

Environmental Fate and Impacts of Sulfometuron on Watersheds in the Southern United States

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ABSTRACT

Dissipation of sulfometuron (SM), methyl 2-[[[(4,6-dimethyl-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl] benzoate, in streamflow, sediment, plant tissue, litter, and soil following operational forestry applications at the target rate of 0.42 kg a.i. ha⁻¹ was monitored. Streamflow samples were collected at a weir on the perimeter and 30, 60, and 150 m downstream from the perimeter of the application site. Sulfometuron was detected in streamflow at low levels up to 29 days after treatment (DAT) on the watershed treated with the 75% dispersible granule formulation (Oust; DuPont Chemical Company, Wilmington, DE) and less than 53 DAT on the watershed treated with the experimental formulation (1% pellet). Twenty-four-hour average SM concentration in water ranged from not detected to a maximum of 49.3 µg L⁻¹. Sulfometuron was not detected at quantifiable levels (1 µg L⁻¹) 150 m downstream. Stream sediment, vegetation, litter, and soil were sampled periodically up to 180 DAT. All samples were analyzed for SM by high performance liquid chromatography. Sulfometuron dissipated from these watersheds with half-lives that ranged from 4 d in plant tissues to 33 d in soil. Acidic soil solution on these treated watersheds contributed to their rapid dissipation. Environmental impacts are discussed for these watersheds in the context of available toxicological data.

INTENSIVE FOREST MANAGEMENT, particularly pine management, has been practiced in the southern USA with increasing intensity since the early 1900s. Herbicides have since been recognized as an effective means of controlling competing vegetation and maximizing production of useable fiber. Use of herbicides in forest management in the southern USA increased 53% from 1996 to 1998 (Dubois et al., 1999). Wear and Greis (2001) predict very large increases in wood demand (56% for softwood and 47% for hardwood) throughout the USA by 2040 and report that almost all of the required increases in production will have to come from the southern USA. At the same time the forest land base in the USA is expected to decrease by 2%. Therefore, forest management will become even more intensive, requiring a greater reliance on herbicides for competition control to increase crop yield.

Use of herbicides in forest management is already associated with increased public concerns over human health and safety, and forest and aquatic ecosystem impacts. Any additional uses will intensify those concerns, and the public demands proof of safety. However, it is not possible under tenets of science to "prove" anything. In the face of this conundrum, How can we be sure of the safety of technology when our science is unable to

prove the safety?, we must find alternative methods of making decisions about new scientific developments and the role they will play in society. Currently, the most widely accepted alternative is risk assessment. Risk assessment recognizes and incorporates uncertainty into the process of evaluation and allows for the management of uncertainty in decision making. Risk assessment utilizes a tiered approach in which each tier may generate additional researchable questions and point to potential problems. At the conclusion of each tier more research may be required and more information provided. Risk assessments require toxicological data and knowledge of the fate of herbicides in the environment.

The active ingredient of the herbicide Oust is sulfometuron (SM). Sulfometuron is a member of a new class of herbicides that inhibit acetolactate synthase (ALS, also referred to as AHAS, acetohydroxy acid synthase, two acceptable names for the same enzyme system). Sulfometuron is herbicidally active at very low concentrations, and is registered for use in forest sites at rates up to 420 g ha⁻¹. Solubility of SM in water increases with increasing pH, but is relatively low at pHs typically observed in pine forests of the southern USA (10 mg L⁻¹ at 25°C and pH 5.5; DuPont Chemical Company, 1983). Sulfometuron is stable to hydrolysis at pH 7.0 but unstable in acid aqueous solution with a hydrolytic half-life of approximately 19.8 d at pH 5 and 4.2 d at pH 2 (temperature 25°C; DuPont Chemical Company, 1983). Under acidic conditions, SM hydrolyzes to 1,2-benzisothiazol-3-one, 2,3-dihydro 1,1-dioxide (saccharin), methyl 2-(aminosulfonyl)benzoate, and CO₂ (Harvey et al., 1985; Anderson and Dulka, 1985; Brown, 1990). Degradation is enhanced in the presence of ultraviolet light and similar products are formed. Aqueous photolysis is rapid with an 80 to 90% decrease in SM at the end of two weeks, even at pH 8.6 (Harvey et al., 1985). Sulfometuron, with its low solubility, low vapor pressure (5.5 x 10⁻¹⁶ mm Hg at 25°C; Beyer et al., 1987), and low partition coefficient ($K_d = 0.29$, Dickens and Wehtje, 1986; $K_{ow} = 0.31$, Harvey et al., 1985), would be available for transport in storm runoff unless its movement from or degradation in the upper few centimeters of soil is rapid. The chemistry of sulfonyl urea herbicides has been reviewed by Beyer et al. (1987), Brown (1990), and Hay (1990). Breakdown of SM through metabolism and by chemical degradative pathways significantly decreases SM persistence in the environment. Sulfometuron persistence in the environment is summarized in Table 1. Breakdown also occurs in soils with a more complex mixture of breakdown products formed, which include those observed in hydrolysis and photolysis (Hay, 1990). The instability of SM in acid aqueous solu-

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Table 1. Sulfometuron persistence summarized from the literature.

Matrix	Location	Source	Half-life
			d
Soil	Russia	Khalikov et al. (1992)	16-18
	North Dakota	Lym and Swenson (1991)	31-65
	southern USA	Michael and Neary (1993)	5-33
	Mississippi	Trubey et al. (1998)	14
	Illinois	Trubey et al. (1998)	12
	Texas	Trubey et al. (1998)	15
	California	Trubey et al. (1998)	25
	laboratory	Anderson and Dulka (1985)	≈30 (dark, 25°C)
	Delaware	Anderson and Dulka (1985)	14-21
	North Carolina	Anderson and Dulka (1985)	14-21
Water (hydrolysis)	Saskatchewan	Anderson and Dulka (1985)	35
	laboratory	Beyer et al. (1987)	18 (pH 5, 25°C)
Water (photolysis)	laboratory	Harvey et al. (1985)	14 (pH 5, 25°C)
	laboratory	Harvey et al. (1985)	1-3 (pH 7.4-8.6)

tion may reduce its potential to move off-site and downward through the soil profile in the acid soils of southern U.S. pine forests.

