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Aquatic Plant Control and Disappearance of Terbutryn from Treated Waters in Ontario, Canada, 1974-1977.

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ABSTRACT

Between 1974 and 1977, 13 ponds and 12 plots in open lakes were treated with 2-(*tert*-butylamino)-4-(ethylamino)-6-(methylthio)-*s*-triazine(terbutryn). The treatments were based on applying the equivalent of 50 to 400 μg of active ingredient per liter of water body assuming all the herbicide dissolved. Analyses of water and sediment revealed that about half of the applied terbutryn remained in the water column while half was deposited in the sediment. Where currents and water movement carried the terbutryn out of the treated area, little effect was observed on aquatic plants. Where water flow was interrupted for 10 days, control of aquatic plants was usually excellent. However,

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rotting plant material depleted oxygen levels affecting some fish species. The best control occurred under static or nearly static water conditions. Filamentous algae were the most easily destroyed by terbutryn, submersed vascular aquatic plants were intermediate in their susceptibility to the herbicide, taking a longer period of contact, and emerged aquatic plants were difficult or not controlled by even the highest levels of treatments. Terbutryn appeared to disappear from some systems rapidly and others more slowly. Low residue levels were identified in sediments 12 months after treatment.

INTRODUCTION

Many ponds and lakes that two decades ago were relatively free of aquatic vegetation are now covered in dense mats of algae blooms, and associated with submersed and emerged aquatic plants. The increased density of plant growth has been correlated with increased plant nutrients, primarily nitrates and phosphates which were limiting in the past.

While actions are being taken to reduce the loss of nutrients from land in a number of ways, the impact of these actions are not expected to be immediate but rather gradual over a long term, and in some situations no practical solutions are readily available. As a stop gap measure to reduce the growth of excessive vegetation in bodies of water, herbicides have been employed. One of the herbicides being tested by Heuss (1976) for this purpose was a triazine, called terbutryn.

In this study terbutryn was used under a variety of conditions to determine its practicability of controlling vegetation in farm ponds, small bodies of water and large lakes where channels were desired through a mat of vegetation.

METHODS AND MATERIALS

Field Work

Treatment Technique. Between 1974 and 1978, 13 ponds and 12 lake plots were treated with terbutryn for the control of a wide variety of aquatic plants (Table 1). Water depths were determined from grid transects across the water surface. The total quantity of herbicide to be applied was calculated based on the surface area, the average depth and the concentration desired. The application of granules or flowable powder was made using a manual or motorized portable seeder operated either from the adjacent land or from a boat. Either way application was in such a manner as to create a uniform blanket of herbicide over the water surface. More material was applied where water depth increased significantly in order to facilitate a more uniform concentration in the water column around the floating plant mass.

Experiments in 1974. Preliminary studies were commenced in 1974 using terbutryn to control a wide range of algae and aquatic plants in several bodies of water. Eight ponds were selected for treatment with terbutryn to give water concentrations of between 50 and 200 μg/L active in-

TABLE 1. LIST OF COMMON SPECIES OF AQUATIC PLANTS FOUND IN 13 PONDS AND 12 LAKE PLOTS WITH TERBUTRYN BETWEEN 1974 AND 1977.

Aquatic Plants	Bodies of water treated (P-Pond L-Lake)			
	1974	1975	1976	1977
Filamentous Algae				
Chara vulgaris L.	P 1-4	P 9, L 6	P 10	
Cladophora sp.	P 1-8		P 10-13	P 11-13
Mougeotia sp.	P 1-4			
Rhizoclonium sp.	P 2,3			
Spirogyra sp.	P 1-4		P 10-13 L 7-12	P 11-13
Submersed Macrophytes				
Ceratophyllum demersum L.—common coontail	L 1,2	L 6	L 7-12	
Elodea canadensis Michx—waterweed	L 1-4		P 11-13 L 7-12	P 11-13
Myriophyllum sp.—watermilfoil		L 6	L 7-12	
Najas flexilis (Willd) Rostk and Schmidt—slender naiad	L 1,2			
Potamogeton sp.	P 5-8, L 1-4	P 9, L 5,6	P 11-13 L 7-12	P 11-13
P. amplifolius Tuckerm—largeleaf pondweed				
P. crispus L.—curlyleaf pondweed				
P. natans L.—floating leaf pondweed				
P. pectinatus L.—sago pondweed				
P. richardsonii (Benn) Rydb—claspingleaf pondweed				
Vallisneria americana Michx—vallisneria	L 1-4	L 5		
Emerged Aquatics				
Lemna sp.				P 11-13
L. minor L.—lesser duckweed				
L. trisulca L.—star duckweed				
Nuphar sp.—spatterdock		L 5		
Scirpus validus Vahl—great bulrush	P 8			
Typha sp./cattail	P 8			
Wolffia columbiana Karst—watermeal				P 11-13

