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Conservation cotton production in the southern United States: herbicide dissipation in soil and cover crops

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Soil and surface residues from cotton field studies in Stoneville, MS (1994 through 1996) and Florence, SC (1995 through 1996) were sampled to evaluate effects of cover crop and tillage on herbicide dissipation. Mississippi treatments included tillage (conventional [CT]; none [NT]) and cover crop (ryegrass; none [NC]). South Carolina treatments included tillage (CT; reduced tillage [RT]) and cover crop (rye; NC). Fluometuron was applied preemergence (PRE) in both Mississippi and South Carolina, and norflurazon was applied PRE in Mississippi. Soils were sampled various times during the growing season (depths: 0 to 2 cm, 2 to 10 cm). Cover crop residues were sampled from RT or NT cover crop areas. Soil and cover crop sample extracts were analyzed for herbicides. Soil organic carbon tended to increase with tillage reduction and presence of cover crop and was positively correlated with herbicide sorption, especially in the surface. Across locations, herbicide half-lives ranged from 7 to 15 d in the soil surface. Tillage had mixed effects on herbicide persistence in surface soil, with higher herbicide concentrations in CT at early samplings, but differences were insignificant later on. The most consistent effects were observed in RT/NT with cover crops, where cover crop residues intercepted applied herbicide, impeding subsequent movement into soil. Herbicide dissipation in cover crop residues was often more rapid than in soil, with half-lives from 3 to 11 d. Herbicide retention in cover crop residues and rapid dissipation were attributed to strong herbicide affinity to cover crop residues (e.g., fluometuron K_d = 7.1 [in rye]; K_d = 1.65 [in Mississippi Dundee soil CT, NC]) and herbicide co-metabolism as cover crop residues decomposed. A fluometuron metabolite, desmethyl-fluometuron, was observed in most soil and cover crop samples after 1 wk. Only minimal herbicide or metabolite moved into the subsurface, and little treatment effect could be ascribed to herbicide or metabolite movement below 2 cm.

Nomenclature: Desmethyl fluometuron; fluometuron; glyphosate; norflurazon; paraquat; pendimethalin; cotton, *Gossypium hirsutum* L. 'Stoneville 506', 'Delta & Pine Land DES 119'; Italian ryegrass, *Lolium multiforum* Lam.; rye, *Secale cereale* L.

Key words: Cover crop, crop residue, herbicide degradation, reduced tillage, soil depth.

Conservation crop production involves a variety of management practices such as cover crops, crop rotation, or reducing tillage (Locke and Bryson 1997). Cover crops and tillage reduction usually result in a net accumulation of plant residue on the soil surface, thereby providing many benefits, including enhancing soil quality, reducing runoff, and preserving soil water. Decomposing plant residue eventually increases soil organic carbon (C) concentrations in the soil surface and stimulates soil biota (Locke et al. 2002; Locke and Zablotowicz 2004; Reddy et al. 1997, 2003; Zablotowicz et al. 1998). Pore structure due to aggregation is improved in undisturbed soils (Rhoton 2000), whereas soil fauna, such as earthworms and insects, burrow through the soil, developing organic-rich tunnels. Because these soils are not often disturbed or mixed, the accumulation of organic C and associated biotic activity diminish dramatically with soil depth (Zablotowicz et al. 2000).

Soil conditions under conservation management may have implications for pesticide dissipation and transport (Locke and Bryson 1997). Increased quantity and diversity of microorganisms may enhance degradation of xenobiotic compounds including pesticides, thus reducing their potential for transport. Additionally, there is the potential for increased quantities of soil organic C available to sorb pesticide (Locke and Harper 1991; Locke 1992; Locke et al. 1997), thus impeding mobility. On the other hand, tunnels created by fauna activity and improved pore structure due to soil aggregation could provide the means for increased leaching of chemicals through the soil profile (Locke and Bryson 1997).

A growing number of farmers are using conservation practices in cotton production systems, but information on the environmental impact of these management practices is limited. Evaluating herbicide fate in soil is part of the risk assessment process needed to address environmental concerns related to Total Maximum Daily Loads (TMDLs). Also, increased soil organic C or plant residues may influence the herbicide rate needed for adequate weed control (Teasdale et al. 2003). The present studies were conducted in two locations to evaluate the effects of cover crop and tillage on the dissipation of fluometuron and norflurazon herbicides, both widely used in cotton production. Portions of the information in this article have been presented previously (Locke et al. 1995, 2002; Wagner et al. 1995).

Materials and Methods

Stoneville, MS, Evaluations

In the Mississippi study, a split-plot experiment with four replications was established in 1990 at the Southern Weed Science Research Unit farm near Stoneville, MS. Tillage (conventional tillage or no-tillage) was the main effect. Beginning in 1993, plots were split with a cover crop treatment of annual ryegrass or no cover crop. Conventional tillage (CT) consisted of disking and rowing into beds in the fall, then reforming row beds in the spring. Between-row cultivation was done during the season as needed. No-tillage (NT) involved planting directly into the row beds from the previous year's crop (NT row beds were established in 1990 and left undisturbed for the duration of the study). The winter cover was planted in the fall each year and killed with paraquat or glyphosate 2 to 4 wk before planting cotton the following spring. Cotton variety Stoneville 506[1] was the cultivar used in this study, and planting dates were April 25, 1994, May 11, 1995, and May 1, 1996. Preemergence (PRE) herbicide applications at time of cotton planting included fluometuron (1.1 kg ai ha^{-1}) and norflurazon (0.8 kg ai ha^{-1}). Both burndown and PRE herbicides were applied in 187 L ha^{-1} spray volume and TeeJet 8004[2] spray nozzles.

The soil series was Dundee silt loam (fine-silty, mixed, thermic Aeric Ochraqualf) (0- to 2-cm, 2- to 10-cm depths: % sand 28, 24; % silt 51, 52; % clay 21, 24). Before herbicide application in 1994, 1995, and 1996, soil was collected from the 0- to 2-cm and 2- to 10-cm depths of each plot for characterization. Surface plant residues were removed before sampling the soil. Soil was air-dried, sieved through a 2-mm screen, and analyzed for pH (1 : 1 wt/v, 0.01 M $CaCl_2$) and organic C (Walkley-Black method, Nelson and Sommers 1996).

Florence, SC, Evaluations

Two different sites were used for this experiment in Florence, SC. The site and experimental treatments for the South Carolina study in 1995 were established in the fall of 1990 with the first planting of the rye cover crop (Bauer and Busscher 1996). In 1996, this experiment was moved to a new site that had been in CT in previous years (i.e., 1996 samplings were made on soil that had not been plowed since 1995, so 1995 was when the treatment conditions were established). The experimental design for both years was a split-plot (main plot effect was cover, split plot was tillage; four blocks). All vegetation in both experiments was killed with glyphosate or paraquat in the spring 2 to 4 wk before planting cotton into stubble (in reduced tillage plots, RT) or disked soil (CT), followed by PRE herbicides at or just before planting cotton. In both studies, burndown and PRE herbicides were applied in 187 L ha^{-1} spray volume and TeeJet 8004 spray nozzles.