Leaching of SM in agricultural and to a lesser extent forest soils has been described. Lym and Swenson (1991) did not detect SM 1.2 m downslope of treated plots with 2, 8, or 16% slopes and concluded that little lateral movement occurred in these North Dakota soils. Following addition of 45.7 cm of water over a period of 48 h in soil leaching experiments, they observed vertical movement in loamy soil columns in excess of 70 cm, but this is an extraordinary amount of water in such a short period of time and probably not representative of any actual field conditions in the USA. Dickens and Wehtje (1986), using thin layer soil chromatography and an aqueous mobile phase, indicate that SM is potentially mobile in the soil. Trubey et al. (1998) reported movement of SM to depths of 30 cm in agricultural soils and found that degradates or metabolites are generally restricted to the 0- to 15-cm layer of soil. Mobility is known to decrease with decreasing soil pH and increasing soil organic matter (Beyer et al., 1987) and is correlated to cation exchange capacity. In acid soils, Oliveira et al. (2001) found that the soil partition coefficient (K_d) for SM was strongly correlated with soil organic carbon (OC), but that the influence was small ($K_d = 0.29 + 0.12OC$; $R^2 = 0.70$), as for other weak acids. Movement in alkaline soils may be greater than in acid soils due to the higher solubility and longer persistence. Sarmah et al. (1998) described movement of two sulfonylurea herbicides (triasulfuron and chlorsulfuron) in alkaline agricultural soils (pH 8.5-9.5). These two sulfonylurea herbicides moved rapidly to a depth of 50 cm in sandy agricultural soils. Stone et al. (1993) observed movement of SM in acid, low base-saturated sandy soils typical of the National Forests in northern Minnesota, Wisconsin, and Michigan. Sulfometuron applied to soil columns dissipated within 80 d of Oust application and was not observed below 20 cm in the soil columns. There are few data available for SM movement in forest soils in the southern USA.

Michael and Neary (1993) summarized the state of knowledge of offsite movement of forestry herbicides. The fate of sulfonylurea herbicides has been reviewed (Blair and Martin, 1988). Neary and Michael (1989) monitored a 4-ha watershed on deep sandy soil in Florida

treated with SM at the rate of 0.42 kg a.i. ha⁻¹, and found offsite movement for only 7 DAT. The maximum concentration observed in water (7 µg L⁻¹) followed 54 mm of precipitation that began 1 DAT and continued to 3 DAT. There are no other published data concerning offsite movement of SM in forest ecosystems. A study with metsulfuron (another sulfonylurea acetolactate synthase inhibitor with similar chemical structure) found a maximum of 8 µg L⁻¹ in two surface water samples collected 9 DAT following an application of 84 g ha⁻¹ of metsulfuron applied as the herbicide Escort (DuPont Chemical Company, Wilmington, DE). Metsulfuron was not detected in 229 additional surface water samples collected during the two months following application to this poorly drained loamy sand in the Florida flatwoods near Gainesville, FL (Michael et al., 1991).

A few studies have reported offsite movement of SM from agricultural applications. Hubbard et al. (1989) reported that offsite movement of SM exceeded 1000 µg L⁻¹ when plots treated with 0.6 kg a.i. ha⁻¹ received extremely high-intensity precipitation (12.5 mm h⁻¹). GLEAMS (Leonard et al., 1987) model predictions did not compare well with actual observations, because GLEAMS is a long-term simulation model, not an event-based model, and is therefore not capable of predicting single-event concentrations of offsite movement (Hubbard et al., 1989). Wauchope et al. (1990) reported SM concentrations in runoff following application of 0.4 kg ha⁻¹ to agricultural sites. Simulated rainfall (69 mm h⁻¹, total < 30 mm) within 24 h of herbicide application produced runoff concentrations up to 700 µg L⁻¹. They also reported that grass-covered plots yielded the same total offsite movement as observed from bareground plots, indicating that most offsite movement occurred in overland flow as a result of the high-intensity rainfall simulation. Concentrations of SM in runoff from the agricultural applications and simulated rainfall of Hubbard et al. (1989) and Wauchope et al. (1990) are 142- and 100-fold (respectively) greater than values reported by Neary and Michael (1989) for natural rainfall on treated forest sites.

Adverse environmental impacts are a function of herbicide concentration, exposure time, and species susceptibility. Available acute toxicology data for SM (rat [*Rattus norvegicus*], mallard duck [*Anas platyrhynchos*], and bobwhite quail [*Colinus virginianus*] LD₅₀ > 5 g kg⁻¹;

Table 2. Characteristics of soil from the 0- to 15-cm depth for the Mississippi sulfometuron study sites.

Site treatment	Organic matter		CEC†	Sand	Silt	Clay
	%	pH				
Oust dispersible granule	4.0	4.5	8.78	24	44	32
1% pellet formulation	4.2	4.5	11.54	16	49	35
Control	4.2	4.5	11.08	18	48	34

† Cation exchange capacity.

