

Fluridone, A New Herbicide For Aquatic Plant Management

MAX C. McCOWEN, C. L. YOUNG, S. D. WEST,
S. J. PARKA and W. R. ARNOLD

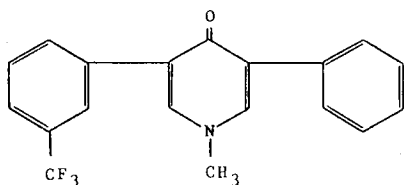
Lilly Research Laboratories
Division of Eli Lilly and Company
Greenfield, Indiana 46140

ABSTRACT

Fluridone, as a 480 g/liter aqueous suspension (4 lb/gal AS) or 2.5 percent granule at rates from 0.1 to 1.0 ppm a.i. total water treatment, applied over the water surface, directly under the water surface, or as a layered treatment to the bottom of the body of water, provided control of many submersed and emersed aquatic plant species. Fluridone did not have any adverse effect on water quality, water chemistry, or aquatic life present in the treated bodies of water.

INTRODUCTION

Fluridone (1-methyl-3-phenyl-5-[3-(trifluoromethyl) phenyl]-4(1H)-pyridinone) is a relatively new preemergence herbi-



$C_{19}H_{14}F_3NO$ fluridone^{1/} M.W. 329.3

1-methyl-3-phenyl-5-[3-(trifluoromethyl)phenyl]-4(1H)-pyridinone

^{1/}The Weed Science Society of American, the American National Standards Institute and the British Standards Institute have accepted the term fluridone as the common (generic) name for the compound.

cide for use on cotton (1, 2). Additional testing revealed that fluridone possessed high activity against aquatic vascular plants at low application rates. Consequently, additional laboratory and field studies were initiated to further evaluate the aquatic herbicidal properties of fluridone.

Technical fluridone is an off-white to tan, odorless crystalline solid. It melts at 151 to 154°C and the solubility in water is approximately 12 ppm. Approximate solubilities in organic solvents are as follows: methanol (>10 mg/ml), diethyl ether (>1 mg/ml), ethyl acetate (>5 mg/ml), chloroform (>10 mg/ml) and hexane (>0.5 mg/ml). The vapor pressure of fluridone is less than 1×10^{-7} mm Hg at 25°C. It is moderately susceptible to decomposition by ultraviolet irradiation in an aqueous solution. Fluridone is stable to hydrolysis at pH 3, 6, and 9.

Acute and 90-day subacute toxicological results for fluridone indicate that this compound has a low order of toxicity (Table 1).

Fluridone was evaluated for a period of three months in mice, rats and dogs. No treatment related effects were noted in mice at dietary doses of 200 ppm, in rats at dietary doses of 1400 ppm or in dogs at oral doses of 200 mg/kg/day.

Preliminary metabolism studies with the rat indicated that fluridone was excreted within 72 hours. More than 80 percent of an oral dose of fluridone was excreted in the feces, with a trace in the urine. Detailed investigation of the metabolism of fluridone in the rat is currently in progress.

Mode of action studies have demonstrated that fluridone

TABLE 1. ACUTE TOXICITY FOR FLURIDONE.

Species	Material	Route	Toxicity
Rat	Technical	Oral	LD ₀ >10,000 mg/kg
	4AS	Oral	LD ₀ >0.5 ml/kg
4AS	Technical	Subcutaneous	LD ₀ >500 mg/kg
	Technical	Inhalation	LD ₀ >9.6 ml/M ³ of air
Mouse	Technical	Oral	LD ₅₀ >10,000 mg/kg
	Technical	Subcutaneous	LD ₀ >500 mg/kg
Cat	Technical	Oral	LD ₀ >250 mg/kg
Dog	Technical	Oral	LD ₀ >500 mg/kg
Rabbit	4AS	Dermal	LD ₀ >2 ml/kg (slight irritant)
	4AS	Ocular	Very slight irritant
Mallard duck	Technical	Diet	LC ₀ >5,000 ppm
Bobwhite quail	Technical	Acute oral	LD ₀ >2,000 mg/kg
Bobwhite quail	Technical	Diet	LC ₀ >5,000 ppm
Bluegill	Technical	Water	LC ₅₀ 14.3 ppm
Rainbow trout	Technical	Water	LC ₅₀ 11.7 ppm
Daphnia	Technical	Water	LC ₅₀ 6.3 ppm

inhibits the biosynthesis of carotenoid precursors. Since the carotenoid pigments protect chlorophyll from photodegradation, the inhibition of their synthesis removes this protective system from the plant and the chlorophyll becomes susceptible to degradation (3, 4, 5).