The soil series for the 1995 sampling was Norfolk loamy sand (fine-loamy, siliceous, thermic Aquic Kandiudult) (0 to 2 cm, 2- to 10-cm depths: % sand 77, 76; % silt 19, 18; % clay 4, 6). Cotton was grown the previous year. Rye was planted in November 1994 and killed with paraquat 5.5 mo after planting. Cotton variety DES 119[3] was planted on May 15, 1995. PRE herbicides pendimethalin (0.92 kg ai ha^{-1}) and fluometuron (2.2 kg ai ha^{-1}) were applied 1 d after planting, and glyphosate was applied to kill any weeds existing at planting.

In 1996, the soil used was Goldsboro loamy sand (fine-loamy, siliceous, thermic Aquic Kandiudult) (0 to 2 cm, 2- to 10-cm depths: % sand 76, 78; % silt 17, 13; % clay 7, 9). Corn was grown in the field the previous year. The study site was established in November 1995 when the rye was planted. The rye was killed with glyphosate 5 mo after planting. The area was subsoiled (in-row), pendimethalin (0.92 kg ha^{-1}) was applied preplant, and cotton (DES 119) was planted May 14, 1996. Fluometuron (2.2 kg ha^{-1}) was applied PRE at planting.

To characterize soil pH and organic C, soil was collected from each series and tillage and cover treatment combination and composited across replications. Surface plant residues were removed before sampling the soil.

Soil and Crop Residue Sampling for Herbicide Dissipation

Soil and surface residues from cotton field studies in Mississippi and South Carolina were sampled in 1994 through 1996 and 1995 through 1996, respectively, to evaluate effects of cover crop and tillage on herbicide dissipation. Soils and residues were sampled, beginning at herbicide application through approximately 30 d after application. Soil sampling depths were 0 to 2 cm and 2 to 10 cm, with a composite of four soil samples randomly collected from the middle rows of each plot using a tulip bulb planter (7.5 cm diam). Surface plant residues were removed before sampling the soil. Plant residue cover was sampled only on NT (Mississippi) or RT (South Carolina) treatments. At both locations, plant residue samples consisted of residues from cover crops killed that spring. Additionally, in South Carolina, aged plant residues, including cotton stubble from previous years, were separately sampled in 1995. Density of cover crop residues was determined by removing three 20 by 20 cm areas from each cover plot and drying the material at 60 C for 3 d to determine dry weight. Soil and plant samples used for herbicide extractions either were processed immediately or were frozen immediately and stored until processing. Water content was determined on subsamples of both the soil and plant samples using gravimetric methods, in which a moist sample was weighed and dried in an oven (100 C) for 24 h, and then the dried sample was reweighed.

Soil Herbicide Extraction and Analysis

Stored soil was thawed and weighed without drying into Nalgene HDPE[4] flasks for further processing. Soil was extracted with methanol (1 : 1 wt : v, soil : methanol) for 24 h, and samples were centrifuged at 10,000 $\times$ *g* for 15 min (Beckman[5] J2-21; JA-14 rotar). Aliquots were then filtered through Whatman 42[6] and Gelman Acrodisc[7] PVDF 25-μm filters and stored in 1-mL high-performance liquid chromatography (HPLC) vials until analysis. Samples were analyzed for fluometuron, desmethyl fluometuron (DMF), and norflurazon using HPLC. Preliminary investigations using spiked samples indicated a recovery > 95% for all three chemicals using these methods.

Plant samples were processed immediately by chopping them into lengths < 2 cm and weighing them into Nalgene flasks. Plant samples were extracted with methanol (1 : 10

wt : v, plant : methanol) for 24 h and processed as described previously for soil samples. Recovery was > 95% using these methods with spiked samples.

Extracts were analyzed with a Waters 2690 HPLC System.[8] HPLC analytical conditions included Waters Photo Diode Array UV Detector at 235 nm wavelength; Waters Scanning Fluorescence Detector 470 at Ex. 294 nm and Em. 398 nm wavelengths for norflurazon and Ex. 294 and Em. 329 nm wavelengths for fluometuron; Alltech C18 Econosil[9] column, 250 mm by 4.6 mm, 5 μm; gradient with initial 55% HPLC-grade water; 45% acetonitrile to 70% acetonitrile at 1 mL min^{-1} flow rate; and 50-μL injection volume. Retention times were 7.3 min for DMF, 9.5 min for fluometuron, and 11 min for norflurazon. Technical-grade fluometuron (99% purity) and norflurazon (99% purity) were obtained from Chem Service, Inc.,[10] and the fluometuron metabolite, DMF (99% purity), was obtained from Novartis, Inc. (now Syngenta Crop Protection[11]).

Herbicide Sorption

Fluometuron and norflurazon sorption in the Mississippi and South Carolina soils (0 to 2 cm, 2- to 10-cm depths, all combinations of tillage and cover crop) was evaluated using batch methods similar to those described in Locke et al. (1997). Dundee soil from the 1995 baseline sampling was used for sorption evaluations. Three initial concentrations of fluometuron or norflurazon ranging from 0.1 to 4 μg mL^{-1} were used, with five replications of each concentration. Norfolk (1995) and Goldsboro (1996) soils represented South Carolina in fluometuron sorption evaluations. Only one initial fluometuron concentration (2 μg mL^{-1}), with four replications, was used to determine the K_d for the South Carolina soils. One initial fluometuron or norflurazon concentration (1 μg mL^{-1}) was used for the cover crop sorption evaluations.

Soil was air-dried and ground to pass through a 2-mm sieve. For each herbicide, solutions were prepared in 0.01 M $CaCl_2$ using technical-grade and ^{14}C-labeled herbicide stocks. Air-dried soil was weighed into 25-mL Corex[12] centrifuge tubes, and herbicide solution was added at a ratio of 1 : 1 (wt : v) for the Dundee soil and 1 : 2 (wt : v) for the South Carolina soils. Killed rye and ryegrass samples were evaluated using 1 g of tissue (chopped to 2 cm lengths) in 12 mL of norflurazon or fluometuron sorption solution. The tubes containing treated soil or plant tissue suspensions were sealed with screw caps lined with polytetrafluoroethylene (PTFE),[13] shaken for 24 h at 25 C, centrifuged (12,000 × *g*, JA-20 Beckman rotar), and the supernatant was decanted. Radioactivity (i.e., herbicide concentration) in the supernatant was measured with a Packard Instruments[14] liquid scintillation counter using Ecolume[15] scintillation cocktail. Herbicide sorption was calculated by the difference between concentration added and concentration in solution after equilibration. Radiolabeled fluometuron (ring-UL-^{14}C label, 99% purity, specific activity 9.675 mCi $mmol^{-1}$) was obtained from Novartis Corp. (now Syngenta Crop Protection[16]). Radiolabeled norflurazon (ring-UL-^{14}C label, 99% purity, specific activity 41.1 mCi $mmol^{-1}$) was obtained from Sandoz, Inc. (now Syngenta Crop Protection[17]). Sorption assays were conducted in quadruplicate and then repeated.