gredient (Table 2). Ponds 1, 2, 3 and 4 were all treated with a 1% granular formulation and water flow was interrupted for 9 to 10 days following treatment. In pond 1, heavy rains prior to treatment cleared the surface water of floating algae and left the water turbid. The main aquatic plants in the four ponds were three species of filamentous algae *Cladophora* sp., *Mougeotia* sp., and *Spirogyra* sp. and the filamentous algae, *Chara vulgaris* L. (Table 1).

Four small ponds (5, 6, 7 and 8) were treated with 50% flowable terbutryn in June, 1974, but because the water was static, no further interruption of flow was necessary. All four ponds were infested with *Cladophora* sp. and *Potamogeton* sp. In addition, pond 8 contained cattails (*Typha* sp.).

Four small plots (Lake 1-4) in Pigeon Lake, were treated with 1% granular terbutryn to give a calculated water concentration of 100, 200, 300 and 400 µg/L active ingredient (Table 3). The plots were infested with a number of submersed vascular plants including vallisneria (*Vallisneria americana* Michx) slender naiad (*Najas flexilis* (Willd) Rostk and Schmidt), three species of pondweed (*Potamogeton crispus* L., *P. richardsonii* (Benn) Rydb and *P. natans* L.) and waterweed (*Elodea canadensis* Michx) (Table 1).

Experiments in 1975. One farm pond and two sites on two lakes were treated with 1% granular terbutryn to give a calculated water concentration of 100 µg/L. The farm pond (number 9) covered 3990 m^2 to a depth of 1.2 m and was infested with *Chara vulgaris* L. and *Potamogeton*, each occupying 50% of the surface but only 15% of the pond bottom (Table 1). In June, a 1% granular terbutryn formulation was distributed over the surface (46 kg) to give the observed concentration in the water (Table 4).

A beach area (Lake 5) on Doe Lake which was infested with spatterdock (*Nuphar* sp.), *Potamogeton* sp., and *Vallisneria americana* Michx, which covered respectively 45, 45 and 10% of the plot surface but collectively only 30% of the lake bottom, was treated with 1% granular terbutryn to give the desired 100 µg/L water concentration. The plot covered 900 m^2 with a mean depth of 1.9 m.

A one hectare plot (Lake 6) averaging 1.2 m deep, located on Puslinch Lake was treated with 1% granular terbutryn (Table 5). The site appeared relatively static and was infested with curlyleaf pondweed (*Potamogeton crispus* L.) and *Chara vulgaris* L. covering 95 and 5% of the surface respectively and collectively 80% of the lake bottom. Samples were collected both inside and outside the treated site to measure movement outside the treated plot.

TABLE 3. RESIDUES IN WATER AND DRIED SEDIMENT FOLLOWING THE APPLICATION OF TERBUTRYN AT FOUR RATES TO FOUR PLOTS IN PIGEON LAKE, 1974.[1]

Pigeon Lake	Terbutryn in water (µg/L) Calculated	Measured	Terbutryn in sediment (µg/g)
Plot 1	100	23	22
2	200	22	19
3	300	50	28
4	400	52	44

[1]Application 25 June, 1974 to plots 3 m^2 and water depth 1.5 to 2.1 m.

TABLE 4. PERSISTENCE OF TERBUTRYN IN A STATIC FARM POND 9, TREATED 27 JUNE, 1975 TO CONTROL *Chara* SP. AND *Potamogeton* SP. WITH CALCULATED HERBICIDE APPLICATION OF 100 µG/L.[1]

Days after treatment	Terbutryn in water (µg/L) Mean	SD	Terbutryn in dried sediment (µg/g) Mean	SD
Pre treatment	ND	—	ND	—
Post treatment				
Day 0	12.1	5.8	3.60	1.40
Day 7	76.7	5.9	0.57	0.23
Day 28	45.0	4.6	0.55	0.20
Day 41	44.8	1.4	2.11	0.71

[1]Pond: surface area 3990 m^2 and average depth 1.2 m.

