Suction filter this extract combining it with the first 50 mL of methanol. Rinse out beaker and Buchner funnel two times with 10 mL methanol and add to 100-mL methanol extract. Transfer methanol extract to a 500-mL flat-bottom boiling flask.

8. Reduce sample extract to 5 to 10 mL by rotary evaporation.

9. Add 100 mL distilled H_2O.

10. Lower pH to 1.5 using 18N H_2SO_4 and pass through a Sep-Pak cartridge to concentrate low polarity organics including 2,4-D components. Use a No. 2 Whatman filter during this step to prevent small particulates from plugging Sep-Pak C_{18}^R .

11. Elute the 2,4-D components from the Sep-Pak C_{18}^R cartridge with 3 mL 100 percent acetonitrile.* HPLC analysis requires 25-mL aliquots of this acetonitrile extract.

Extraction of BEE and 2,4-Dichlorophenol From Soil

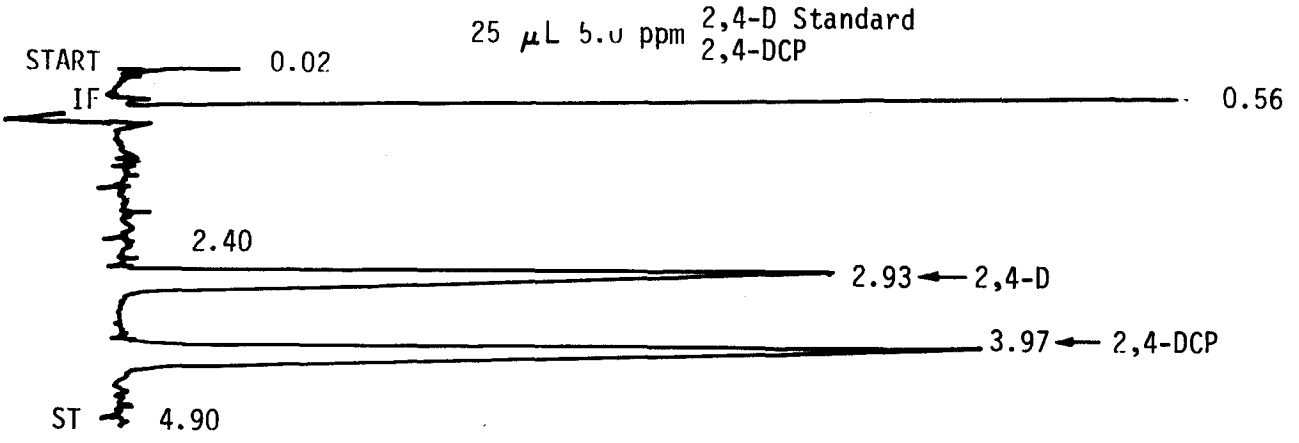
9. Extraction is the same through step 9 with one exception: Add 100 mL 0.2N sodium hydroxide.

10. Shake for ½ hour using mechanical shaker.

11. Lower pH to 1.5 using 18N H_2SO_4 and pass through a Sep-Pak C_{18}^R cartridge with 3 mL 100 percent acetonitrile.**

13. Analyze both DMA, BEE, and 2,4-Dichlorophenol with HPLC using an isocratic system consisting of: (1) 50 percent Acetonitrile/50 percent H_2O (1 percent acetic acid) at 1.5 mL/min, *OR* (2) 40 percent Acetonitrile/60 percent H_2O (1 percent acetic acid) at 2.5 mL/min.

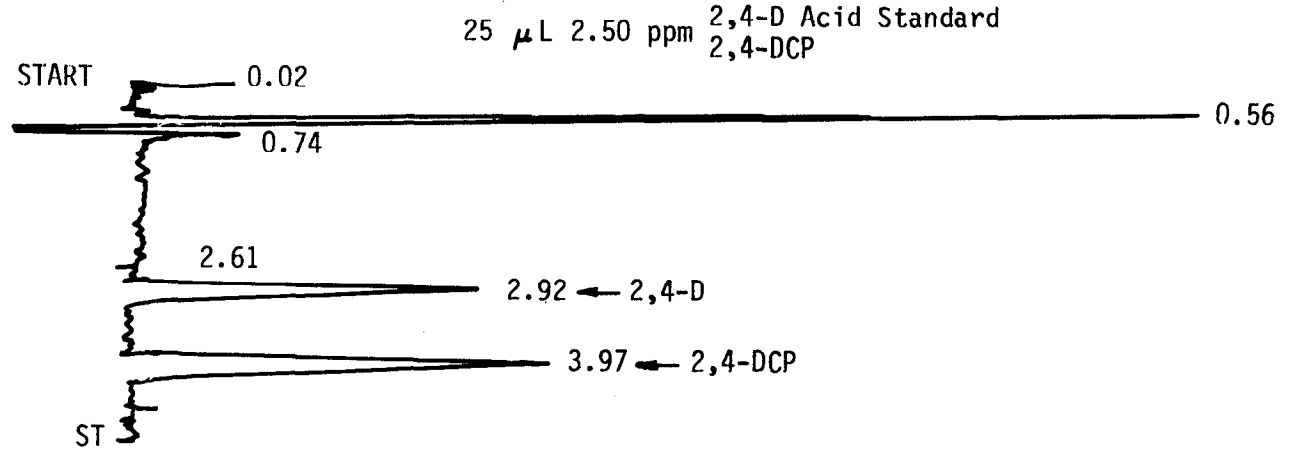
Anticipated Recovery Levels
* Recovery of DMA 80 to 85 percent
** Recovery of BEE 80 to 85 percent
Recovery of 2,4-D Dichlorophenol 60 to 65 percent for DMA



HP RUN # 4 MAY/14/82 TIME 10:52:16
ID: 40-60-2.5
ESTD

RT	EXP RT	AREA	CAL #	AMT
2.93	2.93	12640	(R) 1	123.728
3.97	3.97	18680	2	126.216

DIL FACTOR: 1.000 E+

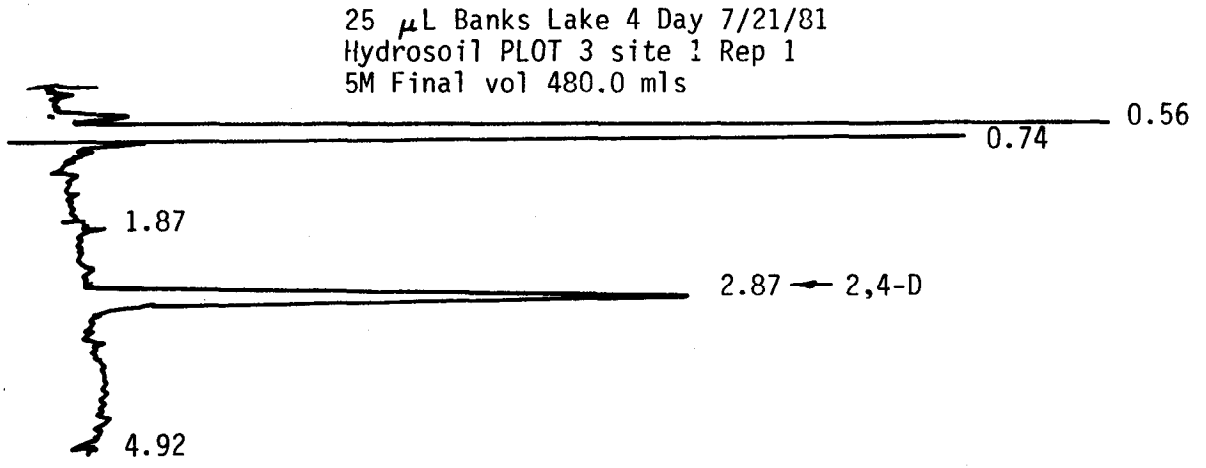


HP RUN # 5 MAY/14/82 TIME 11:00:30
ID: 40-60-2.5
ESTD

RT	EXP RT	AREA	CAL #	AMT
2.92	2.93	5740	(R) 1	56.186
3.97	3.95	8720	2	58.919

DIL FACTOR: 1.0000 E+ 0

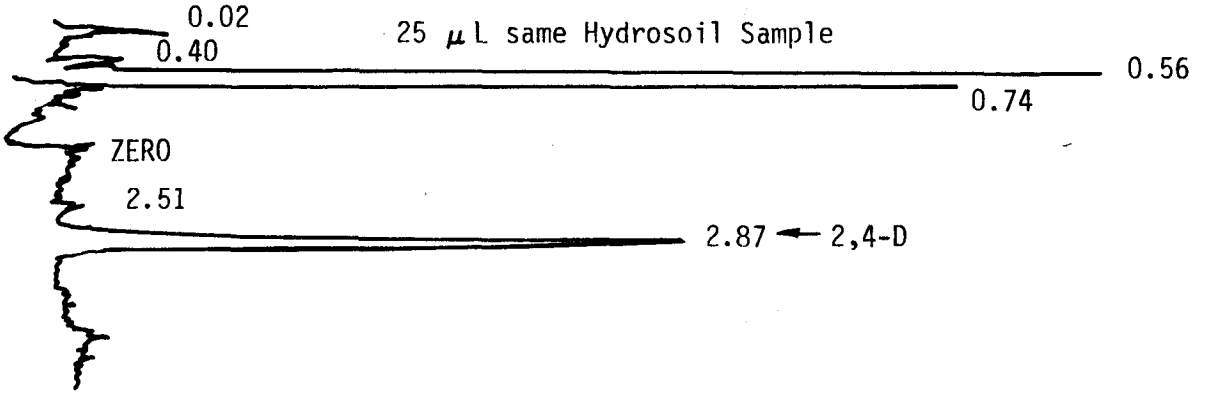
Figure A-6.—Standard HPLC.