Statistical Analyses

SAS (2001) was used to evaluate the statistical significance of treatments. Standard error was calculated for all treatment means. Analysis of variance (PROC ANOVA) general linear model procedures using Fisher's Protected LSD test to separate means was used to assess significance of soil properties for the Mississippi soil. ANOVA also was used to evaluate dissipation data (fluometuron, norflurazon, and DMF) for both Mississippi and South Carolina soils. Separate ANOVA were performed for each year because sampling times and intervals differed among years. For Mississippi soil dissipation data, tillage was the main effect, and tillage by block provided the error term for the *F* tests. The interaction of main plots nested within subplots provided the error term for cover crop (split treatment effect) *F* tests. Three sampling times were selected for ANOVA (first, second, and last sampling of each year). Effects of sampling time and treatment interactions were tested using the residual error term. For South Carolina soil dissipation data, the same ANOVA was used, except that cover crop was the main effect and tillage was the split effect.

Norflurazon and fluometuron sorption coefficients were calculated using the following equation:

$$K_d = (x/m)/C \quad [1]$$

where x/m = μmol kg^{-1}, and C = μmol L^{-1}. Nonlinear regression SAS techniques (PROC NLIN) were used with Equation 1 to estimate model parameter coefficients for sorption in the Mississippi Dundee soil where three initial herbicide concentrations were used.

Norflurazon and fluometuron dissipation in soil and cover crop residues was evaluated with nonlinear regression techniques using SAS (PROC NLIN) and the equations:

$$C = C_0^{-kt} \text{ and } t_{0.5} = 0.693/k \quad [2]$$

where C = herbicide concentration, C_0 = initial herbicide concentration, $t = d$ after herbicide application, and k is a constant.

Results and Discussion

Soil Characteristics

Soil pH and organic C for all soils are shown in Table 1. Because the Dundee soil in the Mississippi study had undergone tillage treatments since 1990 (cover crop since 1993), effects on organic C were evident. The order of organic C levels in surface soils was consistently NT ryegrass cover > NT no cover > CT ryegrass cover = CT no cover (1994 and 1995 $P < 0.05$; 1996 $P < 0.15$) (Table 1). The lack of tillage in NT soils resulted in an accumulation of residue at the soil surface, particularly in the cover crop areas, which increased soil organic C. Tillage obscured most of the cover crop effect in the surface soil (0 to 2 cm), but mixing the soil slightly enhanced organic C in the 2- to 10-cm depth (Table 1). Soil pH in the Mississippi soils was slightly acidic (Table 1). Generally, pH in the surface and 2- to 10-cm depths of NT cover crop soils was lower than that in other treatments, which is similar to reports elsewhere (Locke and Bryson 1997), but no other clear trends for soil pH emerged (Table 1).

Overall, organic C was lower in the South Carolina soils

TABLE 1. Soil organic C and pH in Dundee silt loam soil, Mississippi (1994, 1995, and 1996) and Norfolk and Goldsboro soils, South Carolina. Values for the Dundee soil are means of samples from four blocks, and analysis of variance (ANOVA) was used to assess treatment differences. Values for the South Carolina soils are means of three (organic C) and two (pH) replications from samples composited across replications from each treatment, so ANOVA could not be used to separate treatment means. Standard error is in parentheses below each mean for South Carolina soils.

			Organic C		pH	
Soil	Tillage	Cover	0 to 2 cm	2 to 10 cm	0 to 2 cm	2 to 10 cm
Dundee 1994	No-tillage	Ryegrass	19.8[a,b]	6.90[a]	5.96b[a,b]	5.73
						(0.31)
		None	13.9b	6.10b	6.65a	5.87
						(0.16)
	Conventional tillage	Ryegrass	9.50c	7.30a	6.39a	5.70
						(0.12)
		None	8.00c	6.00a,b	6.49a	5.76
						(0.11)
Dundee 1995	No-tillage	Ryegrass	21.3a	5.90	5.15	5.33
					(0.12)	(0.13)
		None	14.9b	5.70b	6.03	5.78
					(0.08)	(0.15)
	Conventional tillage	Ryegrass	10.1c	6.80a	5.73	5.45
					(0.13)	(0.21)
		None	9.40c	6.00b	6.25	6.06
					(0.02)	(0.09)
Dundee 1996	No-tillage	Ryegrass	24.2	6.72	5.20b	4.98
			(5.93)	(0.41)		(0.21)
		None	12.4	6.02	6.05a	5.24
			(1.27)	(0.36)		(0.16)
	Conventional tillage	Ryegrass	9.62	7.00	5.93a	4.91
			(0.62)	(0.70)		(0.18)
		None	9.10	7.19	6.05a	5.26
			(1.49)	(0.67)		(0.10)
Norfolk 1995	Reduced tillage	Rye	12.6[c]	7.10	6.60[c]	6.58
			(0.67)	(0.57)	(0.02)	(0.02)
		None	12.2	6.67	6.53	6.69
			(0.91)	(0.08)	(0.01)	(0.01)
	Conventional tillage	Rye	6.78	7.94	6.07	6.57
			(0.24)	(0.25)	(0.05)	(0.01)
		None	6.38	8.44	6.14	6.43
			(0.23)	(2.69)	(0.01)	(0.01)
Goldsboro 1996	Reduced tillage	Rye	8.79	8.49	5.50	5.90
			(0.33)	(0.18)	(0.03)	(0.01)
		None	10.3	9.48	5.45	5.53
			(0.69)	(0.59)	(0.05)	(0.03)
	Conventional tillage	Rye	8.72	8.63	5.66	5.51
			(0.36)	(0.33)	(0.03)	(0.01)
		None	9.03	8.56	5.55	5.44
			(0.35)	(0.34)	(0.02)	(0.01)

[a] For Dundee soil in a given year, means within a column followed by the same letter do not differ at a level of $P < 0.05$ (Tillage by Cover interaction).
[b] For Dundee soil, where standard error is shown in parentheses below the mean, Tillage by Cover interaction was not significant at $P < 0.05$.
[c] For Norfolk and Goldsboro, the number in parentheses below the mean is standard error.

than in the Mississippi soil regardless of management. Tillage effects on soil organic C were observed in the Norfolk soil, but not the Goldsboro soil (Table 1). Similar to the Mississippi soil, reducing tillage resulted in increased organic C in the surface of RT Norfolk soils (both rye cover and no cover). Having a cover crop alone, however, did not appear to increase organic C in the surface. Mixing due to tillage did increase organic C in the 2- to 10-cm depth for all treatments in the Norfolk soil. No obvious effect due to tillage was observed in the Goldsboro soil (Table 1), probably because plots used in 1995 (Norfolk soil) at Florence had been in a conventional vs. conservation tillage comparison since 1991, whereas the study area in 1996 (Goldsboro soil) was only in the first year of reduced tillage (not tilled since spring 1995). Soil pH in both South Carolina soils was slightly acidic, and pH tended to be higher in the Norfolk. No effect due to tillage on soil pH was evident for either soil.

Herbicide Sorption

Fluometuron sorption (K_d) for the Dundee soil was greater in the 0 to 2 cm depth than in the 2- to 10-cm depth for all cover and tillage treatments, with the exception of the CT bare soil where sorption in the surface and lower depth was the same (Table 2). No-tillage treatments in the surface soil had higher sorption than corresponding CT treatments, with the highest sorption occurring in the NT

TABLE 2. Fluometuron and norflurazon sorption to soil as affected by tillage, cover crop, and soil depth in Dundee soil, Mississippi; and Norfolk and Goldsboro soils, South Carolina. For the Dundee soil, linearized K_d values were calculated using nonlinear regression with five replications. For the South Carolina soils, K_d was based on only one concentration, and values given are the mean of four replications.