MATERIALS AND METHODS

The aquatic herbicidal properties of fluridone were field tested at the Lilly Research Laboratories at Greenfield, Indiana, in 265 liter containers planted with the following aquatic plants: Hydrilla (*Hydrilla verticillata* Royle), Eurasian watermilfoil (*Myriophyllum spicatum* L.), Coon-

tail (*Ceratophyllum demersum* L.), Cabomba (*Cabomba caroliniana* Gray), Slender naiad (*Najas flexilis* Willd.), and common duckweed (*Lemna* spp.). In later field experiments Sago pondweed (*Potamogeton pectinatus* L.) was substituted for Cabomba.

Aquatic plants were allowed to become established for approximately two weeks before fluridone was applied to the water surface at total volume rates of 4.0, 2.0, 1.0, 0.5, and 0.25 ppm.

Mosquitofish (*Gambusia affinis* Baird and Girard) were added to each container to evaluate the effects of fluridone on the fish. Dissolved oxygen was recorded and water and hydrosol samples were collected for chemical analysis at frequent intervals during the experiments.

Laboratory studies were then conducted to determine the effects of fluridone on aquatic plants of the following species: Salvinia (*Salvinia* spp.) (Table 5), Bladderwort (*Utricularia* spp.) (Table 6), Vallisneria (*Vallisneria*, spp.) (Table 7), water hyacinth (*Eichornia crassipes* (Mart.) Solms.) (Table 8), and hydrilla tubers.

Field experiments were also conducted to evaluate the aquatic herbicidal properties of fluridone in Michigan and New York. The treated ponds ranged in size from 0.004 to

TABLE 3. THE CONTROL (%) OF SEVERAL AQUATIC PLANTS TREATED WITH FLURIDONE EVALUATED 8 WEEKS AFTER TREATMENT.

Fluridone (ppm)	Duckweed	Coontail	Watermilfoil	Hydrilla	Sago Pondweed	S. Naiad
0.03	100	95	100	100	100	100
0.10	100	100	100	100	100	100
0.30	100	100	100	100	100	100
Control	0	0	0	0	0	0

TABLE 2. THE EVALUATION OF FLURIDONE AT VARIOUS RATES ON CONTROL OF SEVERAL AQUATIC PLANTS AND THE RESIDUE ASSAYS OF WATER AND HYDROSOIL FOR FLURIDONE. ALSO THE DISSOLVED OXYGEN CONCENTRATIONS OF THE WATER AT VARIOUS RATES OF FLURIDONE WERE EVALUATED.

Fluridone (ppm)	Duckweed	Coontail	Watermilfoil	Hydrilla	Cabomba	Naiad	D.O. ^a (ppm)	Water ^b Assay (ppm)	Hydrosol Assay (ppm)
Percent Control—13 weeks									
2 Week Old Plants	4.0	100	100	100	100	100	5.8 5.2	0.14	0.44
	2.0	100	100	100	100	100	5.3 4.6	0.08	0.27
	1.0	100	100	100	100	100	5.5 5.8	0.02	0.14
Control	—	0	0	0	0	0	7.8 9.5	NDR	NDR
Percent Control—8 weeks									
6 Week Old Plants	1.0	100	97	90	100	85	7.9 7.3	0.19	0.02
	0.5	100	100	85	85	100	8.7 6.8	0.07	0.05
	0.25	100	100	85	85	100	7.5 7.8	0.03	0.04
Control	—	0	0	0	0	0	7.8 9.5	NDR	NDR

^a Dissolved oxygen at top and bottom respectively.

^b NDR indicates no detectable residue.

TABLE 4. AQUATIC PLANTS SUSCEPTIBLE TO FLURIDONE AT AN APPLICATION RATE OF 0.3 PPM.