RUN # 26 MAY/14/82 TIME 13:43:26
40-60-2.5

RT	EXP RT	AREA	CAL #	AMT
2.87	2.93	10240	(R) 1	100.235

FACTOR: 1.0000 E+ 0



RUN # 27 MAY/14/82 TIME 13:53:03
40-60-2.5

RT	EXP RT	AREA	CAL #	AMT
2.87	2.93	10400	(R) 1	101.801

Figure A-7.—Banks Lake Hydrosoil HPLC.

Attachment 6

GC/MS (GAS CHROMATOGRAPHY/MASS SPECTROMETRY) CONFIRMATION

Confirmation of 2,4-D in hydrosoil was performed by GC/MS (gas chromatography/mass spectrometry).

Reconstructed gas chromatograms and mass spectra from Banks Lake and Ft. Cobb samples (appendix A,fig. A-8 and fig. A-10, respectively) and from the 2,4-D methyl ester standard (appendix A, fig. A-12) confirm the presence of 2,4-D.

Confirmation was based on the comparison of the mass spectrum of 2,4-D methyl ester standard taken at scan No. 287, (appendix A, fig. A-13) and matching with mass spectra of samples taken at scan No. 287 (appendix A, fig. A-11, Ft. Cobb) and scan No. 286 (appendix A, fig. A-9, Banks Lake).

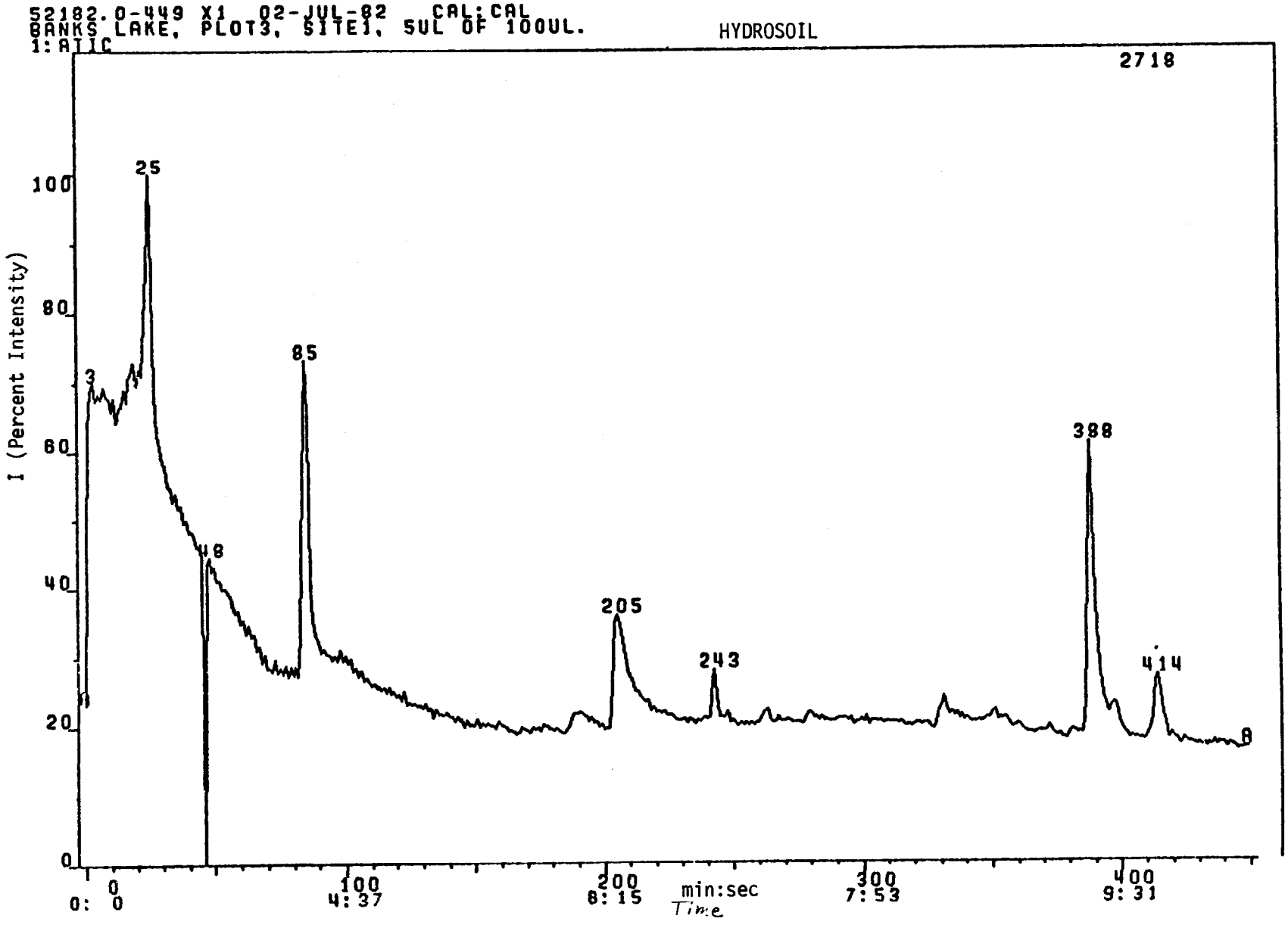


Figure A-8.—Banks Lake Hydrosoil GC.

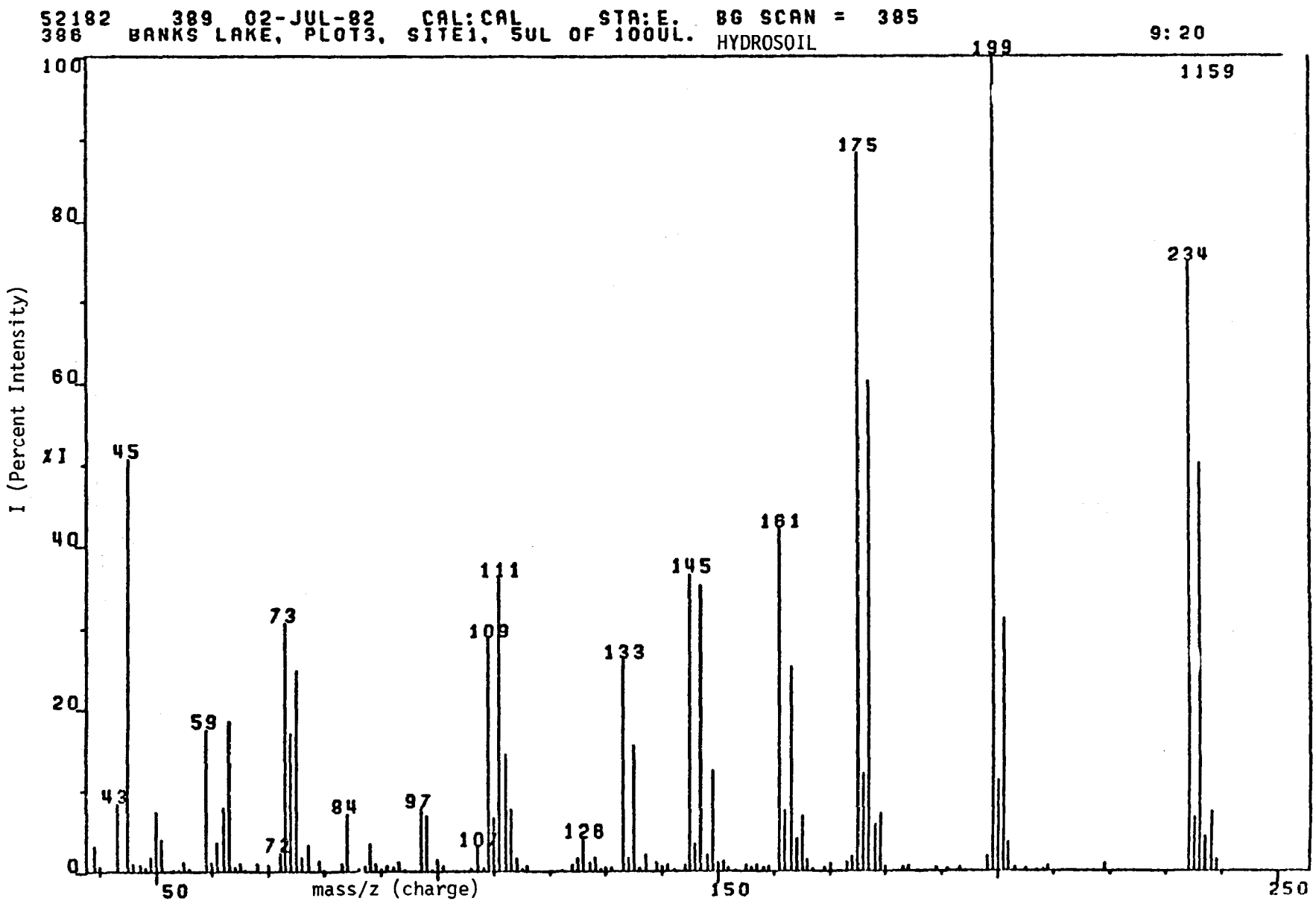


Figure A-9.—Banks Lake Hydrosoil MS.