Experiments in 1976. A pond, a sequence of ponds and six plots on a lake were treated with 1% granular terbutryn to control a wide range of aquatic plants. The first pond, (10), which covered an area of 2330 m^2, to a mean depth of 1.3 m, was treated with 0.16 kg a.i. terbutryn to give a calculated water concentration of 50 µg/L on 28 June, 1976. The main aquatic plants were filamentous algae. Because of regrowth of phytoplankton, the pond was treated a second time on 24 August, 1976.

A sequence of three ponds (11, 12 and 13) was selected for treatment. The head pond was left untreated, while ponds 12 and 13 were treated with terbutryn to provide a calculated water concentration of 100 µg/L a.i. on 2 June, 1976. The water flow from the head pond through ponds 12 and 13 was 4.80 m^3/minute so that a complete turnover of water occurred in 6 days and 20 hrs. Pond 12 was 1.9 m deep and had a surface area of 2388 m^2, while pond 13 was 2.5 m deep and covered 8200 m^2. The amounts of terbutryn used were 0.86 and 2.05 kg respectively. The ponds were infested with *Cladophora* sp., *Elodea canadensis* Michx, *Spirogyra* sp., and *Potamogeton crispus* L.

TABLE 2. DETAILS ON EIGHT PONDS TREATED WITH TERBUTRYN IN 1974.

Pond Location	Terbutryn concentration in water (µg/L) Treatment Date	Calculated	Measured Day 0-1	Flow interruption	Surface Area (m^2)	Depth (m)
1. Bolton	16 May	50	20	10 days	1497	1.3
2. Terra Cotta	5 June	100	30	9 days	3252	1.2
3. Milton	6 June	100	50	10 days	697	0.5
4. Bolton	7 June	100	40	10 days	4032[1]	1.6
5. Woodbridge	17 June	50	150	static	125	1.1
6. Woodbridge	17 June	100	140	static	6370	2.1
7. Woodbridge	17 June	100	140	static	3172	2.6
8. Woodbridge	17 June	200	320	static	1688	2.3

[1]Only 2016 m^2 of surface treated.

TABLE 5. DISAPPEARANCE OF TERBUTRYN FROM AN AREA TREATED IN PUSLINCH LAKE (L 6) 1975 TO PROVIDE AN INITIAL WATER CONCENTRATION OF 100 µG/KG.[1]

Days after treatment	Terbutryn in water (µg/L)				Terbutryn in dried setdiment (µg/g)			
	Treated area		Outside treated area		Treated area		Outside treated area	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Pre treatment	ND		ND		ND		ND	
Post treatment								
Day 0	40.1	1.6	0.2	0.1	9.48	2.60	20.01	—
Day 3	4.6	1.7	7.6	5.3	1.47	0.69	0.20	0.05
Day 8	0.6	0.1	0.6	0.5	0.34	0.11	0.10	0.04
Day 22	0.2	0.1	0.2	0.1	0.15	0.17	ND	—
Day 49	ND[2]	—	ND	—	ND	—	0.02	0.01

[1]Residues in aquatic plants were 100, 20, and 40 µg/kg fresh weight for Days 8, 22 and 49. Plot size 1 ha, depth 1.2 m.
[2]ND—Not Detected.

Six rates of terbutryn ranging from 3.1 to 9.4 kg a.i./ha of water surface were applied at each of three locations on Buckhorn Lake (Lake plots 7-12) in an attempt to study the feasibility of cutting channels through a major lake infested with watermilfoil (*Myriophyllum* sp.), filamentous green algae (*Spirogyra* sp.), and waterweed. Each area treated was 400 m^2 and the calculated concentration of terbutryn in the water was between 50 and 400 µg/L.