Hydrilla	<i>Hydrilla verticillata</i> Royle
Common elodea	<i>Eleodea canadensis</i> Michx.
Fanwort	<i>Cabomba caroliniana</i> Gray.
Eurasian watermilfoil	<i>Myriophyllum spicatum</i> L.
Coontail	<i>Ceratophyllum demersum</i> L.
Illinois pondweed	<i>Potamogeton illinoensis</i> Morong
Floating pondweed	<i>Potamogeton natans</i> L.
Sago pondweed	<i>Potamogeton pectinatus</i> L.
Southern naiad	<i>Najas guadalupensis</i> (Spreng.) Magnus.
Common bladderwort	<i>Utricularia</i> spp.
Vallisneria (tapegrass)	<i>Vallisneria</i> spp.
Arrowhead	<i>Sagittaria</i> spp.
Cattail	<i>Typha</i> spp.
Bulrush	<i>Scirpus americanus</i> Pers.
Horsetail	<i>Equisetum</i> spp.
Reed canarygrass	<i>Phalaris arundinacea</i> L.
Common duckweed	<i>Lemna</i> spp.
Salvinia	<i>Salvinia</i> spp.

TABLE 5. LABORATORY EVALUATION OF THE EFFECTS OF FLURIDONE (4As) ON SALVINIA (*Salvinia* spp.).

Fluridone (ppm)	% Control 13 weeks after application
0.5	100
0.25	100
Control	0

TABLE 6. LABORATORY EVALUATION OF THE EFFECTS OF FLURIDONE (4As) ON COMMON BLADDERWORT (*Utricularia* spp.).

Fluridone (ppm)	% Control 6 weeks after application
2.0	95
1.0	95
0.5	93
Control	0

TABLE 7. LABORATORY EVALUATION OF THE EFFECTS OF FLURIDONE (4As) ON VALLISNERIA spp. (TAPEGRASS).

Fluridone (ppm)	% Control 6 weeks after application
0.3	100
0.1	95
0.03	95
Control	0

TABLE 8. LABORATORY EVALUATION OF THE EFFECTS OF FLURIDONE (4As) ON WATER HYACINTH.

Fluridone	% Control 14 weeks after application
1.0 lb/A	80
0.5 lb/A	40
0.25 lb/A	28
Control	0

0.69 hectares with water depths ranging from 0.60 to 1.3 meters. Fluridone was applied at rates of 0.1, 0.3, and 1.0 ppm, based on the total water column treated. Fluridone application rates, based on the amount of active ingredient

TABLE 9. DISSOLVED OXYGEN AND TEMPERATURE VALUES OF A POND (0.04 HECTARES) IN MICHIGAN FOLLOWING TREATMENT WITH FLURIDONE (4As) AT 1.0 PPM.

Days after application	D.O. (ppm)	Temp (C)
1	9.5	21.0
7	8.0	25.0
14	7.2	24.0
56	7.5	25.0
110	8.4	18.0

TABLE 10. LABORATORY EVALUATION OF THE EFFECTS OF FLURIDONE (4As) ON HYDRILLA TUBERS.

Fluridone (ppm)	% Chlorosis 5 weeks after application (No inhibition of germination)
0.3	100
0.1	0
0.03	0
Control	0

TABLE 11. FLURIDONE LEVELS (PPM) IN WATER HYDROSOIL.

Site	Days after application	Water	Hydrosoil
Michigan Rate 0.1 ppm	1	0.090	0.026
	7	0.021	0.220
	27	0.002	0.071
	54	<0.0005	0.035
New York Rate 0.3 ppm	1	0.109	—
	7	0.103	0.010
	28	0.027	0.057
	84	0.023	0.078

TABLE 12. FLURIDONE LEVELS (PPM) IN FISH FROM THE PONDS IN MICHIGAN FOLLOWING THE APPLICATION OF FLURIDONE AT THE RATE OF 0.1 PPM.

