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Residues of Fluridone and a Potential Photoproduct (*N*-Methylformamide) in Water and Hydrosoil Treated with the Aquatic Herbicide Sonar

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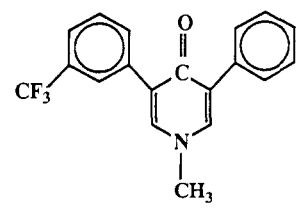
Fluridone is the active ingredient in the aquatic herbicide Sonar. Two ponds in Florida were treated with Sonar AS (an aqueous suspension formulation) and Sonar SRP (a slow-release clay pellet formulation). Both ponds were treated at the maximum acceptable residue level for fluridone in potable water, 0.15 ppm. The dissipation of fluridone and the potential formation of *N*-methylformamide (NMF) as a photolysis product of fluridone were monitored. The fluridone concentration decreased to a nondetectable level (less than 0.001 ppm) in the water of both ponds 324 days after treatment (DAT). NMF was not detected in any of the 192 water samples that were collected on any of the sampling dates at a detection limit of 0.002 ppm. Hydrosoil samples collected at 324 DAT in both ponds contained fluridone residues equivalent to 2.9–3.6% of the amount applied to the pond, but no NMF was detected in the hydrosoil at a detection limit of 0.005 ppm.

Fluridone, 1-methyl-3-phenyl-5-[3-(trifluoromethyl)-phenyl]-4(1*H*)-pyridinone (I), is the active ingredient in the aquatic herbicide Sonar. The biological, chemical, and physical properties of fluridone have been reviewed (West, 1984), and the bioconcentration and field dissipation of the herbicide in aquatic environments have been summarized (West et al., 1983).

A laboratory aqueous photolysis study indicated that *N*-methylformamide (NMF, II) is one of several low molecular weight photoproducts of fluridone in the absence of aquatic substrates other than water (Saunders and Mosier, 1983). However, NMF was not observed in studies conducted outdoors in artificial ponds with radiolabeled fluridone under natural environmental conditions (Berard and Rainey, 1981). Studies conducted with Sonar AS (an aqueous suspension formulation containing fluridone) in two ponds in Florida have also demonstrated that NMF was not a degradation product, even at fluri-



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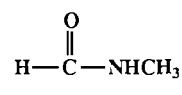

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Table I. Pond Descriptions, Sonar Treatments, and Water Characterization Data[a]

	pond		
	Sonar AS	Sonar SRP	control
pond size, ha	0.73	3.12	4.05
av depth, m	1.24	1.60	1.16
fluridone treatment,[b] kg/ha	1.86	2.35	0.0
total P, mg/L	0.012	0.012	0.059
total N, mg/L	0.52	0.45	2.00
pH	6.4	6.9	6.1
Ca, mg/L	5.6	12.0	4.0
Mg, mg/L	0.31	0.80	0.54
total alkalinity,[c] mg/L	13.0	33.0	10.0
chlorophyll *a*, mg/L	6.10	0.90	75.0
conductivity, μmho at 25 °C	35.0	74.0	28.0
total suspended solids, mg/L	3.2	1.0	4.4
org suspended solids, mg/L	2.2	0.5	3.6
inorg suspended solids, mg/L	1.0	0.5	0.8

[a] Pretreatment values on Oct 23, 1987. [b] The kilogram per hectare rate was based upon the amount of active ingredient (fluridone) required to yield a nominal water concentration of 0.15 ppm in the treated ponds. [c] Milligrams per liter calcium carbonate.

done concentrations that were 4.5 times greater than the acceptable residue level for fluridone in potable water (Osborne et al., 1989).

Since NMF doses of greater than 10 mg/kg are known to produce a teratogenic response in pregnant rabbits (Kennedy, 1986), it was important to confirm that NMF was not present in ponds treated with commercial Sonar formulations. Consequently, a residue method was developed (West and Turner, 1988) so that the potential formation of NMF could be monitored under actual field conditions.