Cover[a]	Tillage[b]	Depth (cm)	Fluometuron K_d			Norflurazon K_d
			Dundee	Norfolk	Goldsboro	Dundee
Yes	NT or RT	0 to 2	5.04 (0.14)[c]	0.70 (0.04)	0.42 (0.01)	3.61 (0.03)[d]
Yes	CT		2.07 (0.02)	0.43 (0.01)	0.43 (0.01)	2.28 (0.03)
No	NT or RT		2.39 (0.04)	0.58 (0.04)	0.47 (0.01)	2.18 (0.03)
No	CT		1.65 (0.03)	0.37 (0.01)	0.41 (0.01)	1.61 (0.03)
Yes	NT or RT	2 to 10	1.44 (0.03)	0.46 (0.01)	0.50 (0.02)	1.57 (0.03)
Yes	CT		2.01 (0.03)	0.51 (0.02)	0.44 (0.02)	2.13 (0.04)
No	NT or RT		1.61 (0.04)	0.42 (0.01)	0.48 (0.02)	1.58 (0.02)
No	CT		1.61 (0.02)	0.41 (0.03)	0.43 (0.03)	1.81 (0.03)

[a] Cover is ryegrass for Dundee soil and rye for Norfolk and Goldsboro soils.
[b] Tillage: NT, no-tillage for Dundee soil; RT, reduced tillage for Norfolk and Goldsboro soils; CT, conventional tillage for all soils.
[c] The number following the linearized K_d value is the asymptotic standard error for the regression.
[d] The number in parentheses following the mean K_d is standard error.

cover crop treatment. Although the magnitude of difference between cover crop treatments was less than in NT treatments, the CT cover crop surface soil had a higher K_d value than soil in the CT no-cover treatment. In the 2 to 10 cm soil depth, sorption was greatest in the CT cover crop soil and lowest in the NT cover crop soil. This reflects increased mixing of organic C from the surface when the CT soils were tilled vs. less organic C in the undisturbed 2- to 10-cm depth of the NT soil. These results support a strong relationship between fluometuron retention and organic C content, especially in the top 2 cm of soil (correlation of K_d vs. organic C in 0 to 2 cm, $r = 0.96^{p < 0.05}$).

Response of fluometuron sorption to tillage was less clear-cut and differed between the two South Carolina soils (Table 2). In the Norfolk soil, fluometuron sorption was greatest in the surface of RT soils, especially where a rye cover crop was used. Fluometuron sorption was higher in the surface of both RT soils, but was comparable if not slightly lower than the 2- to 10-cm depth in the CT soils. The lower fluometuron sorption in surface CT soils again reflected mixing due to tillage. These sorption results for Norfolk soil exactly follow the organic C trends for the soil treatments and depths, with higher sorption occurring where there was higher soil organic C (correlation of K_d vs. organic C, across soil depths $r = 0.90^{p < 0.01}$) (Table 1).

In the Goldsboro soil, there was little difference in fluometuron sorption that could be attributed to tillage (Table 2), likely because of its recent establishment as RT. The highest K_d values occurred in RT treatments, but these differences were only marginal. There was no significant correlation between organic C and fluometuron sorption. Again, these sorption results reflect the relatively low organic C levels for all treatments and soil depths (Table 1).

Norflurazon sorption to the Dundee soil followed patterns similar to fluometuron (Table 2). Norflurazon sorption was highest in the NT cover crop surface soil, and sorption in both cover and bare NT surface soils was higher than in the 2- to 10-cm depth. Sorption in the CT surface and 2- to 10-cm depth was similar in the cover crop treatment but was higher in the 2- to 10-cm depth for the bare treatment. These observations are attributed to organic C levels (correlation of K_d vs. organic C across soil depths, $r = 0.89^{p < 0.01}$) and soil mixing due to tillage.

Herbicide sorption to cover crop residues (rye: fluometuron K_d = 7.1 [SE 0.3], norflurazon K_d = 15.3 [SE 1.1]; ryegrass: fluometuron K_d = 7.8 [SE 0.3], norflurazon K_d = 19.5 [SE 0.4]) was considerably greater than sorption to soil, indicating substantially greater capacity for immobilizing herbicide intercepted during application. Affinity of norflurazon for these grass cover crop materials was greater than that of fluometuron, likely a reflection of its higher octanol water coefficient (norflurazon 280 at 25 C; fluometuron 242 at 25 C) and lower water solubility (norflurazon 28 mg L^{-1} at 25 C; fluometuron 110 mg L^{-1} at 25 C) (Vencill 2002). Norflurazon sorption to ryegrass was greater than its sorption to rye, but no major difference in fluometuron sorption was observed between the two cover crop materials.

Herbicide Dissipation in Cover Crop Residues

In RT cover crop treatments, a majority of the soil surface was covered by cover crop residue at the time of herbicide application (7,000 and 3,000 kg ha^{-1} in 1995 for Mississippi and South Carolina, respectively). Warm temperatures and adequate moisture contributed to rapid decomposition of residues during the sampling period. For example, 50 and 46% decomposition of cover crop residues for Mississippi and South Carolina, respectively, was measured in 1995.

Fluometuron and norflurazon dissipation in ryegrass residues was relatively rapid in Mississippi (Figure 1). Norflurazon dissipation half-lives ranged from 7 d in 1996 to 11 d in 1994, whereas fluometuron dissipation half-lives ranged from 3 d in 1994 to 9 d in 1995 (Table 3). In South Carolina, in 1995, aged plant residues (e.g., cotton stubble) from the previous year were separated and analyzed for fluometuron (Figure 2). Similar to Mississippi, short half-lives for fluometuron were observed in South Carolina rye residues as well as in the aged residues (Table 3). Rapid dissipation might be expected because of multiple factors. More exposure to the sun may subject the herbicides to photodegradation if rainfall does not occur soon after application (Vencill 2002). One factor that possibly contributed to rapid herbicide degradation in cover crop residues was moisture content. Zablotowicz et al. (1998) measured equivalent fluometuron degradation rates in ryegrass residues and soil but only under higher moisture conditions for ryegrass. Some of