bluegill sunfish and rainbow trout [*Oncorhynchus mykiss*] 96-h $LC_{50} > 12.5 \text{ mg L}^{-1}$, where LD_{50} and LC_{50} are the dose or concentration that causes the death of 50% of a group of test subjects; Beyer et al., 1987) indicates it is almost nontoxic to mammals, birds, and fish, and there is no evidence for mutagenic or teratogenic properties (Beyer et al., 1987; DuPont Chemical Company, 1983). No adverse effects were observed on embryo hatch or larval survival and growth of fathead minnows (*Pimephales promelas*) from long-term exposure to 1.2 mg L^{-1} (DuPont Chemical Company, 1983). The impacts of SM on some aquatic plant species have also been studied. Growth of *Cladophora* spp. was not reduced by exposure to 5 mg L^{-1} of SM (USDA Agricultural Research Service, 1982). Hydrilla [*Hydrilla verticillata* (L. f.) Royle], a submerged aquatic plant, was not affected by exposure to $50 \text{ } \mu\text{g L}^{-1}$ of SM, but exposure to $100 \text{ } \mu\text{g L}^{-1}$ resulted in a significant reduction in growth (USDA Agricultural Research Service, 1982). Byl et al. (1993) found that hydrilla exposed for 5 d to $10 \text{ } \mu\text{g L}^{-1}$ of SM exhibited significant increases in peroxidase activity and decreased growth, but these changes were not significant after 3 d of continuous exposure. Hartnett et al. (1987) found wild-type green alga *Chlamydomonas reinhardtii* completely resistant to $144 \text{ } \mu\text{g L}^{-1}$ SM, but growth inhibition was observed at $364 \text{ } \mu\text{g L}^{-1}$ following two weeks of continuous exposure. Wild-type chronically exposed to $364 \text{ } \mu\text{g L}^{-1}$ developed resistant strains that were unaffected by up to 36.44 mg L^{-1} . Clearly, susceptibility of aquatic species varies just as it does for terrestrial species, but because of generally shorter generation times than exist for terrestrial plants, recovery options exist for some aquatic species if environmental exposures reach the extremely high levels tested in laboratories. Rooted or Boating aquatic macrophytes should be more sensitive to SM than other aquatic organisms due to the similarity of their metabolism to terrestrial plants. Growth of Eurasian water-milfoil (*Myriophyllum spicatum* L.) was reduced at water concentrations of $1 \text{ } \mu\text{g L}^{-1}$ (USDA Agricultural Research Service, 1982).

There is little information available on environmental fate and aquatic ecosystem contamination from SM use in forest management and this lack of information hinders adequate risk analysis. This study was conducted to determine the environmental fate of SM and aquatic organism exposure on two forestry sites in Mississippi following operational application.

MATERIALS AND METHODS

Study Area

The three watersheds used in this study, two treated and an untreated control, are located near Scooba, Kemper County, Mississippi. All three sites are gently rolling typical loblolly pine (*Pinus taeda* L.) forest land. Streams draining these watersheds are ephemeral to intermittent over most of their length, but become perennial before exiting the treated area. Maximum watershed relief is about 11 m for one watershed (1.4% slope), 10 m for the second (1.2% slope), and 8 m for the control (1.0% slope). All watersheds were instrumented with standard recording rain gauges located near the center of each treated area. Stream discharge was gauged continuously at the output end of 0.5-m rectangular weirs attached to long rectangular approaches. The gauging weirs were placed in the stream at the lower perimeter of each herbicide-treated area. Programmable automatic water samplers (Isco, Lincoln, NE) were also located at the output end of the gauging weirs.

Soils for all three watersheds (Table 2) are predominately Wilcox silty clay loam to clay loam (very-fine, smectitic, thermic Chromic Dystrudert), acidic, and somewhat poorly drained.

The principal plants observed within the watersheds at the time of treatment were typical of those seen on forest sites in this area before regeneration of loblolly pine. They included grasses (*Panicum* spp.), pokeweed (*Phytolacca americana* L.), blackberry (*Rubus* spp.), oak sprouts (*Quercus* spp.), and wood sorrel (*Oxalis* spp.).

Treatments

The watersheds were mechanically site-prepared by shearing, windrowing, discing, and bedding across the slope in June 1984 followed by planting of loblolly pine in January and treatment with SM in April 1985 following an operational approach to pine regeneration. The disking and bedding are accomplished long before planting to permit adequate time for settling of the soil in the beds. Sulfometuron is normally used on these sites as a preemergence to early postemergence treatment for the control of herbaceous weeds, but application rates typically range from 0.075 to $0.26 \text{ kg a.i. ha}^{-1}$.

One watershed was treated with Oust, a commercial formulation of dispersible granules containing 75% SM (sprayed). The sprayed watershed is 253 ha, of which only the lower 148 ha (58%) was sprayed. The second watershed is 247 ha, of which the lower 44 ha were treated with an experimental pelleted formulation containing 0.9% SM (henceforth called the pelleted formulation). The target treatment rate for both SM formulations was $0.42 \text{ kg a.i. ha}^{-1}$. The control site is a smaller watershed in which 33 ha had a cultural history similar to the two treated watersheds.

Sulfometuron was applied aerially over the sprayed site early postemergence at the target rate of $0.42 \text{ kg a.i. ha}^{-1}$ (9-10 Apr. 1985). The spray was applied without surfactant in a total spray volume (water) of 112 L ha^{-1} from a Hughes H500D helicopter (McDonnell-Douglas Corp., St. Louis, MO) fitted with a 9-m Through-Valve boom. The pelleted formulation was applied at $0.37 \text{ kg a.i. ha}^{-1}$ (18 Apr. 1985) by a Hughes H500D helicopter equipped with an Isolair Series 2600 applicator-spreader (Bell Helicopter Textron, Hurst, TX). A 15-m untreated streamside management zone (SMZ) was maintained on each side of the streams throughout their lengths where water was visible at the time of SMZ establishment.

Sample Collection

Samples of water, sediment, plants, litter, and soil were collected from both watersheds, transported to the laboratory,

and frozen until analyzed. Samples collected from the untreated control watershed were used mainly in analytical methods development, freezer storage stability studies, and for quality control. The sample collection schedule for all matrices was approximately -1, 0, 1, 3, 7, 14, 30, 45, 60, 90, 120, and 180 DAT. Water was intensively sampled by prearranged timed sequence and stream flow according to precipitation events. Phosphate buffer sufficient to buffer water samples to pH 7.0 was added to each water bottle before samples were collected to prevent hydrolysis of SM.

