Residues in Crops and Soils Irrigated with Water Containing the Aquatic Herbicide Fluridone

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Field studies were conducted to determine residue levels of fluridone, 1-methyl-3-phenyl-5-[3-(trifluoromethyl)phenyl]-4(1*H*)-pyridinone, in soil and 26 different crops irrigated with water containing the aquatic herbicide Sonar. Depending on the crop or location, the crops were sprinkler-, furrow-, or flood-irrigated with a total of 7.6–10.2 ha cm of water containing 0.123 ppm of fluridone. These irrigation volumes resulted in a cumulative soil application of 0.09–0.12 kg of fluridone/ha. Of 26 crops assayed at a detection limit of 0.05 ppm, fluridone was detected only in orchardgrass (0.11 ppm) and alfalfa (0.07 ppm). Of eight soil samples collected 0–12 months after irrigation, only two contained detectable levels of fluridone, and none contained detectable levels of a potential soil metabolite, 1,4-dihydro-1-methyl-4-oxo-5-[3-(trifluoromethyl)phenyl]-3-pyridinecarboxylic acid.

INTRODUCTION

Fluridone, 1-methyl-3-phenyl-5-[3-(trifluoromethyl)-phenyl]-4(1*H*)-pyridinone (I), is the active ingredient in the aquatic herbicide Sonar. Single annual applications of Sonar have resulted in the management of troublesome vascular aquatic weeds at low application rates (Parka et al., 1978; McCowen et al., 1979; Grant et al., 1979; Rivera et al., 1979; Arnold, 1979; Sanders et al., 1979). The bioconcentration and field dissipation of fluridone and its degradation products have been reported previously (West et al., 1979; Muir et al., 1980; West and Parka, 1981; Muir and Grift, 1982; West et al., 1983), and a mathematical model for predicting the half-life of fluridone in pond water has been developed (West et al., 1983).

Prior to the irrigated crop and soil study, it was necessary to characterize the nature of the residues that might be expected to occur in irrigated crops and soil. Uptake and metabolism studies with representative crops that were furrow- or sprinkler-irrigated with water containing [^{14}C]-fluridone have indicated that the parent compound was the primary residue and that no major metabolites had formed (Berard and Rainey, 1981, unpublished results). Also, an investigation of the uptake, translocation, and metabolism of [^{14}C]fluridone in corn, soybean, and cotton plants indicated that the parent compound was not metabolized by these crops (Berard et al., 1978).

Laboratory studies with [^{14}C]fluridone in water–sediment systems have resulted in the formation of a single major soil metabolite, 1,4-dihydro-1-methyl-4-oxo-5-[3-(trifluoromethyl)phenyl]-3-pyridinecarboxylic acid (II) (Berard and Rainey, 1981, unpublished results; Marquis et al., 1982; Muir and Grift, 1982). However, II has not been detected in the hydrosoil of outdoor ponds under natural conditions (Berard and Rainey, 1981, unpublished results; Muir and Grift, 1982; West et al., 1983).

On the basis of the results of the ^{14}C metabolism studies, it was determined that the only major residue in irrigated crops would be the parent compound, while the irrigated soil could potentially contain a metabolite (II) as well as fluridone. Consequently, the present study was designed to determine residues of fluridone in crops and fluridone plus II in the soil.

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Several methods for determining fluridone residues have been published previously (West, 1978, 1981; West and Burger, 1980; West and Parka, 1981; West and Day, 1981, 1986, 1988; West and Turner, 1988), and this topic has been reviewed (West, 1984). For the irrigated crop study, methods published previously were used for crops (West and Day, 1988) and soils (West, 1984).

Our present paper describes the results of an irrigation study conducted to determine residue levels in crops and soils resulting from irrigation with water containing fluridone under field conditions. The study was designed to maximize the residues that might be expected to occur.

EXPERIMENTAL PROCEDURES

Irrigation of Crops. The irrigated crop residue studies were conducted in the United States at locations in California, Florida, Indiana, Alabama, and Mississippi. A summary of the field trial information is presented in Table I.

Two representative crops from each of the 13 agricultural commodity groups listed in Table II were irrigated with water containing the aquatic herbicide fluridone. Field plots for row crops consisted of single rows approximately 3 m in length, and plots for alfalfa, orchardgrass, wheat, and strawberries were approximately 1 m wide and 3 m long. Single trees were irrigated for tree crops.