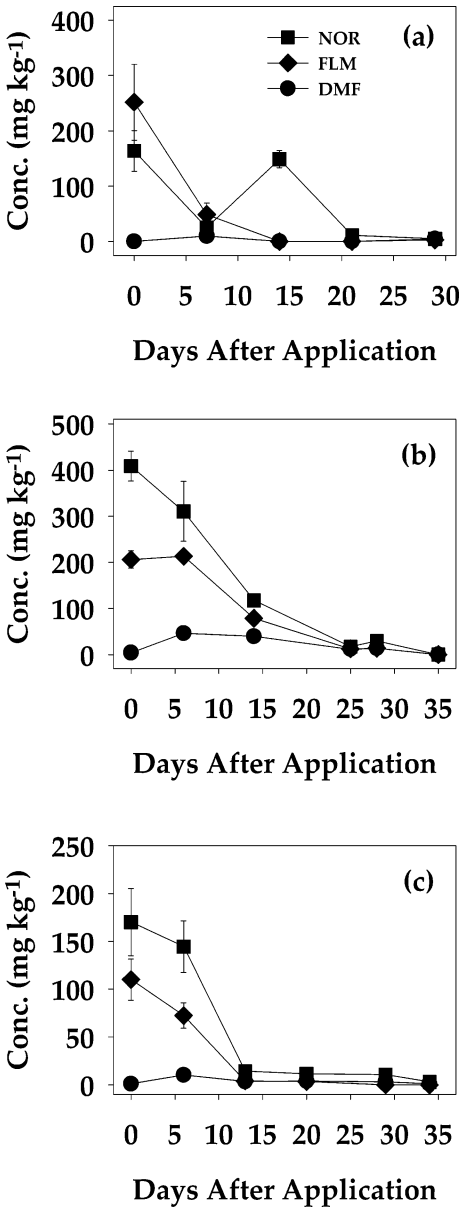

FIGURE 1. Dissipation of fluometuron (FLM) and norflurazon (NOR) and occurrence of desmethyl fluometuron (DMF) in ryegrass cover crop residues in Mississippi for (a) 1994, (b) 1995, and (c) 1996. Values shown are means of four replications, and error bars represent standard error.

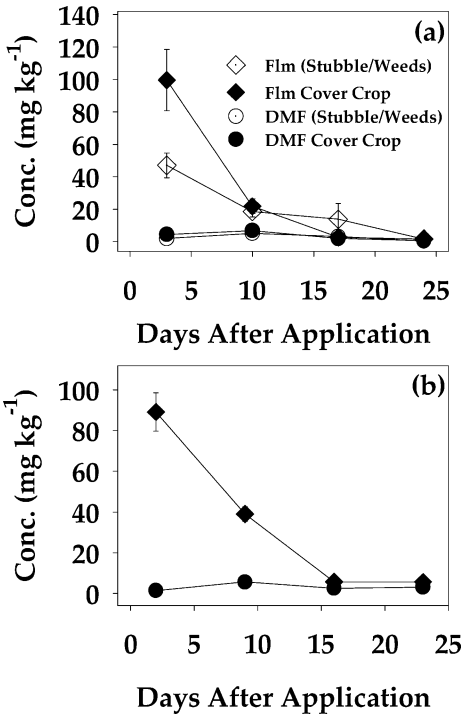

FIGURE 2. Dissipation of fluometuron (FLM) and occurrence of desmethyl fluometuron (DMF) in rye cover crop or aged weed and crop stubble residues in South Carolina for (a) 1995 and (b) 1996. Values shown are means of four replications, and error bars represent standard error.

the dissipation likely occurred because of elution of the herbicide from the cover crop material by rainfall. Rainfall soon after herbicide application can result in significant foliar washoff (Reddy et al. 1994; Reddy and Locke 1996).

DMF was present in ryegrass and rye cover crop residues throughout the periods of evaluation, but concentrations were very low (Figures 1 and 2). Ratios of the concentration of N-dealkylated metabolite to that of parent herbicide were used by Thurman et al. (1998) to characterize the environmental fate of certain herbicides such as atrazine. In the present study, ratios of DMF to FLM were calculated to help assess the dynamics of DMF appearance and fate over the course of the season. Higher ratios indicate increases in DMF concentration or reductions in fluometuron concentration. This ratio increased consistently over the course of the sampling periods for every year and at both locations,

TABLE 3. Dissipation of fluometuron and norflurazon in ryegrass residues in Mississippi and dissipation of fluometuron in rye residues and plant residues from the previous year in South Carolina. Nonlinear regression techniques were used to calculate *k* values and half-life (*d*).

Mississippi	Fluometuron calculated *k* ryegrass residues	Fluometuron half-life (*d*) ryegrass residues	Norflurazon calculated *k* ryegrass residues	Norflurazon half-life (*d*) ryegrass residues
1994	0.24 (0.09)[a]	3	0.07 (0.03)[a]	11
1995	0.074 (0.01)	9	0.09 (0.01)	8
1996	0.129 (0.02)	5	0.10 (0.02)	7

South Carolina	Fluometuron calculated *k* rye residues	Fluometuron half-life (*d*)	Fluometuron calculated *k* plant residues from previous yr	Fluometuron half-life (*d*) plant residues from previous yr
1995	0.22 (0.06)	3	0.11 (0.03)	6
1996	0.14 (0.02)	5	—	—

[a] Number in parentheses following the *k* value is the asymptotic standard error for the regression.

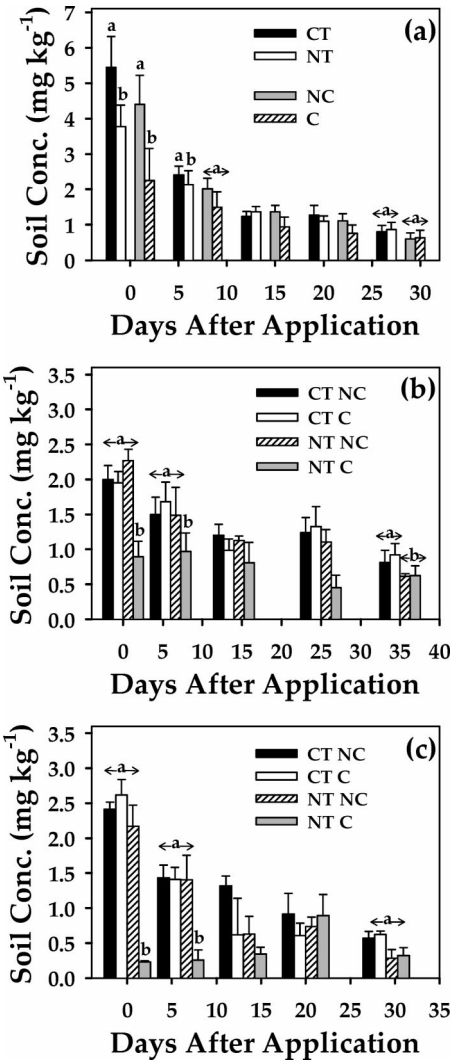

FIGURE 3. Fluometuron dissipation in Dundee surface (0 to 2 cm) soil in (a) 1994, (b) 1995, and (c) 1996, Stoneville, MS, showing effects of tillage (NT, no-tillage; CT, conventional tillage) and cover crop (NC, no cover crop; C, cover crop). Values shown are means of four replications, and error bars represent standard error. For the first, second, and last sampling times, the same letter above a bar within each sampling time indicates no significant treatment difference. The Tillage by Sampling Time and Cover by Sampling Time interactions for 1994 were significant, $P < 0.05$. The Tillage by Cover by Sampling Time interactions were significant for 1995 ($P < 0.06$) and 1996 ($P < 0.05$) but not significant at any level in 1994.