Water

The watersheds were instrumented at 0.5-m rectangular gauging weirs with pressure transducers for water level recording and automatic water samplers capable of collecting discrete volume samples up to 1 L. Water samples were also collected by grab sampling. Time sequence samples were collected every 6 h (500 mL) and composited by the autosampler into 12-h samples for the first 37 DAT. Another automatic sampler was set to initiate sampling with an increase in stream stage without compositing. Grab samples were taken 3, 5, 9, 15, 21, 27, 35, 43, 50, 63, 92, and 12.5 DAT at 0, 30, 60, and 1.50 m below the gauging weirs. All water samples were immediately frozen and maintained frozen until thawed for analysis.

Sediment

Sediment samples were separated from water samples by decantation after the water samples had been thawed for analysis. The decanted water sample was filtered and the residue combined with the sediment sample. Suspended sediment samples were composited by week with bedload sediment collected weekly at the weir for total sediment analysis. Sediment samples were air dried, weighed, and frozen until analyzed.

Plants

Plant tissues were collected according to the predetermined sampling schedule (with minor variations due to weather, etc.) from grasses, pokeweed, blackberry, oak sprouts, and pine seedlings at 10 randomly located positions on each watershed and composited by species. The entire aboveground portion of each plant was collected when possible. Several branch tips from each of 10 plants were collected from species that were too large for collection of the entire plant to be feasible. All plant sampling was discontinued on the sprayed watershed with the 27 DAT sample and on the pelleted watershed at 60 DAT, because plants remaining on the sites were inadequate to provide sufficient material for analysis.

Litter

Litter samples were collected from three 900-cm² areas randomly selected at ridge, midslope, and toe-slope positions. These samples were composited by slope position and frozen until analyzed.

Soil

Soil samples were collected in 46-cm-deep (18-in) cores from bare ground and from under litter at three slope positions (upper, middle, and lower) by driving 5-cm-i.d., Schedule 40 PVC pipe into the ground and extracting the core. Three cores were collected from each slope location. All cores were frozen until analyzed. Immediately before analysis, the frozen cores were cut into 15-cm sections representing depth from the soil surface. Samples were composited by depth for each soil cover condition, slope position, and sampling date. Composited sam-

ples were subsampled for SM analysis with a 14-channel proportional splitter, and a subsample was taken for oven-dry weight determinations.

Analytical Methods

Sulfometuron was determined by high performance liquid chromatography in water by the method described by Wells and Michael (1987), in plants and litter by the method of Zahnow (1985a), and in sediment and soil by the method of Zahnow (1985b). Limits of quantitation (LOQ, a signal to noise ratio of 5) were 1 µg L⁻¹ for water, 50 µg kg⁻¹ for plants and litter, and 20 µg kg⁻¹ for soil and sediment. Limits of detection (LOD, a signal to noise ratio of 2) were established at 0.2 µg L⁻¹ for water, 10 µg kg⁻¹ for plants and litter, and 2 µg kg⁻¹ for soil. All values falling between the LOQ and LOD were identified as trace quantities. Average values were computed substituting half of the LOD for all samples containing trace or nondetectable amounts. Quality control (QC) samples consisted of blank and fortified samples from appropriate matrices. Materials for these samples were collected from the control watershed or as pretreatment samples from the treated watershed. Samples for QC were interspersed among actual samples for extraction and analysis. Analytical standards were included as every fourth sample during routine analysis.

Freezer storage stability studies were conducted to determine whether any degradation occurred during sample storage. Samples of water, soil, and plants collected from the study areas were fortified with analytical standard (water was buffered to pH 7), frozen simultaneously with the field samples, and analyzed periodically.

Sulfometuron dissipation was evaluated on the basis of half-life. Half-life ($t_{1/2}$) was calculated from the slope of the best fit line:

$$t_{1/2} = -0.301/\text{slope}$$

where $t_{1/2}$ is the time for SM residue level to decline to 50% of the original concentration. This equation implies first-order kinetics that seldom occurs in field situations, but half-life is often used in discussions of dissipation. For soil, half-life was calculated for the surface 15 cm of exposed soil only.

RESULTS AND DISCUSSION

Chemical Analysis and Analyte Stability

Quality control samples for each matrix were used to determine the efficiency of extraction and to ensure that the analytical method was performing as expected. Recovery of SM from fortified samples ranged from a low of 72.7% to a high of 93.6% (Table 3). Concentrations of SM in water samples were corrected for extraction efficiency, because of the importance of knowing, as exactly as possible, the exposure of aquatic organisms in this study. Recovery of SM from water samples was

Table 3. Recovery of sulfometuron from spiked quality control samples of water, sediment, plant tissue, litter, and soil.

Matrix	Number of samples	Average recovery	Standard deviation
		%	
Litter	8	12.1	9.9
Plant tissues	16	93.6	15.3
Sediment	2	90.5	3.5
Soil	30	74.7	13.4
Water	31	91.2	2.9

good and variability was low, making such corrections reasonable. Corrections were not applied to sediment, plant tissues, litter, or soil because recoveries were generally lower and highly variable.

No change in SM concentration was observed in samples stored at -20°C during the 34-week freezer storage stability study. Most samples from the field study were analyzed within 12 weeks of collection; however, a few were stored for up to 32 weeks. All water samples from the field study were buffered to pH 7 and frozen at -20°C until analyzed. Significant degradation did not occur during the freezer storage of field samples.