EXPERIMENTAL SECTION

Pond Descriptions and Treatments. In order to maximize the concentration and persistence of fluridone, ponds were treated rather than lakes or canals (West et al., 1983). Three ponds near Lake City, FL, were selected that had not been treated with Sonar within the last 5 years. The ponds were fully contained with no outflow of water, so that the dissipation of fluridone could be attributed primarily to degradation rather than dispersal and dilution. The ponds contained aquatic vegetation, but the weeds did not completely cover the surface of the water so that the aqueous photolysis of fluridone could readily occur.

A description of the ponds and their treatments is summarized in Table I. Treatment 1 in this study consisted of an application of Sonar AS (an aqueous suspension) at a rate of 1.86 kg of fluridone/ha. Treatment 2 consisted of an application of Sonar SRP (a slow-release clay pellet) at a rate of 2.35 kg of fluridone/ha. These rates were 66 and 40% above the recommended label rates for the AS and SRP formulations, respectively. The rates were increased in order to obtain a theoretical fluridone concentration of 0.15 ppm, which is the maximum acceptable residue level for fluridone in potable water (*Fed. Regist.*, 1986).

Both Sonar formulations were applied to the surface of the water in the appropriate ponds on Oct 30, 1987. The AS formulation was applied by handgun using an airboat-mounted spray system that incorporated a 38 L/min diaphragm pump and continuous bypass and mechanical agitation. The sprayer was calibrated prior to application to deliver 514 L/ha using a 4-m swath at a boat speed of 5 km/h. Polycontrol (a drift control agent) was added to the spray mix at 0.5% (v/v). Sonar SRP was applied with a 12-V centrifugal spreader mounted on an airboat and calibrated to apply the appropriate amount of herbicide with a 12-m swath at a boat speed of 5 km/h.

The third pond in this study was an untreated control. The characteristics of the water in the three ponds are summarized in Tables I and II, and the light extinction coefficients are listed in Table III.

Water Sample Collection. Water samples were collected prior to treatment on three separate dates to determine background levels or interference levels of fluridone and NMF. Water samples were not collected on the date of application to allow for better equilibration of the fluridone concentration. Water samples were collected daily for 1–7 days after treatment (DAT), weekly for the next 3 weeks, every other week for 4 weeks, and then monthly (Table IV). The samples were collected from four stations within each pond on each sampling date. Two separate sampling stations were located in shallow shoreline areas, and two additional stations were located in the deepest part of the ponds.

All water samples were collected within 2.5 cm of the water surface, where maximum photolysis of fluridone was expected to occur. The samples were collected in 1-L Nalgene bottles (leak-proof amber-colored HDPE for protection of light-sensitive compounds, designed for storing and shipping samples). The water samples were transported in ice chests to the Center for Aquatic Plants in Gainesville, FL, where they were stored at 5 °C. The samples were frozen prior to shipment to the analytical laboratory, and they were packed with "freezy cubes" prior to shipment via overnight express delivery.

Hydrosoil Sample Collection. Hydrosoil samples were collected from the treated and the control ponds at 324 DAT. Ten replicate hydrosoil subsamples were collected randomly from each pond to a soil depth of approximately 10 cm with use of a soil sampler probe having an inside diameter of 10 cm. The hydrosoil samples were stored and shipped in the same manner as described above for the water samples.

Analytical Procedures. (*A*) *Water.* Upon receipt at the analytical laboratory, the water samples were stored at 4 °C until analyzed. On the day of analysis, the samples were allowed to warm to room temperature before analysis for fluridone and NMF by a method developed for this purpose (West and Turner, 1988). Briefly, the method involved the passage of a 50-mL aliquot of filtered water sample through a Sep-Pak C_{18} cartridge (Waters Associates) to extract fluridone, while NMF passed unretained through the cartridge with the water. Fluridone was eluted from the cartridge with methanol, which was then concentrated for analysis by high-performance liquid chromatography with UV detection at 313 nm. The water eluate containing NMF was concentrated for analysis by rotary vacuum evaporation at 40–46 °C. Methanol was added to aid the evaporation of the water, and approximately 30 μL of glycerol (glycerin) was added to retain NMF in the flask during the evaporation. The residual NMF was dissolved in methanol for analysis by gas chromatography with a Hall electrolytic conductivity detector operated in the nitrogen mode. The assay limits of detection were 0.001 ppm for fluridone and 0.002 ppm for NMF. These limits of detection have been fully validated and documented in our previous publication (West and Turner, 1988).