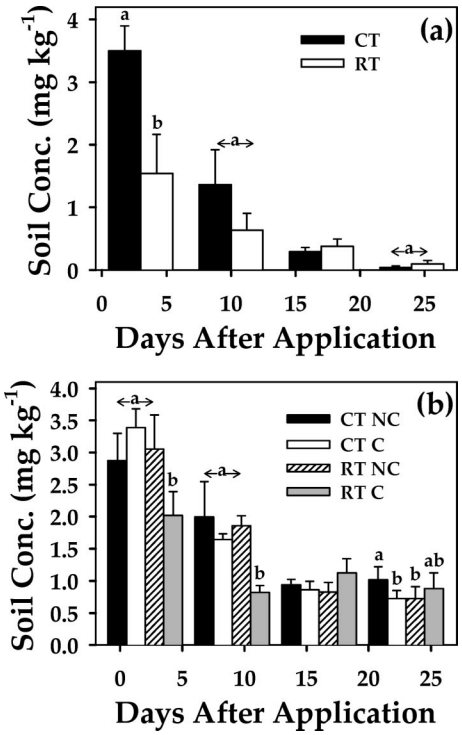

FIGURE 4. Fluometuron dissipation in surface soil (0 to 2 cm) soil, Florence, SC, in (a) 1995, Norfolk soil, and (b) 1996, Goldsboro soil, showing effects of tillage (RT, reduced tillage; CT, conventional tillage) and cover crop (NC, no cover crop; C, cover crop). Values shown are means of four replications, and error bars represent standard error. For the first, second, and last sampling times, the same letter above a bar within each sampling time indicates no significant treatment difference. The Tillage by Sampling Time interaction for 1995 was $P < 0.05$, and the Tillage by Cover by Sampling Time interaction for 1996 was $P < 0.07$.

primarily reflecting decreasing fluometuron concentrations rather than DMF accumulation (Figures 1 and 2), eventually rendering DMF concentrations equivalent to or greater than fluometuron concentrations. For example, DMF to FLM ratios in Mississippi cover crops ranged from 5.4 : 206.2 (SE 0.011) initially to 13.7 : 13.6 (SE 0.22) at the end of the 1995 sampling period. Similarly, in South Carolina cover crops, DMF to FLM ratios changed from 1.3 : 89.2 (SE 0.002) to 3.0 : 5.6 (SE 0.10) over the course of the 1996 sampling period.

Herbicide Dissipation in Soil

Fluometuron concentrations in the 0- to 2-cm soil depth of conservation tillage treatments (RT or NT) were often lower than in conventional tillage in both Mississippi and South Carolina for the first one or two samplings after planting, especially in the cover crop treatment (Figures 3 and 4). Similar trends were observed for norflurazon (Figure 5). For both norflurazon in Mississippi and fluometuron in Mississippi and South Carolina, the treatment differences in herbicide concentration observed earlier in the season diminished by the end of the sampling period (Figures 3–5).

For conservation tillage with no cover crop and all CT soils (cover crop and no cover crop), herbicide dissipation in the surface 0 to 2 cm followed first-order kinetics in both Mississippi and South Carolina, and dissipation constants, k, were calculated. The k values are not shown because there were no statistically significant effects observed among the dissipation parameters. Half-lives among the various treatments, locations, and years ranged from 7 to 15 d after herbicide application, and dissipation patterns can be observed in Figures 3, 4, and 5. The dissipation times observed in the present studies are shorter than those observed for fluometuron dissipation in a laboratory study (Brown et al. 1994) but are similar to those reported for these soils in a related laboratory study (Zablotowicz et al. 2000). The relatively rapid dissipation reflected the warm temperatures and rainfall occurring soon after herbicide application that could have facilitated either degradation or movement in surface runoff or leachate.

The low concentrations of fluometuron and norflurazon and their pattern of dissipation in the conservation tillage cover crop soils did not fit well with first-order kinetics, and

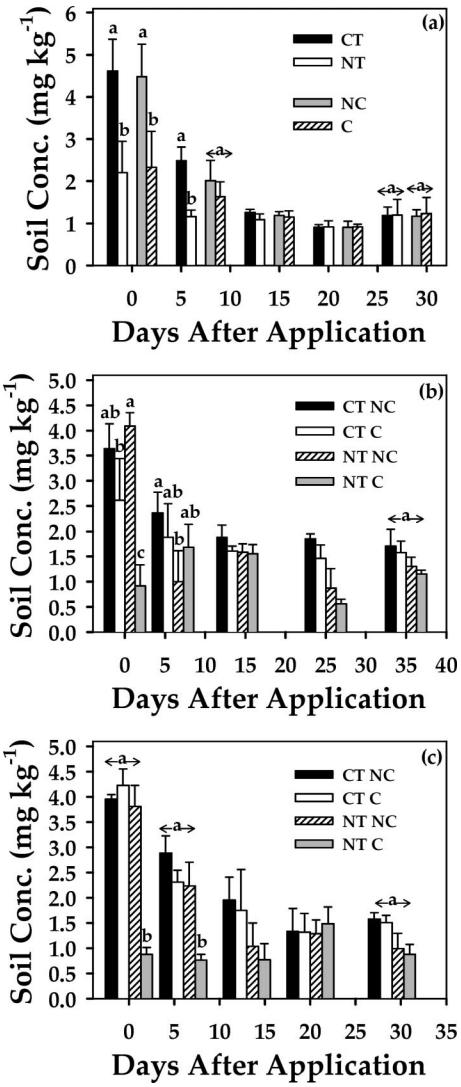

FIGURE 5. Norflurazon dissipation in Dundee surface (0 to 2 cm) soil in (a) 1994, (b) 1995, and (c) 1996, Stoneville, MS, showing effects of tillage (NT, no-tillage; CT, conventional tillage) and cover crop (NC, no cover crop; C, cover crop). Values shown are means of four replications, and error bars represent standard error. For the first, second, and last sampling times, the same letter above a bar within each sampling time indicates no significant treatment difference. The Tillage by Sampling Time and Cover by Sampling Time interactions for 1994 were significant, $P < 0.05$. The Tillage by Cover by Sampling Time interactions were significant for 1995 ($P < 0.05$) and 1996 ($P < 0.05$) but not significant at any level in 1994.

dissipation kinetics were not assessed for that treatment. The dissipation patterns can be observed in Figures 3, 4, and 5. Interception of herbicide by the cover crop, delayed washoff from crop residues, and herbicide degradation within the cover crop residues (Figures 1 and 2) were major factors attributed to the low concentrations in conservation tillage cover crop soils. As mentioned previously, the plant residues typically decomposed to < 50% of initial biomass over the course of the growing season in Mississippi and South Carolina. Either the herbicide degraded in situ in the cover crop residue (Figures 1 and 2) or washed into the soil. Lower levels of fluometuron and norflurazon in surface soil of the RT and NT cover crop areas also may indicate that the plant residues enhanced degradation of herbicide that eluted through the residues to soil (Bottomley et al. 1999), but there is no way to substantiate this route of dissipation.