Sulfometuron Dissipation and Half-Life

Sulfometuron residues were higher on all plant tissue, litter, and surface soil samples from the sprayed watershed than observed in the pellet-treated watershed during the first few days following application (Table 4). Residue concentrations increased significantly in plant tissues and soil under litter on the watershed treated with the pellet formulation after sufficient precipitation had fallen (5 DAT) to activate the pellet. Following the accumulation of peak concentrations, SM dissipated rapidly from all matrices. Interception alone by vegetation, litter, and soil frequently does not result in the highest concentrations observed in these matrices, especially on sprayed sites. Additional uptake or mass transfers among the matrices may result in higher concentrations during the first several days following application. Mass transfer, presumably from mechanical causes, was observed in this study between 0 and 1 DAT. Significant losses of SM from sprayed plant tissues and litter are evident (Table 4) in the absence of any precipitation. This disappearance cannot be explained by photolysis, which occurs only in the aqueous state. A trace amount

Table 4. Average sulfometuron residue concentrations in samples collected from sulfometuron spray- and pellet-treated watersheds in Mississippi. Samples were collected at the intervals indicated following treatment, except where hyphens indicate that the sample was not collected or was lost in the analysis. The values given for plants are average values of all species collected excluding loblolly pine.

Sample matrix	Days after treatment									
	0	1	3	7	14	21	27	45	60	
	mg kg ⁻¹									
	Loblolly pine									
Sprayed	6.14	4.07	1.49	0.42	0.19	0.19	-	-	-	-
Pelleted	ND†	0.06	ND	0.06	-	0.20	ND	2.59	0.61	-
	Plants—competing									
Sprayed	24.08	21.92	11.38	2.97	1.90	0.20	-	-	-	-
Pelleted	ND	0.02	ND	0.06	-	0.05	0.11	0.02	0.03	-
	Litter									
Sprayed	8.49	5.26	6.28	2.45	1.10	0.60	0.64	0.13	0.11	-
Pelleted	2.17	5.93	-	2.10	-	2.99	2.88	2.83	ND	-
	Soil—under litter									
Sprayed	0.07	0.21	0.17	0.12	0.40	0.17	0.15	0.07	ND	-
Pelleted	0.03	ND	-	-	1.14	-	0.14	0.11	0.42	-
	Soil—bare ground									
Sprayed	0.20	0.28	-	0.37	0.18	-	0.12	ND	ND	-
Pelleted	0.20	0.06	-	ND	-	0.01	ND	ND	0.06	-

† Not detected.

of precipitation (3 mm, Table 5) occurred 2 DAT and plant residue concentrations decreased by approximately 50% during this time. This is in agreement with the findings of Michael et al. (1992), who reported that up to 100% of applied herbicide could be washed from leaf surfaces by just 3 mm of precipitation 1 h after application and approximately 30% when the 3 mm fell within 48 h of application.

Sulfometuron washed from sprayed foliage during the first 3-mm precipitation event was intercepted and retained by the litter until the next precipitation events 4 and 5 DAT. Precipitation on 4 and 5 DAT removed additional SM from foliage and washed SM into the first 15 cm of bareground soil. Litter concentrations began to decrease at this time, probably because sufficient precipitation had occurred to wet the litter and allow hydrolysis of SM to begin in this very acid environment. Litter retention of SM continued to attenuate surface soil concentrations of SM until an additional precipitation event 13 DAT washed SM from litter and into the surface 15 cm of litter-covered soil. The concentration of SM in litter-covered soil increased by 14 DAT in response to this transfer while additional leaching and hydrolysis continued to decrease the amount of SM detectable in bare-ground soil. Several additional precipitation events following 13 DAT had an insignificant impact on soil, litter, or plant concentrations via mass transfers, but did provide an environment in which SM hydrolysis could proceed.

Sulfometuron half-life was calculated for bare-ground soil (33 d), litter (9 d), pine foliage (6 d), and cornposited plants (4 d) from the sprayed watershed. The very short half-lives for plant and litter reflect the inordinately large impact of high values on regression analysis, and mechanical transfers and washoff of large portions of the applied herbicide just a few days after application. The half-life in soil was affected by these same conditions, but the impact was to attenuate the half-life be-

Table 5. Precipitation as it occurred on the days after treatment (DAT) for the watersheds treated with sulfometuron spray (Oust) and 1% pellets.

DAT	Spray applied, 10 April	Pellet applied, 18 April
	m m	
2	3	0
3	0	0
4	5	0
5	38	0
6	0	46
8	0	13
10	0	3
13	48	53
14	0	36
15	19	0
19	0	3
20	0	3
21	3	0
22	15	3
27	0	3
28	58	0
30	3	0
33	0	5
53	0	28
59	0	3
61	0	20
69	23	0

cause the soil was the ultimate receptor of herbicide transfers due to mechanical effects and washoff.

Calculation of half-life normally starts with day of application, but in the case of soil in this study, the maximum concentration did not occur until 7 DAT. Using all data from bare soil on the sprayed site in this study, soil half-life was determined to be 33 d. However, SM was not washed from foliage and into the soil until the first storm event 5 DAT. Using the residue data for samples collected after this precipitation event resulted in a half-life of 14 d. The latter half-life is very close to the hydrolytic half-life for pH 5.0 (19.8 d at 25°C; Dupont Chemical Company, 1983). Hydrolysis was probably the **most** important route of dissipation in this study.

Plant Residues

Sulfometuron was detected in cornposited plant tissue samples with a minimum detection limit of 0.050 mg kg⁻¹. Dissipation of herbicide on competing vegetation occurred rapidly. Initial residue concentrations for all species averaged about 24 mg kg⁻¹. The highest SM concentrations were observed on sprayed plants with the largest horizontal leaf intercept area (Table 6). By 27 DAT, SM had dissipated to near nondetectable levels for most sprayed species, but continued to increase in vegetation on the pellet-treated site. Loblolly pine **still** contained low levels of herbicide residue that may represent additional uptake from the soil or storage at inactive sites. Plants from pellet-treated sites showed little uptake from soil. Any plants that had emerged at the time of treatment and perennial species had small amounts of SM as the result of root uptake; however, species **that** germinated after treatment were killed and therefore did not emerge to be collected as plant samples.