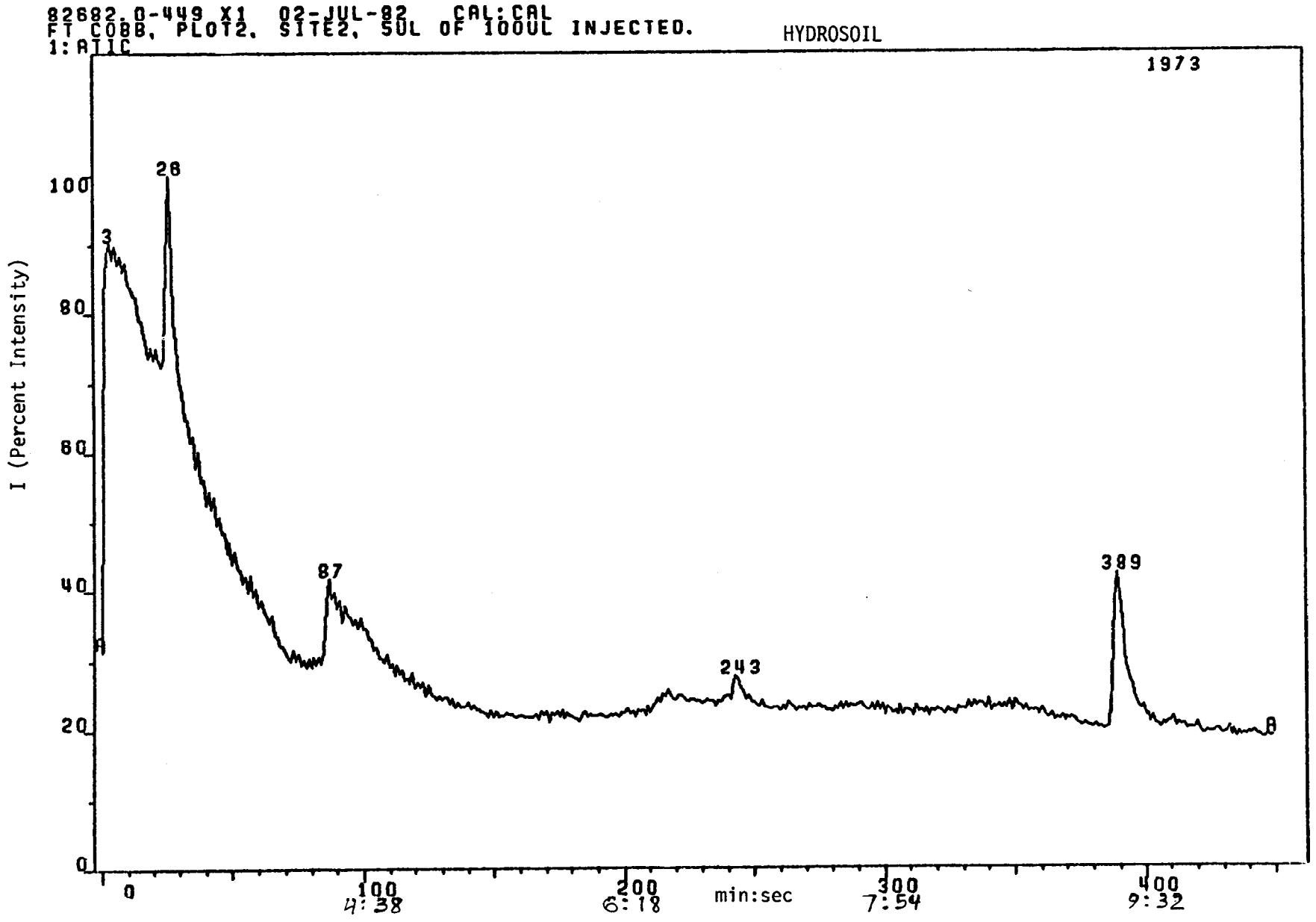


Figure A-10.—Ft. Cobb Res. Hydrosoil GC.

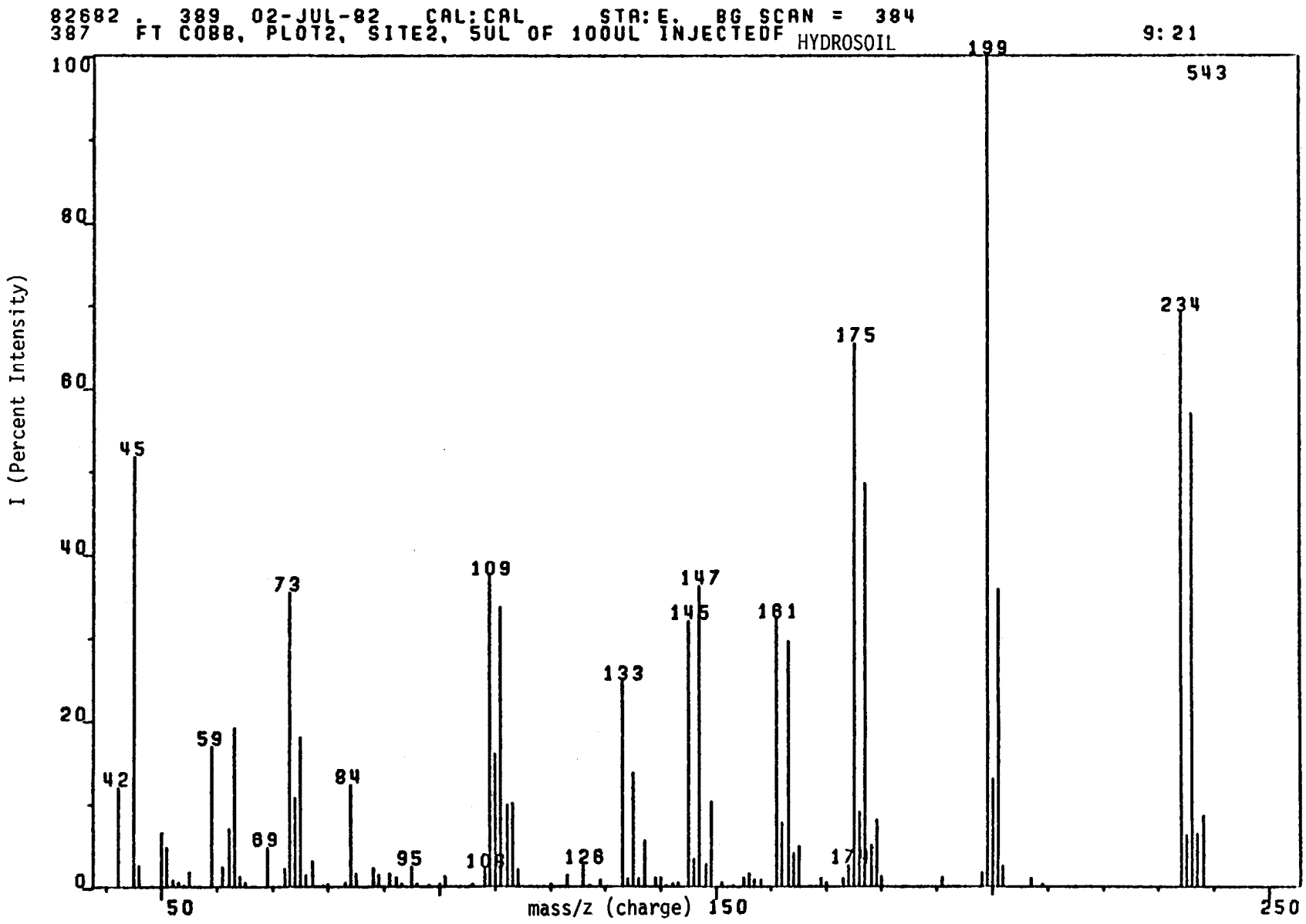


Figure A-11.—Ft. Cobb Res. Hydrosoil MS.