(*B*) *Hydrosoil.* Upon receipt at the analytical laboratory, the 10 replicate hydrosoil subsamples were composited and mixed to form a homogeneous sample from each pond. The composited hydrosoil samples were analyzed for fluridone with an existing method that utilized HPLC with UV detection (West, 1984). The hydrosoil samples were also analyzed for NMF by a modification of the method for the determination of NMF in water (West and Turner, 1988). Due to its extremely high water solubility, NMF was found to be extractable from hydrosoil with water. A representative 50-g hydrosoil sample was extracted with 100 mL of water by shaking on a gyratory shaker at 250 rpm for 30 min. A 50-mL aliquot of the extract was then filtered into a graduated cylinder, and the aqueous extract was analyzed in a manner identical with that described above for water samples.

Analytical Recovery Efficiencies. Analytical recovery efficiencies were determined in the laboratory during the analysis of each set of field samples. The recovery samples were prepared just prior to sample analysis by fortifying an aliquot of untreated control pond water with 0.025 ppm of fluridone and NMF and analyzing these recoveries simultaneously with the field samples. The analytical results for the field samples were corrected for the level of recovery efficiency obtained on a given analysis date.

Table II. Water Temperature (°C), Dissolved Oxygen (DO, ppm), pH, and Turbidity (Turb,[a] NTU) at Each Sampling Time

	Sonar AS pond					Sonar SRP pond					control pond				
date	time	temp	DO	pH	Turb	time	temp	DO	pH	Turb	time	temp	DO	pH	Turb
10/23/87	12:20	16.0	5.5	6.4	NA[b]	11:15	18.0	9.0	6.9	NA	10:20	15.0	7.0	6.1	NA
10/30/87	8:45	15.0	7.5	7.1	1.9	9:39	18.0	8.7	6.8	1.0	10:40	16.0	9.0	6.4	4.5
10/31/87	11:12	15.5	6.8	6.2	1.5	10:31	18.0	11.0	7.5	1.4	9:49	14.7	8.2	5.2	1.8
11/01/87	11:09	16.5	8.5	7.1	1.6	10:30	18.0	12.0	7.5	1.4	9:57	16.0	10.0	7.0	1.5
11/02/87	10:57	18.5	8.3	5.2	1.6	10:15	19.5	11.0	7.2	1.4	9:37	16.5	9.3	6.0	3.0
11/03/87	10:40	19.0	6.5	6.0	1.7	10:04	20.0	11.6	7.1	2.2	9:20	18.2	7.4	5.9	2.5
11/04/87	10:50	20.5	10.2	6.1	1.5	10:07	21.0	11.7	6.9	2.0	9:30	19.5	11.0	5.9	1.5
11/05/87	10:55	20.0	8.8	6.0	1.2	10:15	21.0	10.8	7.1	1.5	9:45	19.0	9.5	6.0	2.2
11/06/87	10:50	16.5	8.4	6.1	1.3	10:11	19.5	11.0	6.9	0.7	9:33	18.0	10.2	6.9	1.3
11/13/87	10:55	13.0	8.5	5.6	1.1	10:17	16.0	12.0	7.1	0.8	9:36	14.5	9.8	5.2	1.3
11/20/87	11:04	16.0	11.0	6.4	1.4	10:17	17.0	13.1	7.6	0.7	9:35	14.5	13.5	6.4	1.4
11/25/87	10:14	15.5	11.8	6.5	1.4	9:36	16.5	15.6	7.5	1.4	9:03	14.5	12.2	6.8	1.0
12/11/87	11:38	16.0	13.4	6.4	1.0	11:05	17.0	15.4	7.3	0.9	10:10	14.0	15.2	6.7	1.2
12/22/87	9:00	13.0	6.0	7.1	1.2	8:10	16.5	8.5	7.0	1.7	7:30	15.0	8.3	6.5	1.9
01/20/88	11:20	16.0	16.0	6.4	0.8	10:30	18.2	18.2	7.1	0.9	9:55	17.2	16.9	7.2	1.9
02/17/88	11:25	11.0	12.5	7.3	1.0	10:52	12.0	14.9	7.5	1.2	9:58	9.0	14.0	7.1	0.9
03/16/88	11:50	14.8	7.7	7.8	1.7	11:04	16.0	12.0	7.3	1.0	10:26	14.2	10.8	7.5	1.1
04/14/88	11:10	17.5	7.8	6.5	1.4	10:36	19.0	10.2	7.2	1.1	10:10	16.5	12.1	6.9	1.0
05/18/88	11:36	NA[b]	NA	NA	1.6	10:59	NA	NA	NA	1.6	10:30	NA	NA	NA	1.5
06/15/88	11:40	27.0	10.0	6.7	1.4	11:05	27.0	10.2	7.2	1.0	10:36	22.5	9.4	6.2	1.6
07/22/88	11:30	30.0	6.5	6.5	3.4	10:58	31.0	6.2	6.4	2.0	10:22	26.8	5.3	6.5	1.5
08/19/88	11:30	30.9	11.2	7.1	2.5	10:55	31.0	8.3	6.4	2.0	10:30	22.5	7.4	6.9	6.5
09/16/88	12:46	29.5	11.2	7.1	2.9	10:40	29.0	11.4	7.2	3.4	9:30	26.0	10.4	7.3	1.3