The fluometuron metabolite, DMF, was observed in most samples in the 0- to 2-cm soil depth within 1 wk after application, indicating degradation as a significant mechanism of fluometuron dissipation in soil in these studies (Table 4). For most treatments in both Mississippi and South Carolina, DMF tended to be initially low, gradually increasing to a peak 2 to 3 wk after fluometuron application, then either remaining the same or declining by the last sampling (Table 4). There were some deviations from this pattern; for example, in South Carolina, in 1995, CT treatments had higher initial DMF concentrations that diminished during the sampling period (Table 4). Cover crop rarely had an effect on DMF, with the exception of South Carolina in 1996 where DMF in cover crop soil was lower than in soil with no cover at the second sampling (Table 4). In several instances, DMF tended to be initially the same regardless of tillage but by the second sampling more DMF was measured in CT (Table 4). By the end of the sampling period, tillage differences usually diminished (Table 4).

DMF : FLM ratios were calculated to determine whether any other dissipation patterns might be revealed. In most instances, patterns in the surface soil mirrored previous observations where the DMF : FLM ratio began low and increased to a peak over the first 2 to 3 wk, then declined or remained constant. This is indicative of low initial DMF concentrations that increased as fluometuron was degraded. A few additional patterns in the surface soil, however, were observed that could be attributed to management. The average of the two highest DMF : FLM ratios during the season (i.e., the peak) for each treatment combination are shown in Table 5. Most of the DMF : FLM peaks in the surface soil occurred during the last portion of the sampling period. The highest DMF : FLM ratios often occurred in cover crop surface soils (Table 5), supporting observations that cover crop may enhance fluometuron degradation. In the soil surface, conventional tillage with no cover never had the highest ratio, and conservation tillage was often higher than CT, especially in Mississippi, indicating that higher plant residues associated with conservation management provide an environment conducive for herbicide degradation.

Average fluometuron and norflurazon concentrations measured in the 2- to 10-cm depth were typically less than one-third of the highest concentration observed in the surface, with the highest concentrations occurring approximately 1 to 4 wk after application. No effects on fluometuron concentration due to the main effects of tillage or cover were observed for Mississippi at the $P < 0.05$ level of significance in 1994, 1995, or 1996. Similarly, few consistent trends in herbicide movement into the 2- to 10-cm depth during the sampling periods were observed that could be related to tillage or cover crop in Mississippi, although some effects over sampling time were observed for fluometuron in 1995 and norflurazon in 1996 (Table 6).

Fluometuron concentrations in the 2- to 10-cm depth for South Carolina were less than 1.2 mg kg^{-1} in 1995 (data not shown) and less than 0.8 mg kg^{-1} in 1996. Although statistical comparisons between South Carolina and Mississippi were not made, apparent higher herbicide concentrations in the 2- to 10-cm depth (e.g., Table 6) for South Carolina may be reflective of the coarser textured soils. Fluometuron in the subsurface soil was influenced by both till-

TABLE 4. Effects of tillage or cover crop and sampling time on DMF concentration in the soil surface at the two locations (Mississippi and South Carolina) and the first, second, and third samplings. Tillage by Sampling Time interactions were significant for Mississippi in 1994 and 1996 and for South Carolina in 1996; Cover by Sampling Time interactions were significant for South Carolina in 1996; and only Sampling Time was significant for Mississippi in 1996. No other interactions were significant.[a]

Location and year	DAA	DMF		Location and year	DAA	DMF		Location and year	DAA	DMF	
		CT	NT			CT	NT			CT	RT
		mg kg^{-1}				mg kg^{-1}				mg kg^{-1}	
Mississippi 1994	0	0c[b],a[c]	0b,a	Mississippi 1996	0	0.19b,a	0.23a,a	S. Carolina 1995	3	0.36a,a	0.14a,b
	7	0.33a,a	0.13a,b		6	0.32a,a	0.22a,b		10	0.41a,a	0.18a,b
	28	0.24b,a	0.21a,a		34	0.22b,a	0.22a,a		24	0.09b,a	0.15a,a
		$n = 8$, $P < 0.05$				$n = 8$, $P < 0.07$				$n = 8$, $P < 0.05$	
						DMF					
Location and year	DAA	DMF		Location and year	DAA	NC	C				
		mg kg^{-1}				mg kg^{-1}					
Mississippi 1995	0	0.11b[b]		South Carolina 1996	2	0.12c,a	0.19c,a				
	6	0.25a			9	0.39b,a	0.30b,b				
	35	0.29a			23	0.53a,a	0.46a,a				
		$n = 24$, $P < 0.05$				$n = 8$, $P < 0.05$					

[a] Abbreviations: DAA, days after application; DMF, desmethyl fluometuron; CT, conventional tillage; NT, no-tillage; RT, reduced tillage; NC, no cover crop; C, cover crop.
[b] For a given location and year, means within a column followed by same letter are not significantly different ($P < 0.05$, except Mississippi 1996 where $P < 0.07$).
[c] For a given location and year, means within a row followed by the same letter are not significantly different ($P < 0.05$, except Mississippi 1996 where $P < 0.07$).

TABLE 5. Mean of the two highest incidences of the ratio of DMF to FLM in soil, and DAA when the DMF : FLM ratio was the highest.[a]

	DFM : FLM in 0 to 2 cm depth	DAA	DFM : FLM in 2 to 10 cm depth	DAA
Mississippi				
1994				
CT NC	0.30 (0.03)[b]	28	0.42 (0.12)[b]	14
CT C	0.44 (0.09)	21	0.36 (0.04)	14
NT NC	0.52 (0.08)	28	0.33 (0.08)	14
NT C	1.32 (0.41)	14	0.35 (0.05)	14
1995				
CT NC	0.34 (0.02)	28	0.87 (0.23)	25
CT C	0.34 (0.03)	25	1.41 (0.38)	25
NT NC	0.47 (0.03)	28	0.70 (0.15)	25
NT C	0.70 (0.12)	25	2.32 (0.91)	6
1996				
CT NC	0.71 (0.09)	29	1.92 (0.47)	13
CT C	2.85 (0.79)	13	22.4 (11.0)	29
NT NC	1.47 (0.29)	34	3.18 (1.16)	29
NT C	1.11 (0.19)	34	1.46 (0.16)	29
South Carolina				
1995				
CT NC	1.02 (0.13)[b]	17	0.78 (0.08)[b]	24
CT C	1.55 (0.36)	17	0.69 (0.06)	24
RT NC	0.84 (0.04)	24	0.52 (0.11)	24
RT C	1.55 (0.36)	24	0.69 (0.06)	24
1996				
CT NC	0.53 (0.02)	23	0.75 (0.06)	23
CT C	0.63 (0.04)	23	0.80 (0.06)	23
RT NC	0.72 (0.05)	23	0.61 (0.05)	23
RT C	0.55 (0.04)	23	0.83 (0.04)	23

[a] Abbreviations: DAA, days after application; FLM, fluometuron; DMF, desmethyl fluometuron; CT, conventional tillage; NT, no-tillage; RT, reduced tillage; NC, no cover crop; C, cover crop.
[b] Number in parentheses after the mean is standard error.

age and cover crop during the 1995 sampling period in South Carolina (Table 6). For CT, fluometuron was initially higher (3-d sampling), possibly reflecting 0.3 cm rainfall that occurred 2 d after herbicide application. This initial, elevated fluometuron concentration was not observed in RT, but fluometuron increased in subsequent samplings for RT. Fluometuron initially did not differ between cover crop and NC in 1995, but did dissipate faster in the cover crop soil (Table 6). In 1996, fluometuron concentrations in CT were higher than RT (CT 0.55 vs. NT 0.33 mg kg^{-1}, $P < 0.05$), but neither cover crop nor any interactive effects were significant. As occurred in 1995, fluometuron concentrations in 1996 were elevated at the 2-d (initial) sampling and remained so until the end of the sampling period when concentrations declined (Table 6). Again, this initial, elevated concentration possibly reflects 0.1 cm rainfall received at this site 2 d after herbicide application.