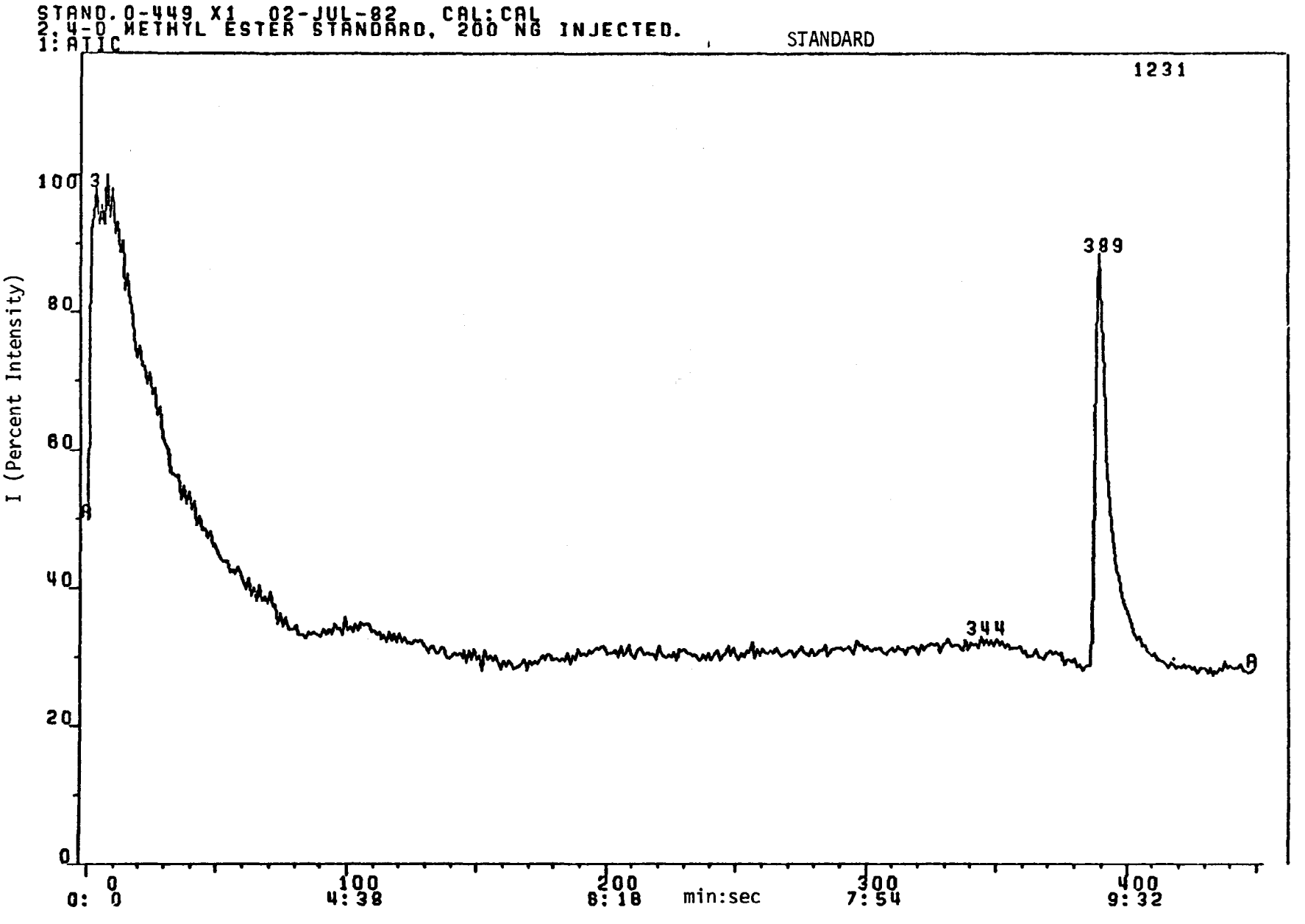


Figure A-12.—Standard GC.

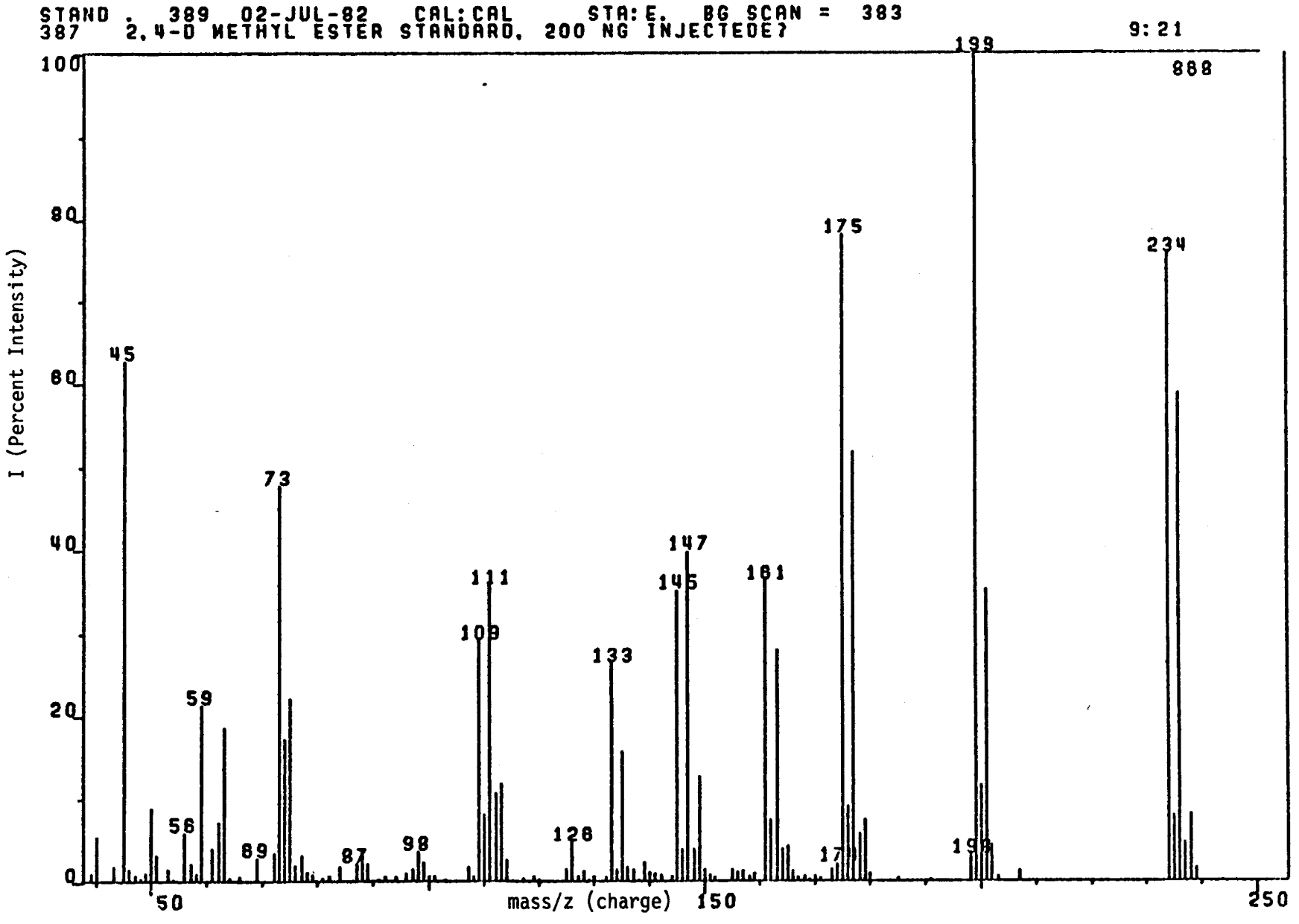


Figure A-13.—Standard MS.

PROTOCOL FOR FISH COLLECTION FOR 2,4-D RESIDUE ANALYSES

1. *Purpose of study.*—Residues in fish flesh resulting from the application of 2,4-D formulations DMA and BEE applied for control of eurasian watermilfoil in the Western United States reservoirs are required. This study is designed to be conducted concurrently with the 2,4-D water dissipation study to aid in supporting a proposed tolerance for 2,4-D use in western reservoirs. Fish flesh food tolerances have been previously established in support of 2,4-D labels for eurasian watermilfoil control in reservoir systems of the Tennessee Valley Authority. Data from this proposed study will provide information on geographically different environments and fish species of those previously established by TVA.

2. *Method of fish exposure to 2,4-D residues.*

A. Sites.—The same geographical locations and treatment plots utilized for the DMA and BEE dissipation studies will be sampled to determine resident fish population exposure.

B. Sampling techniques.—Electrofishing or gill net sampling techniques will be used, as appropriate. The success of electrofishing is dependent on water conductivity. Samples should be collected from three areas:

(1) In the vicinity of plots which were treated with both 2,4-D formulations and at both rates of application. Fish collected should be in the 178-254 mm (7-10 in) size range.

(2) Approximately 0.4 to 1.5 km (¼ to 1 mi) outside of each treated plot (dependent on circumstances).

(3) One untreated area should be sampled to determine exposure of fish to possible background levels of 2,4-D. The same site utilized for sampling of inlets to multiple use systems could be used.

C. Fish species.—Resident fish populations will be sampled in an effort to obtain at least two species at two trophic levels within the geographical area under study. The 178 to 254 mm (7-10 in) size range is preferable. Whenever possible, triplicate samples should be collected to represent each of the two species in a given sampling location.

The Banks Lake, Washington, study site was represented by the following fish species for residues: White sucker (*Catostomus commersoni*), white fish (*Coregonus lupeaformis*), and carp (*Cyprinus carpio*).

Large-mouth bass (*Micropterus salmoides*), bluegill (*Lepomis macrochirus*) and carp (*Cyprinus carpio*) were the species selected for the residue study at Fort Cobb Reservoir, Oklahoma.

3. *Sampling*

A. Methods.—Sampled fish are to be weighed, measured, cut into two edible portion fillets, wrapped in aluminum foil, placed in a plastic bag, and frozen on dry ice. Each fish sample will be labeled with location, sample date, and time. Replicates should **not** be composited.