Experiments in 1977. Ponds 11, 12 and 13 were retreated in 1977 on a continuous flow basis over a 7 day period, 10 to 17 June. A 2% flowable formulation was metered into the three ponds over a 6 days 20 hr period, i.e. the complete turnover time for water in the ponds. The total volume of the three was calculated at 4563 m^3 and terbutryn was released at 11.6 ml of the 2% flowable formulation/minute to provide a calculated overall concentration of 50 µg/L in a 24 hr period and maintained according to flow rate for the 6 day 20 hr period. The aquatic plants included submersed macrophytes (*Ceratophyllum demersum* L.) and floating aquatic plants (*Lemna* sp. and *Wolffia* sp.).

Collecting Samples

Composite water samples were collected by lowering a narrow-necked litre glass jar, in a weighted metal can, quickly to the bottom and raising the same at a measured rate to allow the jar to be completely filled by the time it was raised to the surface. A minimum of four such samples were taken per 0.1 ha of surface area for residue analysis. These were capped underwater and retained on ice for transport to the laboratory the same day.

Sediments were collected using a standard 15 cm square brass Eckman dredge, which collected the top 4 to 5 cm of pond sediment. Water collected in the dredge was decanted with minimal disturbance of the sample. The mud was placed in heavy duty black polyethylene bags, tagged and placed on ice for immediate transport to the laboratory. Usually four such grab samples, randomly taken from the test site, were combined to provide a standard one kilogram mud sample for residue analysis.

Control Assessment

All ponds and lake plots were assessed for aquatic plants before and after treatment. Species were identified and an estimate of surface and bottom area being occupied was made for each species. At varying intervals throughout the season the treated areas were visited and visually assessed for control.

Analytical Procedure

Water (1.0 L) was extracted with two successive 100 mL portions of chloroform after adjusting the pH to 9 with dilute ammonia. The chloroform extract was dried by passage through dry absorbent cotton and the combined extracts were evaporated to about 2 ml; 10 ml of iso-octane was added and further evaporated to dryness. The dried extract was taken up in methanol. The method followed was only a slight modification of that described by Ramsteiner *et al.* (1974) and Sirons *et al.* (1973). Dried sediments and plant tissues were prepared and extracted as described for soils by Sirons *et al.* (1973) for other triazines.

Terbutryn was measured by gas-liquid chromatography (GLC) using a column packed with 5% Carbowax 20M on 80/100 mesh Varaport 30. The column temperature was 210 C. The carrier gas was helium flowing at 50 ml/min. The gas-liquid chromatograph was equipped with a Coulson conductivity cell operated in nitrogen mode. Terbutryn was recovered from spiked samples with an 80 to 90% efficiency. The detection limit was 0.04 µ/L. Data were presented uncorrected.

RESULTS

Experiments in 1974

In ponds 1, 2, 3, and 4, water samples taken 2 hrs after application showed no differences between ponds, and the concentration of terbutryn averaged 41 ± 13 µg/L (Table 2). In pond 1, all algae and emerged plants were no longer visible on 22 August, or 14 weeks after treatment. Cattails and bulrushes (*Scirpus validus* Vahl) that occupied the perimeter were unaffected. Residues of terbutryn in the water immediately after treatment was 18 µg/L and the level increased to 37 µg/L by day 11; thereafter it declined. At the same time, the dried mud from the pond bottom had residues of 6.2 µg/g immediately following distribution of the terbutryn, and declined to 0.72 µg/g at day 11, 0.30 µg/g by day 28 and 0.13 µg/g by day 48.

In pond 2 where 95% of the surface area was covered with algae mats, the water was free of aquatic plants by 18 June, 12 days after treatment and no apparent adverse effects were observed. By July 6, about 5% of the surface was infested by *Rhizoclonium* sp., and this increased until 13 August when they covered 35% of the water surface. Terbutryn residues in the sediment 45 days after treatment were 0.02 μg/g.

In pond 3, where the entire surface water was covered with *Cladophora* sp. and *Chara* sp., the water was virtually free of such vegetation on 14 June, 7 days after treatment. *Chara vulgaris* L. were not entirely dead until 9 July. The initial terbutryn concentration measured in water was 50 μg/L (Table 2). The pond remained free of aquatic plants throughout the summer, however by 1 September, *Rhizoclonium* sp. were evident. Analyses of the dried sediment 48 days after treatment revealed a residue of 0.28 μg/g.

By 9 July, 34 days after treatment of pond 4, *Chara* sp. were dead and decomposing except for a few plants around the input zone. Residues could be detected 48 days after treatment.