Irrigation water was prepared in large containers by the addition of fluridone as an aqueous suspension (Sonar AS) to contain a nominal concentration of 0.123 ppm of fluridone. This concentration was equivalent to that obtained from a typically recommended application rate of Sonar (e.g., 0.45 kg of active ingredient/ha to a body of water with an average water depth of 0.9 m). This concentration slightly exceeded the 0.114 ppm maximum concentration of fluridone observed in water from 36 field dissipation trials (West et al., 1983).

The crops were sprinkler-, furrow-, or flood-irrigated with a total of 7.6–10.2 ha cm of water, depending on the crop or location (Table I). The irrigations were conducted during periods when water would normally be applied if it were needed. These irrigation volumes resulted in the cumulative soil application of fluridone at a theoretical rate of 0.09–0.12 kg/ha. The timing and frequency of irrigation, as well as the volume of water applied (Table I), were dependent upon cultural practices for the individual crops. For grapes, both sprinkler and flood irrigation techniques were used, since both techniques are commonly employed.

Residue Sample Collection. The dates of irrigation and harvest are listed in Table I. The crops were harvested for residue analysis at the earliest practical time following the last irrigation

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Table I. Description of Fluridone-Irrigated Crop Field Trials

crop	location	type of planting	planting date	irrigation method	irrigation date	irrigation ha cm	harvest date
orange	Florida	established	NA[a]	sprinkler	11/18/81	5.1	1/05/82
					11/24/81	5.1	
					total	10.2	
grapefruit	Florida	established	NA[a]	sprinkler	11/18/81	5.1	1/05/82
					11/24/81	5.1	
					total	10.2	
cucumber	Indiana	direct seeded	7/13/81	sprinkler	8/03/81	1.3	9/03/81
					8/05/81	1.3	
					8/10/81	1.3	
					8/15/81	1.3	
					8/18/81	1.3	
					8/20/81	1.3	
					8/26/81	1.3	
					total	8.9	
zucchini squash	Indiana	direct seeded	7/13/81	sprinkler	SAC[b]	SAC[b]	9/03/81
orchardgrass	Indiana	established	NA[a]	sprinkler	7/03/81	2.5	7/31/81
					7/10/81	2.5	
					7/17/81	2.5	
					7/24/81	2.5	
					total	7.6	
corn (forage)	Indiana	direct seeded	7/13/81	sprinkler	SAC[b]	SAC[b]	9/29/81
alfalfa	Indiana	established	NA[a]	sprinkler	SAO[b]	SAO[c]	7/31/81
soybean (forage)	Indiana	direct seeded	7/13/81	sprinkler	SAC[b]	SAC[b]	9/08/81
tomato	Indiana	transplant	7/13/81	furrow	SAC[b]	SAC[b]	10/01/81
green pepper	Indiana	transplant	7/13/81	sprinkler	SAC[b]	SAC[b]	9/08/81
corn	Indiana	direct seeded	7/13/81	furrow	SAC[b]	SAC[b]	10/20/81
wheat	Mississippi	direct seeded	11/14/81	sprinkler	4/13/82	2.5	6/21/82
					4/22/82	2.5	
					4/30/82	2.5	
					5/11/82	2.5	
					total	10.0	
cabbage	Indiana	transplant	7/13/81	sprinkler	SAC[b]	SAC[b]	9/17/81
lettuce	California	direct seeded	8/04/82	furrow	10/18/82	7.6	10/25/82
almond	California	established	NA[a]	flood	8/25/81	10.2	9/09/81
walnut	California	established	1974	sprinkler	8/06/81	10.2	9/09/81
apple	California	established	NA[a]	sprinkler	8/12/82	10.2	9/07/82
pear	California	established	NA[a]	sprinkler	8/12/82	10.2	8/25/82
carrot	Indiana	direct seeded	7/13/81	furrow	SAC[b]	SAC[b]	10/01/81
potato	Indiana	established	NA[a]	sprinkler	SAO[c]	SAO[c]	7/31/81
snapbean	Florida	direct seeded	1/22/82	sprinkler	3/19/82	5.1	3/31/82
					3/24/82	5.1	
					total	10.2	
soybean	Indiana	direct seeded	7/13/81	sprinkler	SAC[b]	SAC[b]	10/21/81
strawberry	Florida	established	NA[a]	sprinkler	NA[a]	5.1	2/03/82
					1/29/82	5.1	2/09/82
					total	10.2	2/16/82
							2/24/82