Days after application		Fluridone ^a ppm
1	Green sunfish (<i>Lepomis cyanellus</i>)	0.054 0.023
	Pumpkinseed sunfish (<i>Lepomis gibbosus</i>)	0.023
7	Largemouth bass (<i>Micropterus salmoides</i>)	0.010
	Black bullhead (<i>Ictalurus melas</i>)	0.010
27	Pumpkinseed sunfish (<i>Lepomis gibbosus</i>)	NDR
	Largemouth bass (<i>Micropterus salmoides</i>)	NDR
54	Black bullhead (<i>Ictalurus melas</i>)	NDR

a NDR = no detectable residue at a test sensitivity of 0.010 ppm.

per surface area treated, ranged from 0.9 kg/ha (0.1 ppm) to 12.8 kg/ha (1 ppm), depending upon the depth of the water column. Several application techniques were used to determine the effect of the mode of application. Fluridone, as a 480 g/liter aqueous suspension (4 lb/gal AS), was ap-

plied with a CO₂ pressure sprayer and a single nozzle held 10 to 15 cm under the water surface or near the bottom. A 2.5 percent granular formulation was applied with a rotary spreader to the water surface, and allowed to sink to the bottom.

Water quality was measured immediately prior to application and 1, 3, 7, 14, 21, 28, and 64 days following applications in Michigan and New York. Water temperature and dissolved oxygen were measured using a YSI meter. Samples of water, hydrosol, and fish were collected on the above schedule for residue analysis. Weed populations were mapped prior to applications and evaluations of the herbicidal activity against all species were made at the time water samples were collected.

RESULTS AND DISCUSSION

In the 265 liters container tests, fluridone provided excellent control of floating and submersed plants at the lowest dose tested (1 ppm) when plants were established for two weeks before treatment and 0.25 ppm when plants were established for six weeks before treatment (Table 2). Fluridone continued to provide excellent weed control at levels as low as 0.03 ppm (Table 3). Fluridone symptoms on submersed aquatic plants appeared as progressive albescence of young leaves followed by leaf necrosis. The symptoms began to appear three to six days after application but total control was fairly slow, occurring at two and four weeks after treatment. The aquatic plants gradually sank to the bottom and amount of open water increased. Table 4 lists the submersed aquatic plants and emerged or bank vegetation controlled in the laboratory and field experiments at Greenfield, Indiana; Michigan; and New York. As a result of the slow vegetation decline, there was little change in the dissolved O₂ content of the water (Tables 2, 9). Mosquitofish (*G. affinis*) survived and produced young at all rates of

fluridone. In laboratory studies, fluridone was found to affect new growth of hydrilla tubers by causing delayed germination and subsequent chlorosis (Table 10).

Fluridone did not affect the water quality parameters such as pH, BOD, color, dissolved solids, hardness, nitrate nitrogen, total phosphates, and turbidity.

Water, hydrosol, and fish samples were analyzed for fluridone. The results indicated that fluridone exhibited a half-life of 14 days or less in pond water. Factors contributing to the dissipation of fluridone from the water included rapid uptake by the aquatic plants, adsorption onto the hydrosol, and photodegradation (Table 11). Fluridone did not accumulate in fish (Table 12).

Observations of other aquatic life, including bluegills, bass, catfish, crayfish, frogs, and water snakes were made in the ponds prior to and during the studies. No adverse effects were observed on these species from any application.

ACKNOWLEDGMENT

The authors would like to express their thanks to Dr. G. S. Probst, Dr. J. L. Hamelink, and Mr. C. C. Kehr of the Lilly Research Laboratories, Greenfield, Indiana, for some of the data provided.

LITERATURE CITED

1. Waldrep, T. W. and H. M. Taylor. 1976. 1-Methyl-3-phenyl-5-[3-(trifluoromethyl)phenyl]-4(1H)-pyridinone, a new herbicide. *Agric. Food Chem.* 24:1250-51.
2. Anonymous (1976) Technical report on EL-171, an experimental herbicide. Lilly Research Laboratories, Indianapolis, IN.
3. Bartels, P. G. and C. W. Watson. 1978. Inhibition of carotenoid synthesis by fluridone and norflurazon. *Weed Sci.* 26:198-203.
4. Berard, D. F., D. P. Rainey and C. C. Lin. 1978. Absorption, translocation and metabolism of fluridone in selected crop species. *Weed Sci.* 26:252-254.
5. Devlin, R. M., C. N. Saras and M. J. Kisiel. 1978. Influence of fluridone on chlorophyll production. *ABSTR. Weed Sci. Soc. Am.*, p. 3.