Analysis of dried sediments for the four ponds 12 months after application revealed a mean residue of 0.11 ± 0.13 μg/g.

In ponds 5, 6, 7 and 8, submersed aquatic plants were being visually controlled within 10 days of application and complete death of vegetation was achieved 17 days following treatment. Water remained free of aquatic plants for the remainder of the season.

In the lake plots 1 to 4, initial concentrations in water had not attained an equilibrium condition 0.5 hrs after treatment (Table 3). Water contained from 22 to 52 μg/L of terbutryn (mean 37 ± 12 μg/L) while sediments ranged from 19 to 44 μg/g or a mean of 28 ± 11 μg/g. Control of a mixed array of vascular submersed plants was nonexistent even with 400 μg/L of herbicide. This was probably due to the lateness of the application (25 June) when plants were of advanced maturity and the fact that water was moving across the plots.

Experiments in 1975

In pond 9, where the water was static, aquatic plants exhibited signs of injury 7 days following treatment. By 25 July, 28 days after treatment, no plants were evident near the surface and only decaying plant material was noted on the pond bottom. The pond was still clear of vegetation on 8 August. The residue level of terbutryn was followed in both water and sediment for 41 days (Table 4). Terbutryn took several days to reach an equilibrium condition and then declined in water and sediment very slowly. The variation of residue levels in the sediment reflected variations in the distribution of terbutryn over the pond surface.

In the beach plot (Lake 5) on Doe Lake, the terbutryn treatment had little effect on yellow water lily and caused only minor damage to tape grass, but had a marked effect on *Potamogeton* sp. 11 days after treatment. By 7 August or 35 days after treatment, the overall effect on all three groups of plants was negligible. The lack of control was attributed to water currents moving through the treated area. This was confirmed by the analyses of water and dried sediment when residues of only 1.0 ± 0.5 μg/L and 0.06 ± 0.02 μg/g were respectively measured within a few hours of treatment.

In the plot on Puslinch Lake (Lake 6), terbutryn caused the death of aquatic plants in the treated area within 12 days of application, but not outside the area although residues were found in both water and sediment (Table 5). On a fresh weight basis, dying aquatic plants contained 0.10 μg/g terbutryn. Over the 49 days of the study, terbutryn residues declined in water from 40 μg/L to a level of less than 0.1 μg/L, and in sediment from a high of 9.48 μg/g to less than 0.01 μg/g to less than 0.01 μg/g. While adjacent waters contained higher residues on day 3, these declined at the same rate as the treated area. Very small amounts of terbutryn accumulated in sediments outside the treated area.

Experiments in 1976

In pond 10, filamentous green algae was controlled by applying terbutryn to give a calculated concentration of 50 μg/L; however, by mid August a wide variety of phytoplankton species had recolonated the pond. The second treatment on 24 August resulted in a very low terbutryn concentration (0.8 μg/L) and was insufficient to provide the required control.

Curlyleaf pondwood remained healthy in the untreated head pond 11; while in ponds 12 and 13, except for the inlet, this species was eliminated by day 23. Mean concentrations of terbutryn in water of the two ponds appear in Table 6. Concentrations declined slowly as the two ponds flushed over a 6 day 20 hr period. By day 5 water residues had rapidly declined as one complete emptying of the system approached. Another marked decline occurred at day 16 following two complete flushings of the system. Residues in sediment were variable, probably reflecting uneven distribution of granules on the pond bottom as a result of the difficulty experienced in keeping the boat on station and the lateral movement due to the strong current passing through the ponds. Total control of filamentous green algae occurred within 9 days of the treat-

TABLE 6. RESIDUES OF TERBUTRYN IN WATER AND DRIED SEDIMENT IN PONDS 12 AND 13 FOLLOWING TREATMENT AT 100 μG/L ON 2 JUNE, 1976.[1]

	Terbutryn			
Days after treatment	Water (μg/L) Mean	Water (μg/L) SD	Dry sediment (μg/g) Mean	Dry sediment (μg/g) SD
Pre treatment (2 June, 1976)	ND	—	ND	—
Post treatment				
Day 1	22.4	8.8	1.75	1.27
Day 2	23.8	14.3	0.57	0.30
Day 5	4.1	4.1	2.44	1.97
Day 9	2.2	4.1		
Day 16	0.30	0.14	0.32	0.18
Day 23	0.15	0.07	0.77	1.07

[1]Pond 12 was 1.9 m deep and had surface area of 2388 m^2. Pond 13 was 2.5 m deep and covered 8200 m^2.

ment but re-infestation of this species occurred 21 days after treatment. Although waterweed seemed significantly retarded, a complete kill of this species was not observed. No fish mortality occurred in any of these trials.