[a] Information not available. [b] Same as for cucumber. [c] Same as for orchardgrass.

to determine the maximum residues that might occur. The interval between the last irrigation and harvest ranged from 5 days for strawberries to 56 days for soybean and corn grain (Table II). All crops were harvested only one time following the last irrigation except for strawberries, which were harvested four times during a 3-week period following the last irrigation. Walnut and apple trees receiving a sprinkler irrigation were wetted only on the lower one-third of the trees, and residue samples were collected separately from the wetted and nonwetted parts of the trees. The other tree crops were entirely wetted, so that separate sample collections from wetted and nonwetted branches were not necessary.

Soil samples were collected from trials located in Florida, California, Mississippi, and Indiana. Coarse, medium, and fine soil textures were represented (Table V). Soil samples were collected prior to treatment in two trials, immediately after treatment in one trial, at harvest in four trials, and 1 year after irrigation in one trial. Subsamples (20 cores) were collected at depths of up to 30 cm with a 1.9 or 2.5 cm i.d. soil sampler. The subsamples were composited prior to being transported to the analytical laboratory.

Preparation and Storage of Samples. Initial handling of the crop samples was patterned after commercial practices. Some crops were air-dried or gently washed with water before being transported to the analytical laboratory. Upon receipt, the samples were frozen at −15 °C or chilled at 4 °C until being ground up and mixed to form homogeneous samples. Samples were then frozen at −15 °C until analyzed. Storage stability samples, which were prepared by fortifying untreated control crop with 0.1 ppm of fluridone, were stored and analyzed along with the residue samples to determine the stability of fluridone during the period of storage prior to analysis.

The composited soil subsample cores were air-dried (if wet) and mechanically blended to form a homogeneous sample. (Both fluridone and II are stable and nonvolatile, so that losses do not occur upon drying.) The soil samples were stored at 4 °C until analyzed.

Residue Analysis. The determination of fluridone residues in crops was accomplished by means of methods published previously (West and Day, 1988). Fluridone was extracted from the crop samples with methanol. An aliquot of the extract was diluted with aqueous 5% NaCl, partitioned with hexane to remove interfering coextractives, and then partitioned with dichloromethane to extract fluridone. The extracts were further purified by alumina column chromatography, evaporated using a rotary vacuum evaporator, and dissolved in methanol/water (70:30 v/v) for analysis by HPLC with UV detection at 313 nm.

A few of the crops (corn and soybean forage plants, alfalfa, orchardgrass, and grapefruit) produced interfering peaks on the HPLC chromatogram. Consequently, these crops were analyzed

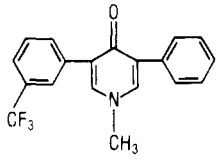
Table II. Residues in Crops Irrigated with Water Containing 0.123 ppm of Fluridone