B. Schedule.—Fish samples will be collected on the same schedule as specified for water and sediment samples, except that the 7 and 28 day samples will be deleted, leaving the following samples dates:

24 hr. pretreatment—one area only
1 day post treatment
4 days post treatment
14 days post treatment
56 days post treatment

Additonal samples may be collected if detectable residues dictate.

4. Residues analysis will be conducted to determine DMA and BEE. All fish samples are to be shipped in dry ice to the Denver Laboratory for analysis. Each fish is to be analyzed as a separate replication according to the attached procedure (attachment 8).

EXTRACTION AND ANALYSIS OF BEE (2,4-D BUTOXY ETHANOL ESTER) AND DMA (2,4-D DIMETHYLAMINE) FROM FISH TISSUE BY GAS CHROMATOGRAPHY

1. Prepare fish samples according to the procedure reported by Benville and Tindle[1]. Blend diced frozen fish in a ratio of 2:1 (dry ice:fish) until a homogeneous mixture is obtained.

2. Place mixture into a 0.5 L (1-pint) canning jar and cover with foil and then place into a freezer for at least 12 hours.

3. Weigh out 20 grams of fish tissue into a 250-mL beaker.

4. Add 90 grams of anhydrous sodium sulfate and mix occasionally for 20-45 minutes until a free-flowing mixture is obtained.

5. Pack mixture in a 20 mm × 400 mm chromatography column containing 2 grams of anhydrous sodium sulfate. Pack samples lightly by tapping column sides with a wooden spatula.

6. Rinse out 250-mL sample beaker using 20 mL of ethyl ether for fish samples taken out of BEE-treated plots and 10 mL of 1% phosphoric/methanol[2] for fish taken out of DMA-treated plots. Add 180 mL of the appropriate solvent to each column and collect effluent in the original beaker. Flow rates should be approximately 3-6 mL/min depending on packing.

7. Concentrate extracts to approximately 3 mL in a warm bath in a fume hood using a stream of nitrogen.

Extracts of Fish taken from BEE treated plots are then treated as follows:

8. Transfer remaining concentrate in 250-mL beaker to a 150-mL separatory funnel using 10 mL of hexane.

9. Rinse out original beaker with 30 mL of hexane saturated with acetonitrile and add to separatory funnel.

10. Extract hexane (saturated with acetonitrile) two times with 30-mL aliquots of acetonitrile saturated with hexane. Pool acetonitrile aliquots and evaporate to about 2 mL.

11. Transfer sample to a Florasil column (prerinse with 30 mL petroleum ether) containing 1 cm anyhdrous sodium sulfate and 2 cm Florasil with 3 mL hexane. Elute the column with 200 mL of 25% ethyl ether in petroleum ether.

12. Collect sample in a 250-mL beaker and concentrate to approximately 5 mL using a warm water bath and stream of nitrogen. Bring up the final volume with appropriate solvent before injecting into gas chromatograph.[3]

Extracts of fish taken from DMA-treated plots are treated as follows:

8. Transfer remaining concentrate in 250-mL beaker to a 150-mL separatory funnel using two 15-mL salt water rinses (2 grams NaCl/15 mL water).

9. Extract salt water two times with 30 mL aliquots of methylene chloride. Use one of the methylene chloride aliquots to rinse original 250-mL beaker.

10. Pool both methylene chloride extracts into a 150-mL beaker and evaporate to about 3 mL. Transfer remaining concentrate to a 150-mL separatory funnel using 10 mL of hexane.

11. Rinse out 150-mL beaker with 30 mL of hexane saturated with acetonitrile and add to separatory funnel.

12. Extract hexane (saturated with acetonitrile) two times with 30 mL aliquots of acetonitrile saturated with hexane. Pool acetonitrile aliquots and evaporate to approximately 2 mL. Evaporate to near dryness and methylate with 1 mL of diazomethane (1%) for 10 minutes.

13. Evaporate methylated samples to near dryness.

14. Transfer methylated sample to a prerinsed Florasil column using 5 mL of hexane.

15. Elute the column with 200 mL of 25% ethyl ether in petroleum ether.

16. Collect sample in a 250-mL beaker and concentrate to approximately 5 mL using a warm water bath and stream of nitrogen. Bring up to a final volume with appropriate solvent for gas chromatography use.[4]

17. The following gas chromotography parameters were used to analyze for BEE and DMA:

BEE		DMA	
Column Flow:	25 mL/min	Column Flow:	25 mL/min
Purge Flow:	12 mL/min	Purge Flow:	10 mL/min
Oven Temp:	200 °C	Oven Temp:	150 °C
Detector Temp:	355 °C	Detector Temp:	355 °C
Injector Temp:	225 °C	Injector Temp:	225 °C

A Model 530 Tracor gas chromotograph with a ^{63}Ni electron capture detector was utilized. The column used was a 2 mm i.d. × 2 m (6-ft) glass column packed with 80/100 mesh 3% OV-101 on Supelcoport. Argon/methane (5%) was used for the carrier gas. Florasil used was 60-100 mesh heated for 2 hours at 130 °C. Florasil was then stored in dessicator containing anhydrous sodium sulfate.

[1] Benville, P.E. and R.C. Tindle, "Dry Ice Homogenization Procedure for Fish Samples in Pesticide Residue Analysis," J. Agr. Food Chem., 18, pp 948–949, 1970.

Anticipated Recovery Levels

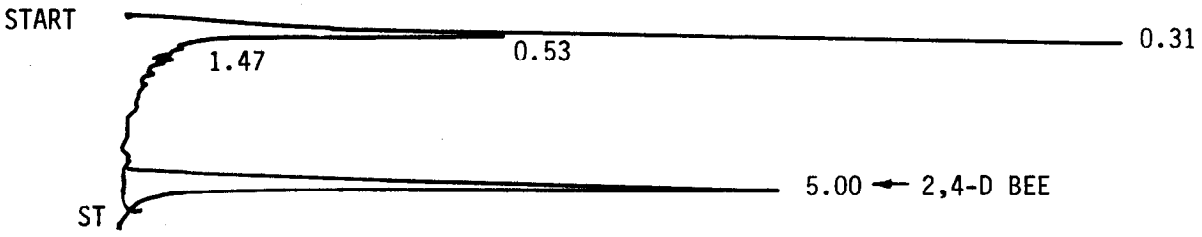
[2] 1% phosphoric acid in methanol

[3] Recovery of 2,4-D Bee from fish 50 to 55 percent

[4] Recovery of DMA from fish 30 to 35 percent.

STOP MIN? 6 . 5 0 @

5λ 0.10 ppm 2,4-D BEE

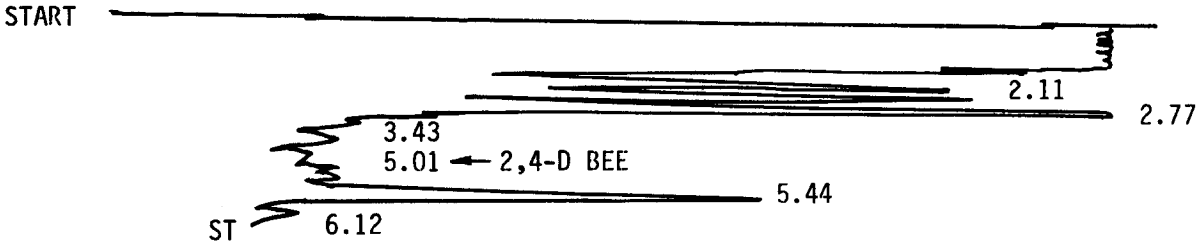


HP RUN # 3 JUL/23/82 TIME 09:59:46
ESTD

RT	EXP RT	AREA	CAL #	AMT
5.00	5.01	5896	(R) 1	0.502

DIL FACTOR: 1.0000 E+ 0

5 μL Banks Lake 7/15/81 Carp
PLOT 4 6 ft Final vol 6.0 mls

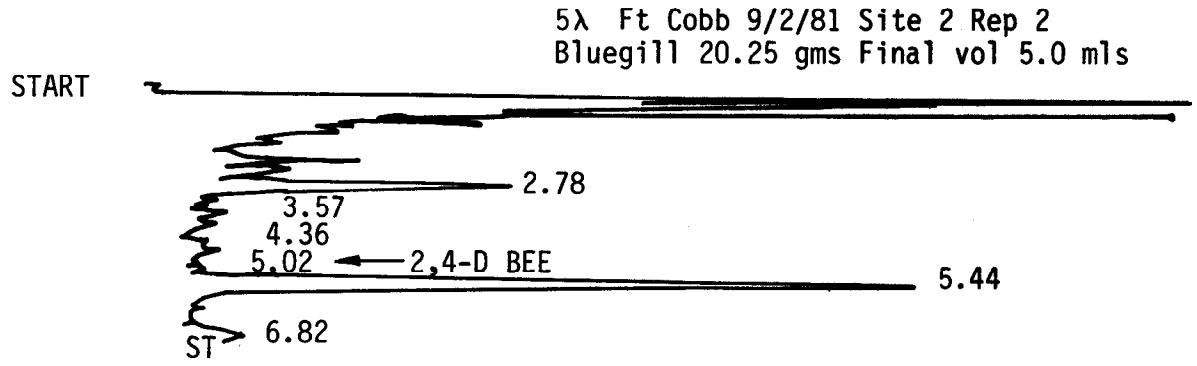


HP RUN # 4 JUL/23/82 TIME 10:09:28
ESTD

RT	EXP RT	AREA	CAL #	AMT
5.01	5.01	523	(R) 1	0.045

DIL FACTOR: 1.0000 E+ 0

Figure A-14.—Banks Lake Fish Tissue HPLC.