The six treatments of terbutryn, which were replicated three times in Buckhorn Lake (Lake 7-12), were observed 1, 2, 3, 4 and 9 weeks following treatment; however, terbutryn failed to control the major plant, water milfoil or minor infestations of *Spirogyra* sp. Analyses of water at day 6 revealed concentrations of 22 μg/L from the highest treatment and 8 μg/L from the lowest. At day 14 the highest level was 122 μg/L and the lowest 4.3 μg/L. The respective calculated water concentrations were 400 μg/L for the highest and 100 μg/L for the lowest.

Experiments in 1977

Concentrations of terbutryn showed a steep gradient across the sequence of the three ponds for days 4 and 7 (Table 7) while the drip system of injection was being operated for 6 days and 20 hrs. By day 10, residues in pond 11 had declined as flushing removed the treated water. The highest concentration was in the second pond. A further decline occurred by day 12, or after one complete flushing of the system. Control of coontail (*Ceratophyllum demersum* L.), and *Lemna trisulca* L. and *Wolffia columbiana* Karst was only partial. There was no observable effect on trout using this drip technique. When the same ponds were treated in 1976 using granular herbicide, the flow of water was interrupted for 10 days and fish suffered oxygen stress following plant collapse and emergency aerators were put into operation.

DISCUSSION

Aquatic plant control in bodies of water over the duration of the study varied depending primarily on the flushing rate of the treated area and to a lesser extent on (a) initial rate of application (b) abundance of vegetation, (c) species of aquatic flora and (d) date of treatment.

In general, control was good under static water conditions be this the natural state or induced by flow interruption. A period of 10 days was required to give adequate time to destroy most vegetation. This period depleted oxygen levels in the water which affected fish survival. In open waters of lakes, movement out of the treated area by currents resulted in poor control.

TABLE 7. A SERIES OF THREE PONDS WERE TREATED WITH A CONTINUOUS FLOW OF 2% FLOWABLE TERBUTRYN FOR 9 DAYS, THE PERIOD REQUIRED TO FLUSH THE WHOLE SYSTEM.[1]

	Water (μg/L)		
Day after commencement	Inlet Pond 11	Mid-Pond Pond 12	Outlet Pond 13
Day 4	34.8	15.7	6.2
Day 7	46.5	33.7	16.2
Day 10	15.1	23.2	20.0
Day 12	9.4	17.7	15.0

[1]Treatment started 10 June, and ended 19 June 1977. Total volume of ponds 11, 12 and 13 was 43,637 m^3.

Where water movement was minimal, application at the rate of 50 μg/L of terbutryn controlled the plant growth within 12 days. By doubling this rate, control occurred within 7 days. Although the aim was to obtain 100 μg/L in water, this was never attained and most often it was no higher than 50 μg/L. This level was only one five hundredth of the reported solubility of 25 x 10^3 μg/L at 20C (WSSA 1979).

The abundance of vegetation and the species present affected the dergree of control. *Chara* required treatment at 100 μg/L to give control and took up to 2 to 3 weeks to be effective depending on the abundance of the algal mats. *Rhizoclonium* sp. appeared to recolonate ponds cleared of susceptible species.

In general, the filamentous algae were the most easily controlled, *Chara* being the least susceptible algae to terbutryn. On the other hand, emerged plants were difficult to affect and were not controlled even with the highest rates of application of 400 μg/L. The submersed vascular aquatic plants were intermediate in their susceptibility and appeared to require longer periods of contact than the algae.

Disappearance of terbutryn from water and sediment were studied and found to vary with the system. Persistence varied from little or no disappearance after 41 days to rapid disappearance in a few days. It was not possible to determine whether disappearance was from degradation or dispersal. None of the metabolites reported by Muir (1980) were determined.

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