commodity group	crop	irrigation method	no. of irrigations	ha cm applied	days to harvest[a]	part assayed	residue, ppm
citrus crops	orange	sprinkler	2	10.2	42	whole fruit	NDR[b]
	grapefruit	sprinkler	2	10.2	42	whole fruit	NDR
cucurbits	cucumber	sprinkler	7	8.9	8	whole fruit	NDR
	summer squash	sprinkler	7	8.9	8	whole fruit	NDR
forage grasses	orchardgrass	sprinkler	4	10.2	7	grass	0.11
	corn plant	sprinkler	7	8.9	34	whole plant	NDR[c]
forage legumes	alfalfa	sprinkler	4	10.2	7	whole plant	0.07
	soybean plant	sprinkler	7	8.9	13	whole plant	NDR
fruiting vegetables	tomato	furrow	7	8.9	36	whole fruit	NDR
	green pepper	sprinkler	7	8.9	13	whole fruit	NDR
grain crops	corn	furrow	7	8.9	56	grain	NDR
	wheat	sprinkler	4	10.2	41	grain	NDR
						straw	NDR
leafy vegetables	cabbage	sprinkler	7	8.9	22	head	NDR
	lettuce	furrow	3	7.6	7	head	NDR
nut crops	almond	flood	1	10.2	15	nut	NDR
						shells	NDR
	walnut	sprinkler	1	10.2	34	nut	NDR[d]
						shells	NDR[d]
pome fruits	apple	sprinkler	1	10.2	26	fruit	NDR[d]
	pear	sprinkler	1	10.2	13	fruit	NDR
root crops	carrot	furrow	7	8.9	36	root	NDR
	potato	sprinkler	4	10.2	7	potato	NDR
seed/pod vegetables	snapbean	sprinkler	2	10.2	7	seed/pod	NDR
	soybean	sprinkler	7	8.9	56	seed	NDR
small fruits	strawberry	sprinkler	2	10.2	5	fruit	NDR
					11	fruit	NDR
					18	fruit	NDR
					26	fruit	NDR
	grape	sprinkler	1	10.2	12	fruit	NDR
	grape	flood	1	10.2	12	fruit	NDR
stone fruits	plum	flood	1	10.2	12	fruit	NDR
	peach	flood	1	10.2	8	fruit	NDR

[a] Number of days from the last irrigation to harvesting of the crop. [b] No detectable residue at a detection limit of 0.05 ppm. [c] Contains an apparent residue slightly below the 0.05 ppm validated limit of detection. [d] None detected in samples from wetted and nonwetted portion of tree.

by gas chromatography with electron capture detection of a brominated derivative of fluridone (III) (West and Day, 1988; West, 1978).

Irrigated soil samples were analyzed for fluridone and the potential soil metabolite (II) according to a method described



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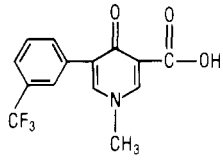
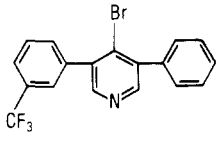
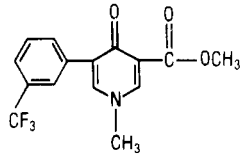


II



III



IV

previously (West, 1984). The metabolite was derivatized with diazomethane to form the corresponding methyl ester (IV) of the potential soil metabolite. Both compounds were then determined by HPLC with UV detection at 313 nm.

Recovery samples for each crop and soil type were assayed in duplicate with each set of residue samples. The recovery samples were prepared by fortifying untreated controls with 0.1 ppm of fluridone. Soil samples were also fortified with 0.1 ppm of II. All assay results for the control, treated, and storage stability samples were corrected for the average net recovery obtained for each sample type on each assay date.

Table III. Recoveries of Fluridone and Its Potential Soil Metabolite (II) from Fortified Soil Samples

location	soil texture	% recovery[a] fluridone	% recovery[a] II
Florida	coarse	72	117
		69	125
California	coarse	58	100
		71	123
		76	67
		83	75
Mississippi	medium	100	48
		98	84
Indiana	fine	58	115
		71	
$\bar{X}$		76	95

[a] All recovery samples were prepared by fortifying untreated control soil with 0.10 ppm of fluridone and II.

Apparatus, Chemicals, and Reagents. All solvents for HPLC and GC were of HPLC grade and pesticide grade, respectively. Anhydrous sodium sulfate was washed with methanol and dried at 50 °C for 16 h. Alumina (Alcoa F-20) was dried at 110 °C for 16 h, deactivated with 4.0% water (v/w), and tumbled for 1 h in a closed container.

The gas chromatograph was a Hewlett-Packard Model 402 equipped with a ^{63}Ni electron capture detector. The column was a 180 cm × 0.4 cm i.d. borosilicate glass tube containing 3% OV-101 on 80/100-mesh Chromosorb W-HP. The oven, detector, and injection block were operated at 195, 275, and 230 °C, respectively.