Soil Residues

Soil residues were highly variable for most sampling dates. On 0 DAT the individual sample concentrations in bare-ground soil ranged from 0.125 to 0.312 mg kg⁻¹. The highest bare-ground average soil SM concentration (0.37 mg kg⁻¹) was measured 7 DAT in the upper 15 cm of soil (Table 4). This peak concentration correlates

with a concomitant loss from plant tissue that may have been intercepted by the bare ground. Sulfometuron was detected in the 15- to 30-cm depth 7, 14, and 27 DAT, but had dissipated to nondetectable concentrations by 45 DAT.

Sulfometuron residues in mineral soil samples under litter peaked 14 DAT at a concentration of 0.397 mg kg⁻¹ and dissipated to nondetectable levels by 60 DAT. The peak concentration measured 14 DAT corresponds to a precipitation event occurring 13 DAT. During this event additional SM was transferred from the litter layer to soil beneath. As with the bare-ground sample, SM residues were detected in soil at a 15- to 30-cm depth, but not in samples below 30 cm at any time during the study. Residue concentrations were highly variable, and the dissipation attenuated, presumably by the litter layer, which held a reservoir of SM for release through time into the upper level of soil. Because of the impact of the litter layer, half-life was not calculated for these data.

Precipitation and Streamflow Residue Concentrations

The first 40 d after treatment contained five storm events on the sprayed watershed and four storm events on the pelleted watershed (Table 5) that individually yielded at least 12 mm of precipitation. Because of the poor drainage on these sites, each storm event resulted in a sluggish rise and relatively rapid fall of stream stage, but base flow continued for several days at a very low rate. After 40 DAT streamflow became discontinuous and sampling was restricted to isolated pools on the treated watersheds.

A total of 200 water samples were collected and analyzed for SM from **streamflow** on the sprayed watershed and 175 on the pelleted watershed. Water from the pelleted watershed contained SM more frequently and at higher concentrations than those of the sprayed watershed (Fig. 1). Only 12.5% of water samples from the sprayed watershed contained quantifiable residues of SM compared with 70.9% of the samples from the pelleted watershed. The remaining samples contained either trace amounts or SM was not detectable in them. The pelleted formulation was an experimental formula-

Table 6. Sulfometuron concentration in various plant species through time for spray- and pellet-treated sites.

Days after treatment	Loblolly pine	Grasses	mg kg ⁻¹		Blackberry
			Sprayed	Pelleted	
0	6.14	6.89	33.60	ND	40.77
1	4.07	5.83	24.16	ND	24.86
3	1.49	5.38	14.06	ND	11.33
7	0.42	1.46	10.55	ND	2.23
14	0.19	1.63	4.55	ND	0.39
27	0.19	ND	0.82	ND	ND†
†	ND	ND	ND	ND	ND
7	ND	ND	ND	ND	ND
18	ND	ND	ND	ND	0.10
45	ND	ND	ND	ND	ND
60	0.61	ND	0.18	ND	ND

† Not detected.

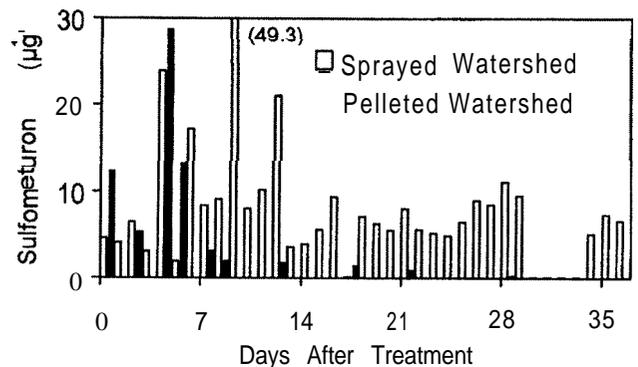


Fig. 1. Twenty-four-hour average sulfometuron concentration in streamflow from two watersheds in Mississippi treated with 0.42 kg a.i. ha⁻¹ (sprayed) or 0.37 kg a.i. ha⁻¹ (pelleted). For graphing purposes, 0.1 µg L⁻¹ was substituted for samples containing either a trace or nondetectable amounts of sulfometuron.

tion that has been dropped from consideration for registration.

Grab samples collected on the sprayed (62 samples) and pellet-treated (44 samples) watersheds contained small amounts of SM. Seven of the sprayed watershed samples contained quantifiable residues ranging from 3 to 44 $\mu\text{g L}^{-1}$ and 15 from the pelleted watershed contained SM ranging from 2.7 to 23.7 $\mu\text{g L}^{-1}$. Samples collected 150 m downstream of the sprayed watershed contained trace residues or nondetectable levels of SM for the duration of the study. Corresponding samples from the pelleted watershed also contained either trace or nondetectable amounts for the entire study except for the sample collected 7 DAT that contained 5 $\mu\text{g L}^{-1}$ SM. Therefore, movement off site was minimal or nonexistent for these two watersheds. Previous reports on picloram (Michael et al., 1989) and hexazinone (Michael et al., 1999) have demonstrated significant and linear dilution effects related to watershed size at distances of 500 to 1600 m downstream. It may be that the flat-topography, braided-stream condition close to the edge of the treated area and pretreatment disking and bedding are partially responsible for the near lack of offsite movement observed in this study.

Time sequence samples were collected every 6 h and composited to 12-h samples for the first 37 DAT. I calculated 24-h average concentrations (Fig. 1) by averaging the two 12-h composites. While the preponderance of samples with nondetectable residues of SM occurred on the sprayed watershed after the third major storm event, a few occurred just after application. For example, the 24-h average residue level of 12.3 $\mu\text{g L}^{-1}$ for 1 DAT (Fig. 1) is the result of averaging the 24.6 found in the composite sample collected at midnight and 0600 h with the nondetectable residue in the composited sample collected at 1200 and 1800 h.