[a] Nephelometric turbidity units. [b] Information not available due to sample loss.

Table III. Light Extinction Coefficients for Pond Water

	extinction coefficient[a]		
date	Sonar AS pond	Sonar SRP pond	control pond
11/03/87	1.00	0.30	1.11
11/04/87	1.01	0.44	0.62
11/05/87	0.92	0.44	0.64
11/06/87	1.06	0.53	0.71
11/07/87	0.81	0.46	1.08
11/20/87	0.83	0.45	0.95
11/25/87	0.70	0.44	0.88
03/16/88	0.73	0.35	0.48
05/18/88	0.65	0.38	0.74
06/15/88	0.62	0.42	0.84
08/19/88	0.87	0.58	NA

[a] $I_z = I_0 e^{-nz}$, where I_z = light intensity at depth z and I_0 = light intensity at surface.

Storage Stability. Since water samples were shipped frozen and then refrigerated prior to analysis, a storage stability study was conducted to determine the stability of the analytes during storage. The stability samples were prepared from the pretreatment water samples collected from the two ponds that were subsequently treated with Sonar. Pretreatment samples (approximately 700 mL) from each of the four sampling stations in both ponds were fortified with 30 μg of both analytes in the same type of amber Nalgene containers that were used for the field samples. Aliquots of the fortified stability samples were removed for a zero-time assay, and the remainder of each sample was frozen at −20 °C. After 1 day, the samples were thawed for analysis and the remainder of each sample was stored at 4 °C for the duration of the stability study. The samples were periodically removed from refrigeration for analysis to determine stability.

RESULTS AND DISCUSSION

Dissipation of Fluridone. The concentration of fluridone in pond water treated with Sonar AS and Sonar SRP is summarized in Table IV. Fluridone was not detected in any of the pretreatment or control samples collected during the course of the study. In the pond treated with Sonar AS, a maximum fluridone concentration of 0.122 ppm was observed, with a maximum average concentration of 0.109 ppm for the samples collected from the four sampling stations. The fluridone concentration decreased very slowly during the late fall and winter months in northern Florida, with a half-life of approximately 3 months. The average half-life in treated pond water is 3 weeks for applications made during the spring, summer, or early fall (West et al., 1983). It has been noted previously that the half-life of fluridone in pond water is related to the time of the year when the herbicide is applied (Muir and Grift, 1982; West et al., 1983), with the half-life increasing with later application dates. Thus, it is likely that the increased half-life of fluridone that was observed in this study was due to the Oct 30 application date.

In the pond treated with the slow-release pellet (SRP), the fluridone concentration gradually increased to a maximum average of 0.032 ppm at 111 DAT. The SRP formulation is designed to maintain a low, steady concentration of fluridone over a period of time. In this pond, the concentration of fluridone was maintained at approximately 0.02–0.03 ppm from 26 to 168 DAT. In both ponds, the fluridone concentration declined to a nondetectable level (less than 0.001 ppm) at 324 DAT.