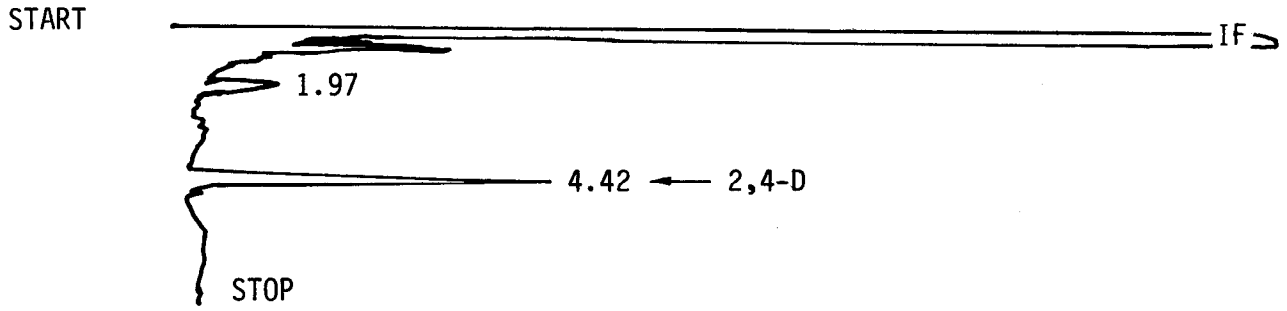
HP RUN # 9 JUL/19/82 TIME 09:50:44
ESTD

RT	EXP RT	AREA	CAL #	AMT
5.02	4.96	54	(R) 1	0.003

DIL FACTOR: 1.0000 E+ 0

Figure A-15.—Ft. Cobb Res. Fish Tissue HPLC.

2γ 0.10 ppm 2,4-D



HP RUN # 3 AUG/18/82 TIME 09:04:00
ESTD

RT	EXP RT	AREA	CAL #	AMT
4.42	4.34	2103	(R) 1	0.161

DIL FACTOR: 1.0000 E+ 0

DELETE ESTD
CALIB ESTD
% RTW: 3 . 0

	RT	AMT
REF:	4 . 4 2	: . 2 0 0
:	0	

DIL FACTOR:

READY
ESTD
% RTW: 3.00 CALIB RUNS 1

CAL #	RT	AMT	AMT/AREA
(R) 1	4.42	2.0000 E- 1	9.5102 E- 5

DIL FACTOR: 1.0000 E+ 0

2λ same Standard

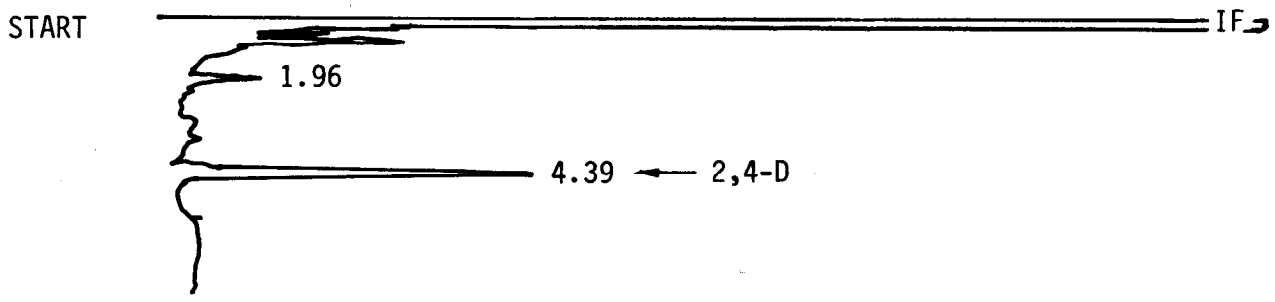
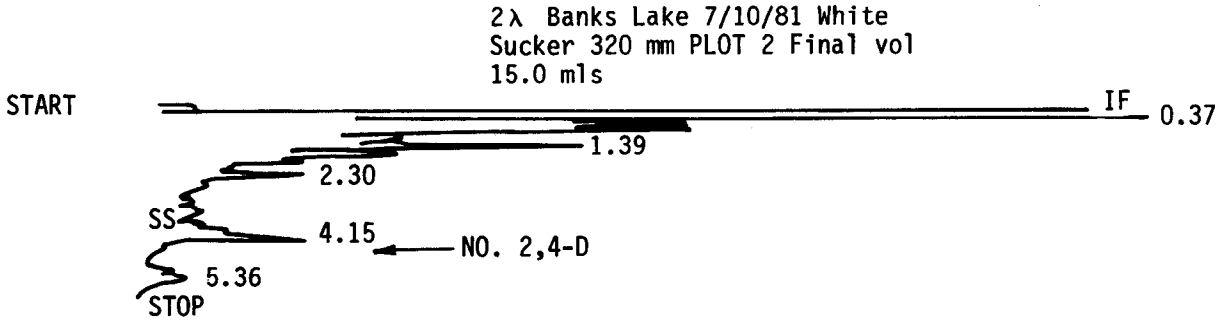


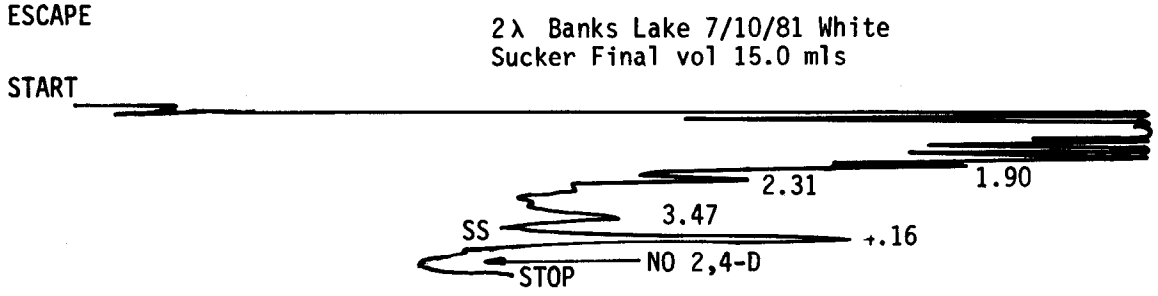
Figure A-16.—Standard HPLC.



S HP RUN # 10 AUG/18/82 TIME 10:59:43
NO PEAKS IN WDOS

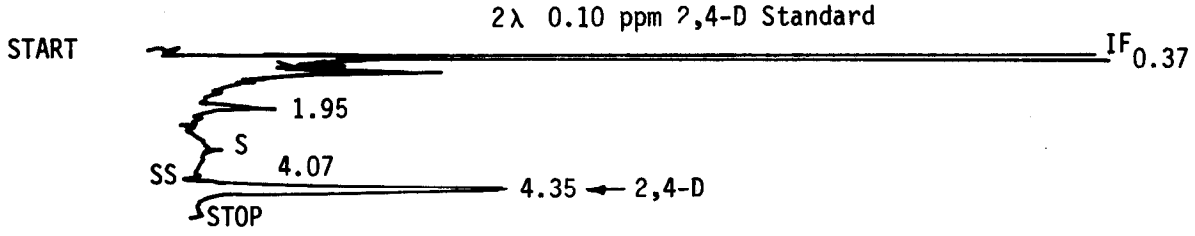
RT	AREA	AREA %

ESCAPE



S HP RUN # 11 AUG/18/82 TIME 11:22:55
NO PEAKS IN WDOS

ESCAPE



HP RUN # 12 AUG/18/82 TIME 12:17:33
ESTD

RT	EXP RT	AREA	CAL #	AMT
4.35	4.42	2064	(R) 1	0.196

DIL FACTOR: 1.0000 E+ 0

Figure A-17.—Banks Lake Fish Tissue HPLC.

Attachment 9

PROTOCOL FOR MONITORING AQUATIC INVERTEBRATES

1. Purpose of Study. The potential effects of experimental treatments of 2,4-D on zooplankton are to be determined following treatment with the DMA and BEE formulations for eurasian watermilfoil control in reservoirs. This study is designed to be conducted concurrently with the 2,4-D water dissipation and fish flesh residue studies that will be used to aid in supporting a proposed tolerance and labeling efforts for 2,4-D use in Western United States reservoirs. The EUP requires that short term field studies be conducted to determine possible impacts on fish and certain aquatic food chain zooplankton such as *Daphnia.* This study is designed to sample existing crustacean zooplankton populations in the reservoir study areas and any change that the herbicide may create in their occulations. Excellent baseline information is available on the zooplankton (including *Daphnia*) and fish populations for the study site at Banks Lake, Washington,[1] for comparative purposes. Less information is available on fish populations and aquatic invertebrates at the Fort Cobb, Oklahoma, study site.