The HPLC system consisted of a Waters Model 6000A solvent delivery system operated at a flow rate of 1.0 mL/min, a Waters Model 440 absorbance detector (fixed wavelength, 313 nm) operated at 0.02 AUFS, a Waters Model 710A intelligent sample processor (100-μL injection), a Houston Instruments Omni Scribe

Table IV. Residues in Soil Irrigated with Water Containing Fluridone

crop and location	irrigation information: method	ha cm	total kg/ha fluridone	sampling information: days after irrigation	soil depth, cm	soil texture	residue, kg/ha: fluridone	II
orange (Florida)	sprinkler	4.0	0.12	42	0–15	coarse	NDR[a]	NDR[a]
grapefruit (Florida)	sprinkler	4.0	0.12	42	0–15	coarse	NDR	NDR
soybean (Indiana)	sprinkler	3.5	0.11	56	0–15	fine	0.04	NDR
wheat (Mississippi)	sprinkler	4.0	0.12	41	0–15	medium	NDR	NDR
					15–30		NDR	NDR
walnut (California)	sprinkler	4.0	0.12	0	0–30	coarse	0.13	NDR
				371	0–15		NDR	NDR
					15–30		NDR	NDR

[a] No detectable residue at detection limits of 0.02 ppm for fluridone and 0.05 ppm for II. These detection limits are equivalent to 0.02–0.06 kg/ha for fluridone and 0.06–0.13 kg/ha for II.

Table V. Characteristics of Irrigated Soil

site	texture	type	% sand	% silt	% clay	% OM	pH
Indiana	fine	clay loam	23.6	40.0	36.4	5.1	7.0
Mississippi	medium	silt loam	10.8	74.0	15.2	1.9	6.9
Florida	coarse	sand	93.2	1.6	5.2	1.7	7.1
California	coarse	N/A[a]	N/A	N/A	N/A	N/A	N/A

[a] Information not available.

strip chart recorder operated at a chart speed of 0.167 cm/min, and a μBondapak C_{18} column with a Co-Pell ODS guard column (Whatman, Inc.). The mobile phase was methanol/water (70:30) for fluridone and (65:35) for IV.

Calculations for both the GC and HPLC methods were based upon manual peak height measurements.

RESULTS AND DISCUSSION

Analytical Recovery Efficiencies and Limits of Detection. The duplicate recoveries obtained for each crop have been summarized previously (West and Day, 1988). Overall, recoveries averaged 87% for the HPLC procedure and 67% for the GC procedure. The lower recoveries for the GC procedure are likely due to the efficiency of the derivatization and the additional partitioning and cleanup steps associated with the derivatization procedure. The derivatization procedure has been discussed previously (West, 1978).

The duplicate recoveries for each soil sample are summarized in Table III. The recoveries averaged 76% and 95% for fluridone and II, respectively.

The validated limit of detection for the analytical method was 0.05 ppm for fluridone in crops, 0.02 ppm for fluridone in soil, and 0.05 ppm for II in soil. The detection limits for these crop and soil methods have been validated previously in our laboratory (West and Parka, 1981; West and Day, 1988), and the validation information and representative chromatograms are contained in the previous publications.

Storage Stability. The storage stability data were corrected for the level of recovery obtained from freshly fortified recovery samples prepared on the date of analysis. The storage recoveries averaged 95% of theory for all of the crops, thus indicating that a loss of fluridone had not occurred in the samples during storage (West and Day, 1988).

Previous studies in our laboratory have indicated that fluridone is stable in soil stored at 4 °C for at least 5 months (West, unpublished results), and the soil samples were analyzed in less than 5 months after collection.

Residues in Crops. The results of the residue assays for fluridone in crops are summarized in Table II. Representative chromatograms have been published previously (West and Day, 1988). Fluridone residues above the detection limit occurred in only two crops, orchardgrass (0.11 ppm) and alfalfa (0.07 ppm). Corn plants grown for forage appeared to contain a residue slightly below the 0.05 ppm validated limit of detection. None of these three crops would be consumed directly by humans, and the residues are below the 0.15 ppm tolerance for forage grasses and legumes established by the U.S. Environmental Protection Agency (*Federal Register*, 1986). The nondetectable residues for the remaining crops at a detection limit of 0.05 ppm are below the 0.1 ppm tolerance for these crops (*Federal Register*, 1986).