In the pond treated with Sonar SRP, no significant difference was observed in the fluridone concentrations in the water samples collected from the deep and shallow portions of the pond. For the pond treated with Sonar AS, the concentration of fluridone in the shallow stations averaged 109% of that in the deep sampling stations up through 6 DAT. After 6 DAT, equilibration had occurred within the pond and the concentration in the shallow stations averaged 99% of those from the deep stations. For this reason, only ranges, means, and standard deviations are reported for the fluridone concentrations in Table IV. Representative chromatograms for the determination of fluridone in pond water are contained in Figure 1.

The water chemistry data (Tables I and II) indicated that the ponds in this study were typical of mesotrophic bodies of water in Florida (Canfield and Moyer, 1988). The light extinction coefficients (Table III) were also typical and indicated that sufficient water clarity existed for the photodegradation of fluridone.

Table IV. Concentrations of Fluridone and *N*-Methylformamide[a] in Pond Water following Applications of Sonar AS and Sonar SRP at 0.15 ppm Fluridone

	fluridone concentration,[b] ppm					
	Sonar AS pond			Sonar SRP pond		
DAT[c]	range	mean	SD	range	mean	SD
pre[d]	ND[e]	ND		ND[e]	ND	
1	0.070–0.092	0.077	0.011	ND–0.004	0.002	0.002
2	0.083–0.114	0.095	0.015	0.002–0.004	0.003	0.001
3	0.086–0.119	0.099	0.014	0.002–0.003	0.003	0.001
4	0.102–0.122	0.109	0.009	0.003–0.005	0.004	0.001
5	0.081–0.118	0.095	0.016	0.003–0.005	0.004	0.001
6	0.097–0.112	0.116	0.007	0.006–0.009	0.007	0.001
7	0.084–0.098	0.090	0.006	0.001–0.009	0.005	0.004
14	0.089–0.093	0.091	0.002	0.011–0.014	0.013	0.002
21	0.078–0.088	0.082	0.005	0.013–0.019	0.015	0.003
26	0.064–0.076	0.072	0.005	0.020–0.027	0.022	0.003
43	0.064–0.079	0.070	0.010	0.027–0.030	0.029	0.001
54	0.048–0.066	0.061	0.009	0.019–0.025	0.021	0.003
83	0.053–0.058	0.055	0.002	0.020–0.022	0.021	0.001
111	0.045–0.055	0.052	0.005	0.027–0.036	0.032	0.004
139	0.038–0.044	0.041	0.003	0.019–0.036	0.027	0.007
168	0.019–0.024	0.021	0.002	0.019–0.022	0.020	0.001
202	0.015–0.016	0.016	0.001	0.016–0.019	0.018	0.002
230	0.014–0.016	0.015	0.001	0.016–0.018	0.017	0.001
268	0.005–0.006	0.006	0.001	0.006–0.008	0.008	0.001
296	0.002–0.003	0.002	0.001	ND–0.002	0.002	0.001
324	ND	ND		ND	ND	

[a] No NMF detected in any samples at a detection limit of 0.002 ppm. [b] Range, mean, and standard deviation (SD) for four samples. [c] Days after treatment. [d] Twelve pretreatment samples collected on three separate dates. [e] No fluridone detected at a detection limit of 0.001 ppm.

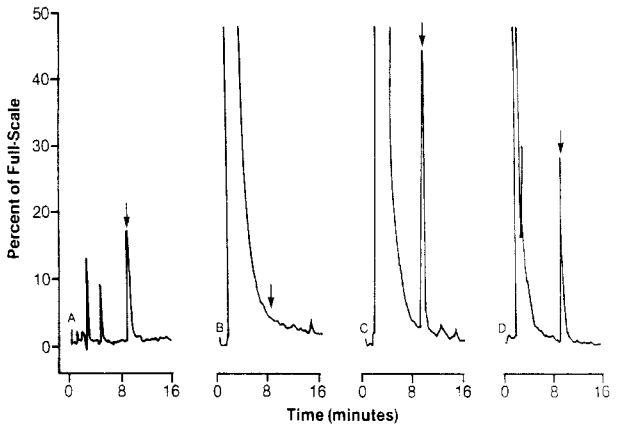

Figure 1. Representative chromatograms demonstrating the determination of fluridone in pond water: (A) fluridone standard, 50 ng; (B) untreated control pond water; (C) untreated control pond water fortified with 0.025 ppm fluridone (96% recovery); (D) pond water treated with 0.15 ppm fluridone, 43 days after treatment (0.078 ppm), diluted to 20-mL final volume.