2. Method of Determining Aquatic Invertebtate Populations.

A. Sites.—The same geographical locations and treatment plots utilized for the 2,4-D dissipation and fish exposure studies will be utilized to monitor zooplankton. The DMA and BEE formulations will be applied in these plots at rates of 22 and 45 kg/ha, a.e. (20 and 40 lb/acre, a.e.).

B. Sampling Locations.—Aquatic invertebrate samples will be collected at each of the study plots on each of the reservoirs as follows:

(1) One site within the treated plots of both 2,4-D formulations and both rates of application.

(2) One site outside of each of the four herbicide treated plots, 90 to 180 m. (100 to 200 yd) away from the treated area.

(3) One untreated site utilizing the same general site area used for sampling of inlets to multiple use system, for the water residue study.

3. Sampling.

A. Method. Zooplankton abundance will be determined to estimate standing stock at each sample location by duplicate vertical hauls from 3 m (10 ft) to the surface using a No. 10 (0.168 mm aperture) closing or Wisconsin type plankton net or similar.[2] Zooplankton samples collected in the plankton net basket will be emptied into a sample jar, washing residue with a squirt bottle to insure total sample collection. The plankton samples will be preserved with a 5% formalin solution for processing.

B. Schedule. Zooplankton vertical hauls will be collected on a schedule similar to that specified for water and sediment as follows:

1, 1, 4, 7, 14, 18, 56 days posttreatment and up to 90 days if water residue data reflects detectable residues.

C. Summary Totals. Duplicate sample/sample site i.e. 2 samples per site

Four samples from within herbicide treated plots	2 × 4 =	8 samples/site/collection day
Four samples from outside herbicide treated plots	2 × 4 =	8 samples/site/collection day
One sample at reservoir outlet sampling site	1 × 2 =	2 samples/site/collection day
		18
Seven sampling times (7 × 18)	=	126 total samples/site

D. Sample Processing. Received samples will be diluted in the laboratory to a concentration of 10 to 200 organisms/mL. Two to four/mL subsamples from each sample will be counted with a low power stereo microscope over a grid. Numbers of each genus of cladoceran and copepods will be recorded. The volume of water sampled will be calculated by multiplying the net mouth area by the distance hauled. From this the number of each zooplankton composition and densities. Major discernible differences of statistical significance between herbicide treated and untreated areas will be interpreted as a possible indicator of herbicide impacts on the aquatic environment.

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[1] Stober, O. J., et al. Report No. REC-ERC-77-5, Operational Effected Irrigation and Pumped Storage on Ecology of Banks Lake, Washington, U.S. Bureau of Reclamation, December 1977.

[2] In areas near dense weed growth a 1-liter Kemmerer or Van Dorn water sample will be used to sample surface and a 3-m (10-ft) sample. A composite will be poured through the above plankton net for sampling.

SUPPLEMENTAL LABELING FOR EXPERIMENTAL USE WITH AQUA-KLEEN[R]—EPA REGISTRATION NO. 254-109

FOR EXPERIMENTAL USE ONLY

PURPOSE. For use in a cooperative experimental program of the Water and Power Resources Service, U.S. Department of the Interior, and the Corps of Engineers, U.S. Army, to determine 2,4-D residue disappearance characteristics when the herbicide is used for control of eurasian watermilfoil (Myriophyllum spicatum L.) in reservoirs and other waters.

EXPERIMENTAL USE PERMIT. This supplemental labeling is valid only when used in accordance with provisions of Experimental Use Permit No. 11683-EUP-3, issued by the U.S. Environmental Protection Agency on July 10, 1980.

Experimental applications are limited to the following waters:

Fort Cobb Reservoir, Oklahoma (Water and Power Resources Service)
Banks Lake, Washington (Water and Power Resources Service)
Lake Seminole, Florida-Georgia (Corps of Engineers)
Robert S. Kerr Reservoir, Oklahoma (Corps of Engineers)

This supplemental labeling must be in the possession of the user at the time of the pesticide application.

Experimental applications are to be made by or under the supervision of Water and Power Resources Service or Corps of Engineers personnel who are certified under an approved State plan or Federal agency plan for application of restricted-use pesticides, or by certified commercial applicators under contract to the Water and Power Resources Service or the Corps of Engineers.

METHOD AND RATE OF APPLICATION. Follow "Directions for Use" given in the EPA-approved labeling of AQUA-KLEEN[R] for eurasian watermilfoil control in programs conducted by the Tennessee Valley Authority in dams and reservoirs of the TVA system.

Surface or aerial application methods may be used.

The experimental applications are to be made at two rates: 100 lbs. and 200 lbs. of herbicide product per acre. (This is to provide treatments of 20 lb. and 40 lb. of 2,4-D acid equivalent per acre.)

PRECAUTIONS. Follow all precautions given in the AQUA-KLEEN[R] labeling.

If aerial application is to be made, remove people and livestock from the area to be treated.

Treated areas will be posted as such and be restricted from swimming for 14 days after treatment.

Potable water.—Do not treat within ½ mile of any municipal or domestic water intake.

Delay the use of water from treated areas for domestic purposes for a period of 3 weeks or until residue analyses show that it contains no more than 0.1 ppm 2,4-D acid.

Irrigation.—Delay the use of water from treated areas for a period of 3 weeks. Do not use treated water for irrigation of sensitive crops such as grapes, tomatoes, and cotton until residue analyses show 2,4-D to be absent.

SUPPLEMENTAL LABELING FOR EXPERIMENTAL USE WITH WEEDAR 64[R] HERBICIDE—EPA REGISTRATION NO. 254-2

FOR EXPERIMENTAL USE ONLY

PURPOSE. For use in a cooperative experimental program of the Water and Power Resources Service, U.S. Department of the Interior, and the Corps of Engineers, U.S. Army, to determine 2,4-D residue disappearance characteristics when the herbicide is used for control of eurasian watermilfoil (Myriophyllum spicatum L.) in reservoirs and other waters.

EXPERIMENTAL USE PERMIT. This supplemental labeling is valid only when used in accordance with provisions of Experimental Use Permit No. 11683-EUP-2, issued by the U.S. Environmental Protection Agency on July 10, 1980.

Experimental applications are limited to the following waters:

Fort Cobb Reservoir, Oklahoma (Water and Power Resources Service)
Banks Lake, Washington (Water and Power Resources Service)
Lake Seminole, Florida-Georgia (Corps of Engineers)
Robert S. Kerr Reservoir, Oklahoma (Corps of Engineers)

This supplemental labeling must be in the possession of the user at the time of the pesticide application.

Experimental applications are to be made by or under the supervision of Water and Power Resources Service or Corps of Engineers personnel who are certified under an approved State plan or Federal agency plan for application of restricted-use pesticides, or by certified commercial applicators under contract to the Water and Power Resources Service or the Corps of Engineers.

METHOD AND RATE OF APPLICATION. Follow "Directions for Use" given in the EPA-approved labeling of WEEDAR 64[R] for eurasian watermilfoil control in programs conducted by the Tennessee Valley Authority in dams and reservoirs of the TVA system.

Surface, subsurface, or aerial application methods may be used.

The experimental applications are to be made at two rates: 5 and 10 gallons of herbicide product per acre. (This is to provide treatments of 20 lb. and 40 lb. of 2,4-D acid equivalent per acre.)

PRECAUTIONS. Follow all precautions given in the WEEDAR 64[R] labeling.

If aerial application is to be made, remove people and livestock from the area to be treated.

Treated areas will be posted as such and be restricted from swimming for 14 days after treatment.

Potable water.—Water to be treated will be a minimum of one-half (½) mile from the nearest intake system. Continuous monitoring is required.

Delay the use of water from treated areas for domestic purposes for a period of 3 weeks or until residue analyses show that it contains no more than 0.1 ppm 2,4-D acid.

Irrigation.—Delay the use of water from treated areas for a period of 3 weeks. Do not use treated water for irrigation of sensitive crops such as grapes, tomatoes, and cotton until residue analyses show 2,4-D to be absent.