Flow sequence samples were collected periodically as a function of stream stage, but not more frequently than every 15 min and were not composited. While the occurrence of SM residues was correlated with storm events, peak concentrations occurred on the pelleted watershed more than 3 h after peak discharge when antecedent soil moisture was low and more than 1 h after peak discharge when antecedent soil moisture was high (Fig. 2). Michael et al. (1999) reported water samples collected from watersheds aerially sprayed with hex-

azinone had peak herbicide concentrations at peak discharge, probably due to overland flow conditions. Pelleted hexazinone applications resulted in peak herbicide concentrations several hours after peak discharge. This latter finding is attributable to the fact that the pelleted form requires rain to break down the pellet and during this process, most of the herbicide percolates into the soil and is moved via subsurface flow. Because of the small number of samples that contained SM from the sprayed watershed, it is impossible to determine whether peak discharge occurred simultaneously with peak SM concentrations.

Samples with no detectable residues were frequently interspersed among samples with quantifiable residues, indicating that movement of SM occurred in more or less discrete pulses. The pulses were correlated with storm events. This pulsed movement is probably a reflection of SM's low K_d , which would minimize advective dispersion both in and on soil and organic matter. It is significant that the concentrations of SM measured in samples from stormflow occurred in more or less discrete pulses. The lack of quantifiable residues of SM in streamflow samples collected at 0400, 0645, 0900, 1115, and 1415 h is similarly significant (Fig. 2). The intervening samples with nondetectable levels of SM indicate that it is inappropriate to consider baseflow or stormflow contributions of SM to streamflow as occurring on a continuing basis. Other herbicides that are strongly adsorbed onto organic matter and clay may also move in discrete pulses (Michael et al., 1989).

The presentation of herbicide residues in runoff in a line graph of positive and measurable concentrations by previous authors, even when sampling periods varied from 7 to 11 d, has contributed to development of the concept that stream contamination is a continuous phenomenon. Modelers also contribute to this concept by displaying their predictions in line graph form. Perhaps the most serious aspect of this continuum concept of stream contamination is the resultant testing of herbicides in 24-, 48-, or 96-h static tests for toxic impacts on aquatic organisms. Toxicological data derived from such tests are almost impossible to interpret in light of a pattern of discrete and discontinuous streamflow contamination that results in short-term exposures for organisms within the affected habitat.

Sediment Residues

A total of five sediment samples from 3, 4, 5, 6, and 7 weeks after treatment were analyzed for SM. Suspended sediment samples did not contain SM residues at the minimum detection limit of 0.02 mg kg^{-1} . Lack of SM residues associated with sediment is a result of its low K_d and indicates that movement was principally in the dissolved state.

Environmental Consequences

The use of herbicides in forest management has resulted in concern over the nontarget impacts that may result from either their movement through the treated site, or from movement off-site. Two issues of special

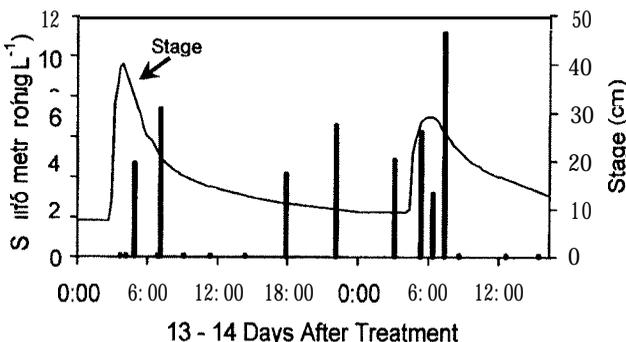


Fig. 2. Sulfometuron in stormflow following treatment with 0.37 kg a.i. ha⁻¹ on the pelleted watershed.

concern are the potential for impacts on aquatic ecosystems, and the potential for impacts on microbial populations encountered during movement through the soil column.

Herbicides are specifically designed to affect plant growth processes such as cell division, photosynthesis, and respiration. Concentrations of SM, which impact alga species like *Cladophora*, are 62 and 160 times higher than observed on the pelleted and sprayed watersheds, respectively (USDA Agricultural Research Service, 1982). Other aquatic species may be more susceptible. Growth of hydrilla is reduced by prolonged exposure to $100 \mu\text{g L}^{-1}$ SM, but not by prolonged exposure to $50 \mu\text{g L}^{-1}$, while Eurasian water-milfoil, an invasive submerged aquatic plant with adverse impacts on U.S. and Canadian aquatic ecosystems, exposed to $1 \mu\text{g L}^{-1}$ suffers reduced growth.

Herbicides can also affect nonplant organisms when concentrations are extremely high. For example, SM can kill fish when water concentrations exceed 12.5 mg L^{-1} for 96 h continuously, but at lower concentrations has no observable impact on fish. Fathead minnow embryo hatch and larval survival and growth are not affected by concentrations of 1.2 mg L^{-1} for extended periods of time. The maximum observed SM concentrations in this intensive monitoring study were 0.089 mg L^{-1} , a component of the 24-h average for 9 DAT on the pelleted watershed and 0.034 mg L^{-1} , a component of the 24-h average 5 DAT on the sprayed watershed (Fig. 1). These concentrations, which lasted for 12 h, are respectively 140 and 360 times lower than the concentrations known to kill bluegill fish, and 13 and 34 times lower than the concentrations known to have no effect on embryo hatch or larval survival and growth for fathead minnows.

Catastrophic events caused by fire (Rinne 1996), flooding (Gray and Fisher, 1981; Minckley and Meffe, 1987), drought (Deacon and Metcalf, 1961), and grazing (Rinne, 1990), which decimate fish and macroinvertebrate populations resulting in complete extirpation, may occur frequently in headwater streams. With but a few exceptions, aquatic ecosystems in general and fish populations in particular have a remarkable ability to recover following such catastrophes provided habitat is not damaged or altered (Olmsted and Cloutman, 1974). Peterson and Bayley (1993) simulated a catastrophic fish kill by poisoning fish in 45- to 113-m-long sections of 18 streams with rotenone. Recolonization began almost immediately, and recovery to 90% of original abundance occurred in four to 11 d, while recovery to 70% of original species composition required only 2.5 to 6 d. Olmsted and Cloutman (1974) reported on fish recolonization following a fish kill caused by pollution from an unknown pesticide. Complete extirpation of fish was observed over a 1.6-km reach of Mud Creek in Washington County, Arkansas on 17 May 1971. Recolonization of Mud Creek (normally 1 to 8 m in width with pools up to 1.5 m depth) began 6 d later with the migration of fish from downstream. Most species originally present had returned within three months, and repopulation was nearly complete after one year.