DMF detected in the 2- to 10-cm depth in all 3 yr in the Mississippi soil typically had peak concentrations 2 to 4 wk after planting (data not shown), but differences over the first, second, and last sampling times were only significant in 1994 (Table 6). No consistent pattern due to tillage or cover crop treatment was evident for DMF concentra-

TABLE 6. Effects of tillage, cover crop or DAA and interactions on FLM, NOR and DMF concentrations in the 2- to 10-cm soil depth at the two locations (Mississippi and South Carolina) and three sampling times (first, second, and third). Sampling time effects were significant for DMF (Mississippi 1994), FLM (Mississippi 1995), NOR (Mississippi 1996) and FLM and DMF (South Carolina 1996). Tillage by Sampling Time interactions in 1995 were significant for FLM in South Carolina, and Cover Crop by Sampling Time interactions in 1995 were significant for FLM and DMF in South Carolina. No other interactions were significant at $P < 0.05$, although some were significant at higher probability values (data not shown).[a]

Mississippi 1994	DMF	Mississippi 1995	FLM	Mississippi 1996	NOR
DAA	mg kg^{-1}	DAA	mg kg^{-1}	DAA	mg kg^{-1}
0	0b[b]	0	0.14b	0	0.19a
7	0.001b	6	0.15b	6	0.28a
28	0.011a	35	0.34a	34	0.02b
	$n = 24$, $P < 0.05$		$n = 24$, $P < 0.05$		$n = 24$, $P < 0.05$

S. Carolina 1995	FLM		FLM		DMF		S. Carolina 1996	FLM	DMF
	CT	RT	NC	C	NC	C			
DAA	mg kg^{-1}		mg kg^{-1}		mg kg^{-1}		DAA	mg kg^{-1}	
3	0.95a[b],a[c]	0.16b,b	0.57a,a	0.53a,a	0.10b,b	0.08b,b	2	0.47a[a]	0.01c
10	0.28b,b	0.60a,a	0.74a,a	0.14b,b	0.22a,a	0.04b,b	9	0.57a	0.16b
24	0.04c,a	0.057b,a	0.07b,a	0.024b,a	0.11b,b	0.12b,b	23	0.27b	0.27a
	$n = 8$, $P < 0.05$		$n = 8$, $P < 0.05$		$n = 8$, $P < 0.05$			$n = 24$, $P < 0.05$	$n = 24$, $P < 0.05$

[a] Abbreviations: DAA, days after application; FLM, fluometuron; NOR, Norflurazon; DMF, desmethyl fluometuron; CT, conventional tillage; NT, no-tillage; RT, reduced tillage; NC, no cover crop; C, cover crop.

[b] For a given location and year, means within a column followed by same letter are not significantly different ($P < 0.05$).

[c] For a given analyte, location and year, means within a row followed by the same letter are not significantly different ($P < 0.05$).

tions in Mississippi. Only in South Carolina in 1995 was a significant treatment interaction observed, where DMF in NC soil was initially low, increased, and then declined (Table 6). There was no change in DMF concentration for the cover crop soil throughout the sampling period, perhaps reflecting retention by the cover crop material at the surface that resulted in less downward migration of either fluometuron or DMF. The opposite trend was observed in 1996, where DMF steadily increased during the sampling period (Table 6).

Using the DMF : FLM ratios helped to illustrate some weak patterns with regard to treatment in the 2- to 10-cm soil depth (Table 5). DMF : FLM ratios in cover crop treatments (either CT or conservation tillage) were highest 60% of the time. Also, it was observed that in CT soils (either cover or no cover), DMF : FLM ratios in the 2- to 10-cm depth were usually higher than in 0- to 2-cm depth. This was possibly influenced by enhanced aeration of the 2- to 10-cm depth by tillage that would have promoted degradation of fluometuron to DMF.

These field observations from two locations and multiple years provide an opportunity to assess conservation practices under diverse conditions. The studies demonstrate the difficulties in making precise determinations under field situations, but the role that conservation management has on the fate of herbicides could be delineated, particularly with regard to cover crop residues. These field observations complemented findings of related laboratory studies (Zablotowicz et al. 2000) showing that increased residues and organic C from cover crops and reduced tillage enhanced the microbial activity conducive to herbicide degradation and intercepted and retained herbicides by sorption.

Sources of Materials

[1] Cotton variety Stoneville 506, Stoneville Pedigreed Seed, 6625 Lenox Park Dr. Ste. 117, Memphis, TN 38115.

[2] TeeJet 8004 spray nozzles, Spraying Systems Co., North Ave. & Schmale Rd., Glendale Heights, IL 60189.

[3] Cotton variety DES 119, Delta and Pine Land Co., 100 Main St., Scott, MS 38772.

[4] Nalgene HDPE flasks, Nalge Nunc International, 75 Panorama Creek Dr., Rochester, NY 14625.

[5] J2-21, JA-14 rotar, Beckman Coulter, Inc., 4300 N. Harbor Blvd., Fullerton, CA 92834.

[6] #42 filters, Whatman, Inc., 9 Bridewell Pl., Clifton, NJ 07014.

[7] PVDF 25-μm filters, Pall Corp., 600 South Wagner Rd., Ann Arbor, MI, 48103-9019.

[8] 2690 HPLC System, Waters, Inc., 34 Maple St., Milford, MA 01757.

[9] C18 Econosil column, Alltech Associates, Inc., 2051 Waukegan Rd., Deerfield, IL 60015.

[10] Technical-grade fluometuron, Chem Service, Inc., 660 Tower Ln., West Chester, PA 19381.

[11] Fluometuron metabolite desmethyl fluometuron (DMF) was a gift from Novartis Corp., now Syngenta Crop Protection, 1800 Concord Pike, Wilmington, DE 19850.

[12] 25-mL Corex centrifuge tubes, Corning, Inc., One Riverfront Plaza, Corning, NY 14831-0001.

[13] PTFE-lined screw caps, DuPont, Inc., Chestnut Run Plaza 705 GS29, Wilmington, DE 19880-0705.

[14] Model TriCarb 4000 series liquid scintillation counter, Packard Instruments Co., 800 Research Pkwy., Meriden, CT 06450.

[15] Ecolume scintillation cocktail, ICN, 3300 Hyland Ave., Costa Mesa, CA 92626.

[16] Radiolabeled fluometuron was a gift of Novartis Corp., now Syngenta Crop Protection, 1800 Concord Pike, Wilmington, DE 19850.

[17] Radiolabeled norflurazon was a gift of Sandoz, Inc., now Syngenta Crop Protection, 1800 Concord Pike, Wilmington, DE 19850.

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