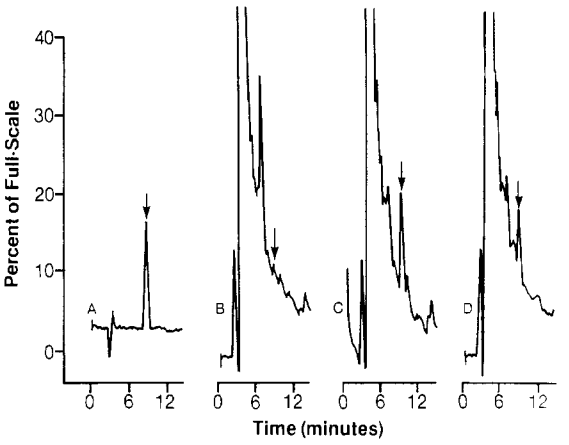

Figure 2. Representative chromatograms demonstrating the determination of fluridone in pond hydrosoil: (A) fluridone standard, 50 ng; (B) untreated control pond hydrosoil; (C) untreated control hydrosoil fortified with 0.10 ppm fluridone (81% net recovery); (D) pond hydrosoil 324 days after treatment with Sonar, containing a residue of 0.07 kg/ha.

The concentrations of fluridone in the hydrosoil at 324 DAT were 0.040 and 0.065 ppm in the ponds treated with Sonar AS and Sonar SRP, respectively. Converting the parts per million values to an equivalent kilogram per hectare rate (West et al., 1983), these residues were equivalent to 0.07 kg/ha in both ponds, which was equivalent to 3.6 and 2.9% of the initial application rates in the ponds treated with Sonar AS and Sonar SRP, respectively. These residue levels at 324 DAT were similar to those reported previously at 1 year after treatment, which ranged from none detected to 0.07 kg/ha (West et al., 1983). The low hydrosoil residues indicated that the dissipation of fluridone from the pond water during the course of this study was due primarily to degradation rather than adsorption onto hydrosoil. Representative chromatograms for the determination of fluridone in hydrosoil are contained in Figure 2.

Formation of NMF. NMF was not detected in any of the 192 water samples from the two treated ponds during the course of the study at a detection limit of 0.002 ppm (Table IV). Representative chromatograms for the determination on NMF in pond water treated with Sonar are contained in Figure 3. NMF was also not detected at a detection limit of 0.005 ppm in the hydrosoil samples that were collected 324 DAT. Representative chromatograms for the determination of NMF in hydrosoil are contained in Figure 4.

Hydrosoil samples were not collected and analyzed on the same frequent sampling schedule as was done for water, because NMF has not been demonstrated to form in hydrosoil. The hydrosoil samples were collected when fluridone was no longer present in the water to ensure that the dissipation of fluridone from the water was due primarily to degradation rather than adsorption onto hydrosoil. Thus, the water and hydrosoil analyses at 324 DAT demonstrated that fluridone in the water had degraded, but it did not degrade to NMF.