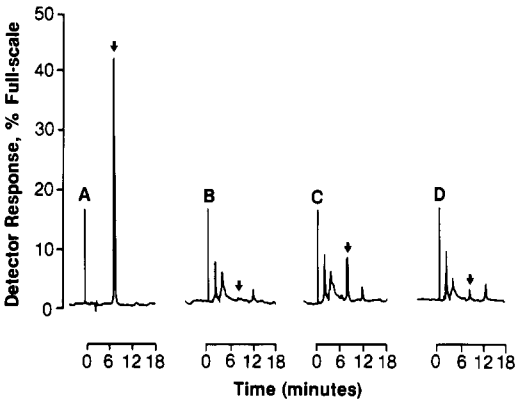


Figure 1. High-performance liquid chromatograms demonstrating the determination of fluridone in soil (letters indicate injection time and arrows indicate retention time of fluridone): (A) fluridone standard (100 ng); (B) untreated control soil containing no detectable fluridone residue; (C) untreated control soil fortified with 0.1 ppm of fluridone (equivalent to a 71% recovery); (D) irrigated soil containing 0.026 ppm (0.04 kg/ha) of fluridone.

Residues in Soil. The results of the residue assays for fluridone and II in the irrigated soils are summarized in Table IV, and representative chromatograms are contained in Figures 1 and 2. Detectable residues of fluridone were present in only two of eight irrigated soil samples at a detection limit of 0.02 ppm (0.02–0.06 kg/ha). In the trial at California, the soil sampled immediately after irrigation contained fluridone at 0.13 kg/ha, which was 108% of the theoretical application rate. However, no detectable residue was present 1 year later. In the Indiana trial, 0.04 kg/ha of fluridone was present in the soil at harvest (56 days postirrigation). None of the other soil samples contained detectable residues of fluridone. In addition, none of the soil samples contained detectable residues of II at a detection limit of 0.05 ppm. The 0.05 ppm detection

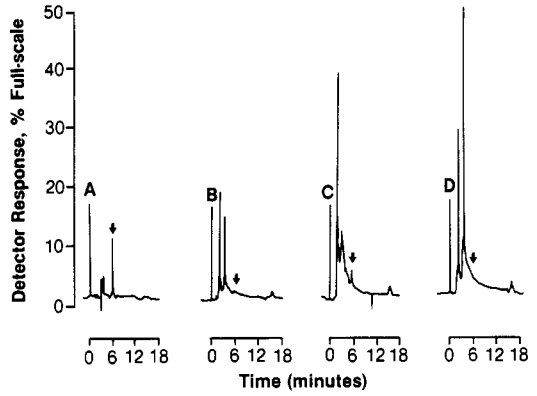


Figure 2. High-performance liquid chromatograms demonstrating the determination of the fluridone soil metabolite (II) as its methyl ester derivative (IV) in soil (letters indicate injection time and arrows indicate retention time of IV): (A) IV standard (100 ng); (B) untreated control soil containing no detectable residue of II; (C) untreated control soil fortified with 0.1 ppm of II (equivalent to a 100% recovery); (D) irrigated soil containing no detectable residue of II.

limit is equivalent to 0.06–0.13 kg/ha, depending upon the density of the soil (West et al., 1979). The characteristics of the soils are contained in Table V.

By design, the residues detected in this study were likely to be the maximum residues that would occur on irrigated crops and soil. The concentration of fluridone in the irrigation water (0.123 ppm) was higher than the maximum concentration of 0.114 ppm observed in 36 field dissipation trials conducted under actual use conditions (West et al., 1983). In addition, fluridone was prepared in the irrigation water at a concentration of 0.123 ppm immediately prior to each irrigation, whereas, under actual use conditions, the herbicide has dissipated with an average half-life of 20 days in ponds and of less than 10 days when portions of lakes were treated (West et al., 1983). Thus, the concentration of fluridone in irrigation water would typically be less than that used in this study.

CONCLUSIONS

Irrigation of crops with water containing fluridone did not result in detectable residues of the aquatic herbicide in any of the agricultural commodities that would be consumed by humans. Low residues were detected in two forage commodities, orchardgrass (0.11 ppm) and alfalfa (0.07 ppm). Corn plants grown for forage appeared to contain a residue slightly below the validated limit of detection of 0.05 ppm. Only two of eight irrigated soil samples contained detectable levels of fluridone, and none contained detectable residues of a hydrosoil-derived metabolite (II) observed in previous laboratory studies.

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