APPENDIX B

Table B-1.—*Water chemical analysis, Banks Lake, Washington—Outlet.*

Water quality parameter	Pretreat	1 day	4 day	7 day	14 day	28 day	56 day
Conductivity, μm/c	134	149	No	121	No	100	No
pH	8.2	8.3	data	8.3	data	8.7	data
TDS/105 °C, mg/L	82.5	85.0		109.0		92.0	
Calcium, mg/L	17.3	18.6		17.0		13.2	
Magnesium, mg/L	2.5	3.2		4.9		1.7	
Sodium, mg/L	3.0	2.5		2.3		1.8	
Potassium, mg/L	1.4	0.8		0.8		0.8	
Carbonate, mg/L	0.0	0.0		0.0		7.2	
Bicarbonate, mg/L	54.9	53.7		56.1		19.5	
Sulfate, mg/L	17.8	16.8		18.2		17.3	
Chloride, mg/L	0.7	0.7		0.7		0.7	
Anion and cation, mg/L	97.5	96.3		100.0		62.2	

Table B-2.—*Water chemical analysis, Banks Lake, Washington—Herbicide plot 1.*

Parameter	Pretreat	1 day	4 day	7 day	14 day	28 day	56 day
Conductivity, μm/c	119	129	No	149	No	119	No
pH	8.3	8.25	data	8.25	data	8.3	data
TDS/105 °C, mg/L	69.7	44.5		51.0		91.0	
Calcium, mg/L	16.7	17.5		19.0		17.6	
Magnesium, mg/L	3.8	4.2		4.0		3.9	
Sodium, mg/L	2.6	2.3		2.3		1.8	
Potassium, mg/L	0.9	0.8		0.8		0.8	
Carbonate, mg/L	0.80	0.0		0.0		0.0	
Bicarbonate, mg/L	54.5	57.4		61.0		56.1	
Sulfate, mg/L	15.5	15.9		17.3		19.7	
Chloride, mg/L	0.7	0.7		0.7		0.7	
Anion and cation, mg/L	95.5	98.5		105.0		101.0	

Table B-3.—*Water chemical analysis, Banks Lake, Washington—Herbicide plot 2.*

Water quality parameter	Pretreat	1 day	4 day	7 day	14 day	28 day	56 day
Conductivity, μm/c	No	139	111	104	No	107	No
pH	data	8.1	8.4	8.4	data	8.4	data
TDS/105 °C, mg/L		107.0	86.0	37.0		89.0	
Calcium, mg/L		21.8	16.4	20.0		16.2	
Magnesium, mg/L		0.854	2.8	0.5		3	
Sodium, mg/L		2.53	2.07	2.3		1.8	
Potassium, mg/L		0.8	0.8	0.8		0.8	
Carbonate, mg/L		0.0	2.4	2.4		2.4	
Bicarbonate, mg/L		61.0	43.9	43.9		46.4	
Sulfate, mg/L		13.9	18.7	14.9		17.3	
Chloride, mg/L		0.7	0.7	0.7		0.7	
Anion and cation, mg/L		102.0	87.8	85.5		89.4	

Table B-4.—*Water chemical analysis, Banks Lake, Washington—Herbicide plot 3.*

Parameter	Pretreat	1 day	4 day	7 day	14 day	28 day	56 day
Conductivity, μm/c	No	123.0	109.0	No	No	109.0	No
pH	data	8.0	8.3	data	data	8.4	data
TDS/105 °C, mg/L		47.0	45.0			88.0	
Calcium, mg/L		17.4	15.8			13.8	
Magnesium, mg/L		4.51	2.81			5.25	
Sodium, mg/L		2.53	1.84			2.07	
Potassium, mg/L		0.782	0.782			0.782	
Carbonate, mg/L		0.0	0.0			2.4	
Bicarbonate, mg/L		58.6	46.4			48.8	
Sulfate, mg/L		15.4	17.8			14.9	
Chloride, mg/L		0.71	0.71			0.71	
Anion and cation, mg/L		99.9	86.1			88.7	

Table B-5.—*Water chemical analysis, Banks Lake, Washington—Herbicide plot 4.*

Water quality parameter	Pretreat	1 day	4 day	7 day	14 day	28 day	56 day
Conductivity, μm/c	124.3	No	109.0	116.0	No	104.0	No
pH	8.4	data	8.5	8.3	data	8.65	data
TDS/105 °C, mg/L	65.5		72.0	40.0		95.0	
Calcium, mg/L	17.9		16.4	21.8		16.0	
Magnesium, mg/L	3.3		2.68	0.12		2.7	
Sodium, mg/L	2.3		2.07	2.3		1.8	
Potassium, mg/L	0.8		0.8	1.2		0.8	
Carbonate, mg/L	1.2		4.8	0.0		4.8	
Bicarbonate, mg/L	53.7		36.6	53.7		36.6	
Sulfate, mg/L	16.1		18.7	16.3		16.3	
Chloride, mg/L	0.7		0.7	0.7		0.71	
Anion and cation, mg/L	96.2		82.8	96.1		79.7	

Table B-6.—*Water chemical analysis, Fort Cobb Reservoir, Oklahoma—Outlet.*

Parameter	Pretreat	1 day	4 day	7 day	14 day	28 day	56 day
Conductivity, μm/c	380.0	337.0	360.0	326.0	454.0	No	435.0
pH	8.2	7.9	7.7	8.1	8.3	data	7.7
TDS/105 °C, mg/L	252.0	303.0	299.0	233.0	273.0		264.0
Calcium, mg/L	35.2	37.4	35.4	22.2	27.4		31.8
Magnesium, mg/L	22.2	9.76	18.8	14.0	22.1		17.4
Sodium, mg/L	2.07	20.0	24.4	19.6	30.6		26.4
Potassium, mg/L	0.782	5.47	8.6	5.86	8.6		7.82
Carbonate, mg/L	0.0	0.0	0.0	0.0	0.0		0.0
Bicarbonate, mg/L	85.4	87.8	83.0	58.6	87.8		87.8
Sulfate, mg/L	101.0	88.8	131.0	91.2	129.0		115.0
Chloride, mg/L	12.1	9.94	17.0	8.88	13.8		12.8
Anion and cation, mg/L	259.0	259.0	318.0	220.0	319.0		299.0

Table B-7.—*Water chemical analysis, Fort Cobb Reservoir, Oklahoma—Herbicide plot 1.*

Parameter	Pretreat	1 day	4 day	7 day	14 day	28 day	56 day
Conductivity, µm/c	No	No	410	No	418	394	475
pH	data	data	7.7	data	8.3	8.1	8.1
TDS/105 °C, mg/L			269.0		287.0	241.0	320.0
Calcium, mg/L			30.4		26.0	32.2	28.4
Magnesium, mg/L			22.4		20.7	14.3	22.3
Sodium, mg/L			31.3		31.3	22.8	36.6
Potassium, mg/L			8.6		9.0	7.0	9.0
Carbonate, mg/L			0.0		0.0	0.0	0.0
Bicarbonate, mg/L			92.7		75.6	85.4	92.7
Sulfate, mg/L			137.0		134.0	108.0	129.0
Chloride, mg/L			13.8		15.3	10.3	14.9
Anion and cation, mg/L			337.0		312.0	279.0	327.0

Table B-8.—*Water chemical analysis, Fort Cobb Reservoir, Oklahoma—Herbicide plot 2.*

Parameter	Pretreat	1 day	4 day	7 day	14 day	28 day	56 day
Conductivity, µm/c	No	No	423	464	426	360	454
pH	data	data	8.0	7.8	8.3	8.0	8.0
TDS/105 °C, mg/L			335.7	288.0	259.0	244.0	337.0
Calcium, mg/L			32.4	34.6	31.4	34.4	36.4
Magnesium, mg/L			20.6	19.2	16.5	17.3	19.0
Sodium, mg/L			28.7	28.8	26.7	23.2	25.1
Potassium, mg/L			8.9	8.2	9.0	7.04	8.6
Carbonate, mg/L			0.8	0.0	0.0	0.0	0.0
Bicarbonate, mg/L			84.6	100.0	78.1	101.0	108.0
Sulfate, mg/L			138.0	123.0	122.0	108.0	117.0
Chloride, mg/L			15.4	15.6	10.7	11.4	11.0
Anion and cation, mg/L			329.6	330.0	295.0	303.0	325.0

Table B-9.—*Water chemical analysis, Fort Cobb Reservoir, Oklahoma—Herbicide plot 3.*

Water quality parameter	Pretreat	1 day	4 day	7 day	14 day	28 day	56 day
Conductivity, µm/c	No	No	426	497	475	402	No
pH	data	data	8.0	7.7	8.2	8.0	data
TDS/105 °C, mg/L			377.0	311.0	286.0	301.0	
Calcium, mg/L			30.4	35.0	28.0	32.0	
Magnesium, mg/L			20.4	21.0	21.5	21.1	
Sodium, mg/L			27.6	32.2	30.8	30.8	
Potassium, mg/L			8.6	9.0	9.0	9.0	
Carbonate, mg/L			0.0	0.0	0.0	0.0	
Bicarbonate, mg/L			80.5	109.0	97.6	109.0	
Sulfate, mg/L			133.0	132.0	125.0	138.0	
Chloride, mg/L			16.0	13.1	11.7	14.9	
Anion and cation, mg/L			316.0	351.0	324.0	355.0	

Table B-10.—*Water chemical analysis, Fort Cobb Reservoir, Oklahoma—Herbicide plot 4.*

Parameter	Pretreat	1 day	4 day	7 day	14 day	28 day	56 day
Conductivity, μm/c	No	No	No	435	367	326	337
pH	data	data	data	8.1	7.9	7.9	8.0
TDS/105 °C, mg/L				258.0	241.0	204.0	222.0
Calcium, mg/L				22.6	30.8	28.0	35.4
Magnesium, mg/L				19.8	18.3	25.4	10.4
Sodium, mg/L				31.7	31.7	30.8	16.1
Potassium, mg/L				9.0	9.0	8.6	4.7
Carbonate, mg/L				0.0	0.0	0.0	0.0
Bicarbonate, mg/L				61.6	112.0	80.5	83.0
Sulfate, mg/L				133.0	104.0	151.0	85.0
Chloride, mg/L				15.3	14.9	12.8	5.7
Anion and cation, mg/L				293.0	321.0	337.0	240.0

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