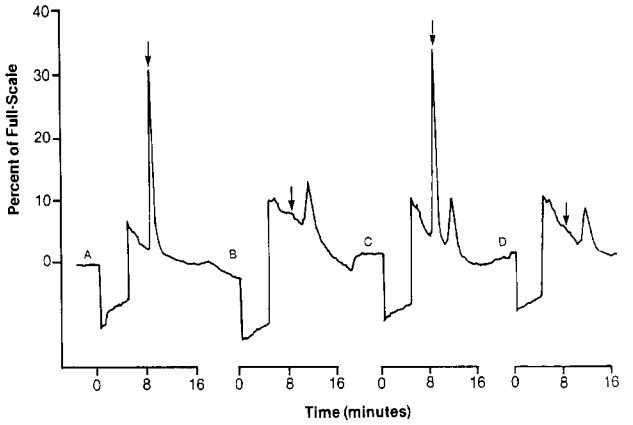

Figure 3. Representative chromatograms demonstrating the determination of NMF in pond water: (A) NMF standard, 0.25 ng; (B) untreated control pond water; (C) untreated control pond water fortified with 0.025 ppm NMF (85% recovery); (D) pond water treated with 0.15 ppm fluridone, 26 days after treatment, containing no detectable NMF.

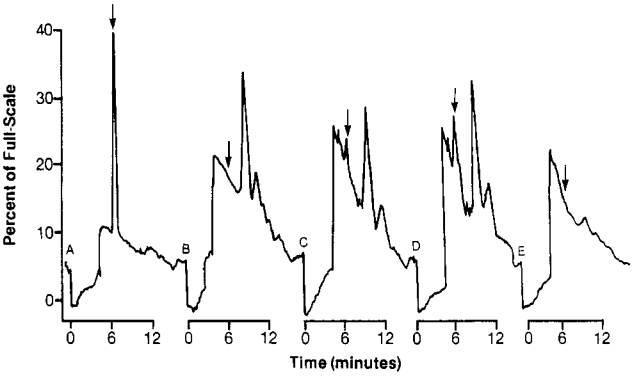

Figure 4. Representative chromatograms demonstrating the determination of NMF in pond hydrosoil: (A) NMF standard, 0.25 ng; (B) untreated control hydrosoil; (C) untreated control hydrosoil fortified with 0.005 ppm NMF (112% recovery); (D) untreated control hydrosoil fortified with 0.01 ppm NMF (74% recovery); (E) pond hydrosoil 324 days after treatment with Sonar, containing no detectable NMF.

Soil Adsorption and Octanol/Water Partition Coefficients. The solubility of NMF in water has been reported to be so great that NMF cannot be extracted by traditional methods for removing organic compounds from water (West and Turner, 1988). The high water solubility suggests that NMF would have a low tendency to accumulate in hydrosoil or fish. The extremely low potential for NMF to accumulate in hydrosoil or fish has been confirmed by the soil adsorption (K_d) and octanol/water partition coefficients (K_{ow}). A value of $K_{ow} = 0.12$ ($\log K_{ow} = -0.92$) was obtained for NMF, and a value of $K_d = 0.054$ was obtained for a loam soil with an organic content of 4.0% (Saunders, 1988). These data indicate that if NMF would have formed in the pond water, it would not have selectively partitioned into or accumulated in the hydrosoil or fish.

Analytical Recovery Efficiencies. Whenever a set of field samples was analyzed, the results were corrected for the level of recovery that was obtained for control pond water samples that had been fortified with 0.025 ppm each of fluridone and NMF. The laboratory recovery efficiencies that were obtained for fluridone and NMF in water averaged 89 ± 5 and 80 ± 11%, respectively (n = 66). The recovery of fluridone from control hydrosoil fortified with 0.1 ppm fluridone averaged 75 ± 8%, while the recovery of 0.005–0.05 ppm NMF averaged 94 ± 23%. Representative chromatograms demonstrating the recovery of fluridone and NMF from the fortified water and hydrosoil samples are contained in Figures 1–4.

Storage Stability. In the storage stability study, fluridone and NMF were both stable for at least 220 days of storage. Since all of the field samples were analyzed within 37 days of sample collection, the validity of the assay results in Table IV is supported by the stability data.

CONCLUSIONS

The data from this study indicate that NMF was not a degradation product of fluridone in natural aquatic environments treated with commercial formulations of Sonar under field use conditions. These results support those obtained previously from a radiolabeled fluridone study that was conducted outdoors in artificial ponds (Berard and Rainey, 1981) and from a study conducted with a commercial Sonar formulation in natural ponds at exaggerated application rates (Osborne et al., 1989). Fluridone degraded in all of the studies, but it did not degrade to NMF.

Registry No. I, 59756-60-4; II, 123-39-7.

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