Macroinvertebrates are also remarkably resistant to catastrophic events. Gray and Fisher (1981) studied macroinvertebrate recolonization in Sycamore Creek following flooding and bottom scouring, which resulted in elimination of 80 to 90% of benthic invertebrates. Recolonization was principally by aerial pathways and nearly 66% of total taxa recolonized in nine weeks. Thus, recolonization can be very rapid, particularly when the affected area is relatively small. Given that recolonization can occur rapidly under such catastrophic conditions, it is reasonable to conclude that similar or more rapid rates of recolonization may occur under considerably lower levels of impact. Such is the case with SM in the watersheds in this study. Residue concentrations observed were of short duration and up to several orders of magnitude less than those shown to cause species mortality under conditions of long exposure. Thus, the potential for long-term adverse impacts on aquatic ecosystem functioning in this treated watershed is very low due to the low SM concentrations observed, and the documented ability of fishes and macroinvertebrates to rapidly recolonize following catastrophic extirpation. Neither are downstream impacts likely, because of the dilution of already low concentrations in streamflow. Sulfometuron was detected at trace levels ($<1 \mu\text{g L}^{-1}$) or not at all 1.50 m downstream of the treated watersheds after 18 DAT on the pelleted watershed and 9 DAT on the sprayed watershed.

Clearly, it is difficult to assess the potential impacts of SM on all aquatic organisms from the available toxicological data and the levels of contamination observed in this study. Contributing factors to this difficulty include variability in species susceptibility, cumulative effects, short generation times potentially leading to development of resistance, and possible interactions with many other environmental elements. Hartnett et al. (1987) reported that *Chlamydomonas reinhardtii* was completely resistant to 0.144 mg L^{-1} SM but growth was inhibited at 0.36 mg L^{-1} . Wild-type exposed to 0.36 mg L^{-1} developed resistant strains, which were unaffected by up to 36 mg L^{-1} . Exposure to sublethal concentrations may, in some species, result in development of strains that are highly resistant.

Movement of SM through soil resulted in exposure of microbial organisms in the surface 15 cm to a maximum concentration of 0.397 mg kg^{-1} . There is little direct evidence of the potential impact of such low concentrations on soil microorganisms. In the few cases studied, concentrations of SM greater than 1 mg kg^{-1} are usually required to produce any measurable impact on microbial populations, and those impacts are not always negative. Xing and Whitman (1987) studied 28 strains of methanogenic bacteria exposed to 200 mg L^{-1} . Of these 28 strains, 7 (two species) were unaffected, while an additional 14 strains were slightly inhibited. The remaining 7 strains showed some growth inhibition. Van Dyk and LaRossa (1986) found several strains of *Salmonella typhimurium* resistant to 51 mg L^{-1} . LaRossa and Smulski (1984) found *Escherichia coli* and *Salmonella typhimurium* resistant to sulfometuron methyl concentrations ranging from 0.36 mg L^{-1} to 36 mg L^{-1} . Rank (1986)

reported resistant to concentrations up to 50 mg L⁻¹. In these studies, exposures sufficient to cause some measurable impact were generally 90 to 500 times greater than observed in the sprayed watershed from the high application rate used in this study. This finding is in agreement with previous findings of herbicide interactions with soil microorganisms discussed in several reviews (Martin, 1963; Audus, 1964; Greaves et al., 1976) and more recently by Rhodes et al. (1980), Greaves et al. (1981), Repova (1985), and Tu (1993). Martin (1963) concluded that quantities of herbicides or insecticides applied to soils in agricultural situations are not sufficient to significantly depress growth of specific soil microbes. Audus (1964) also concluded that populations of microflora are not seriously damaged by herbicides, but that observations of numbers alone may not be sufficient to demonstrate impacts on specific elements in the microflora. Greaves et al. (1976) concluded that no herbicide had been found that had a long-term harmful effect on microbial activity at concentrations arising from normal agricultural usage. A survey of the literature since that time supports the position that soil microorganisms are generally resistant to the low-concentration short-term exposures, which occur from use of herbicides at labeled rates, but the relative degree of resistance is highly variable. Short-term impacts lasting a few days or weeks may be detected in some studies. Tu (1993) studied impacts of 10 herbicides on bacterial and fungal populations in soil treated with 10 mg kg⁻¹. Populations of both groups of organisms either decreased or were not different from the control group during the week after treatment, but by two weeks after treatment, the only statistically significant differences among treatments were a few cases in which populations were greater than observed in the controls. Soil microbial respiration was significantly increased over controls for all herbicide treatments. There seems to be no evidence, direct or indirect, that herbicide use in forestry in general, or specifically SM use, adversely affects soil microbial populations to the extent that one could expect any impact on present or future soil productivity.

While there is much we do not know about SM, this research indicates that on forest sites like those in the southern USA, where soil and water conditions are acidic, sometimes extremely so, SM is short-lived. The half-lives calculated from this research are in line with those reported from Russia, Canada, and around the USA (Table 1). The principal routes of degradation appear to be hydrolysis and photolysis. However, where soil and water conditions are neutral or basic, rates of hydrolysis (but not photolysis) decrease and aqueous solubility increases (Hay, 1990). Under these conditions, longer half-lives and increased potential for off-site movement would probably be observed. Adverse impacts on watersheds in the southern USA are unlikely.

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