Fate of the herbicide sulfometuron methyl (Oust®) and effects on invertebrates in drainages of an intensively managed plantation[1]

J.L. Michael, D.P. Batzer, J.B. Fischer, and H.L. Gibbs

Abstract: The off-site movement and impacts on water quality and aquatic ecosystems of sulfometuron methyl applied as the herbicide Oust® to catchments in short-rotation plantations in the coastal plain of South Carolina were studied. Sulfometuron methyl was applied at the rate of 0.053 kg active ingredient·ha^{-1} to 5.4- and 5.9-ha catchments (C5 and C6, respectively). Off-site movement of sulfometuron methyl in drainage ditches was observed between application on 14 March 2001 and 14 June 2001 for the first five flow-producing rain events on C5 and the first four events on C6. The maximum observed concentrations (24 μg·L^{-1} on C5 and 23 μg·L^{-1} on C6) occurred during the first storm. Subsequent maximum concentrations for flow-producing storms were 10.0, 5.0, 0.5, and 0.1 μg·L^{-1} on C5 and 15.1, 6.7, and 0.5 μg·L^{-1} on C6. Pulsed inputs of sulfometuron methyl to stormflow were ephemeral and the maximum concentration for each storm event lasted 15 min or less. The faunal communities observed in these drainage ditches were dominated by a diversity of invertebrates typical of wetland habitats, such as midges, mosquitoes, water beetles, physid snails, and water fleas. Negative effects of sulfometuron methyl treatment on these communities in treated watersheds were not observed.

Résumé : Nous avons étudié le déplacement à l'extérieur du site ainsi que les impacts sur la qualité de l'eau et les écosystèmes aquatiques du sulfométuron de méthyle appliqué sous forme d'herbicide Oust® dans des bassins hydrographiques aménagés en plantations à courtes rotations, dans la plaine côtière de la Caroline du Sud. Le sulfométuron de méthyle a été appliqué au taux de 0,053 kg de matière active à l'hectare dans des bassins de 5,4 et 5,9 ha (nommés respectivement C5 et C6). Le déplacement du sulfométuron de méthyle à l'extérieur du site dans les canaux de drainage a été observé durant les cinq premiers épisodes de pluie causant un écoulement entre les applications d'herbicide le 14 mars et le 14 juin 2001 dans C5, de même que durant les quatre premiers épisodes dans C6. Les concentrations maximales (24 μg·L^{-1} dans C5 et 23 μg·L^{-1} dans C6) ont été observées durant la première averse. Les concentrations maximales durant les averses subséquentes avec écoulement ont été de 10,0, 5,0, 0,5 et 0,1 μg·L^{-1} dans C5 et de 15,1, 6,7, et 0,5 μg·L^{-1} dans C6. Les apports saccadés de sulfométuron de méthyle dans les eaux de ruissellement ont été éphémères et la concentration maximale s'est maintenue au plus 15 min lors de chaque averse. Les communautés fauniques observées dans les canaux de drainage étaient dominées par une diversité d'invertébrés typiques des milieux humides, notamment des moucherons, des moustiques, des coléoptères d'eau, des escargots et des puces d'eau. Les effets négatifs du traitement au sulfométuron de méthyle sur ces communautés n'ont pas été observés dans les bassins traités.

[Traduit par la Rédaction]

Introduction

Sustainable production from intensively managed plantations is dependent on development of highly productive growing stock and on management practices that conserve the biotic and abiotic components of soil and water resources. Such a system can provide high fiber yields over multiple rotations and protect environmental quality.

Protection of water quality and aquatic ecosystems in the United States is mandated by federal and state governments under the Federal Insecticide, Fungicide, and Rodenticide Act; the National Environmental Policy Act 1969 and as amended; the Endangered Species Act 1973; and the Clean Water Act 1977 and as amended, which define the quality of water in terms of its physical, chemical, and biotic components. The Acts establish water-quality standards that prohibit activities that degrade existing conditions, establish acceptable levels of pollutants, and protect designated uses. These regulations are integrated under Best Management Practices, the regulatory authority for which is vested in state governments. However, most Best Management Practices for silvicultural chemicals focus on the use of streamside management zones (SMZs) that increase in width as stream width increases for the protection of water quality. Michael (2004) found that 70%–90% of states fail to recom-

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mend protection of ephemeral channels, drainage ditches, or canals even if these are intimately associated with perennial streams, and concluded that "increasing the protection of streams to include the ephemeral channels would decrease the amount of stream contamination from aerial applications". This is also likely to apply to ground applications.

Routine applications of herbicides in association with site preparation have been the foundation of intensive long-rotation pine plantations for the last 40 years. In long-rotation plantation management, herbicide use occurs once or twice during 20- to 80-year rotations. Even this minor use has spawned much public concern. In intensively managed short-rotation plantations, weed control may be used several times during the first year and then annually for several years following planting.

Oust® (E.I. DuPont de Nemours, Wilmington, Delaware, USA) is one of the most popular herbicides for weed control in short-rotation systems and is used chiefly in the early phases of establishment. The active ingredient (a.i.) of Oust® is sulfometuron methyl (methyl 2-[[[[(4,6-dimethyl-2-pyrimidinyl)amino]carbonyl]amino]sulfonyl] benzoate). One member of a class of herbicides that inhibit acetolactate synthase (ALS or AHAS), sulfometuron methyl is herbicidally active at very low rates. It is registered for use in forest sites at rates up to 0.420 kg a.i.·ha^{-1} (http://www.dupont.com/ag/us/prodinfo/prodsearch/information/H64342.pdf) and its chemical and physical properties have been reviewed (DuPont Chemical Company 1983; Anderson and Dulka 1985; Harvey et al. 1985; Beyer et al. 1987; Brown 1990). Sulfometuron methyl is a weak acid with pK_a of 5.2 at 25 °C (Beyer et al. 1987). Ionization occurs at the sulfonylurea bridge nitrogen imparting a shared negative charge across the bridge. It is this pH effect that affects sulfometuron methyl's solubility in water, hydrolysis, and partition coefficients (soil, K_d; organic carbon, K_{oc}; and octanol–water, K_{ow}), and therefore its behavior in soil and water. This pH effect is significant, since forest ecosystems range widely in pH from acidic to alkaline and because different matrices (leaf litter, organic and mineral soils) may have pH values that range above and below pK_a.

The sulfometuron methyl molecule is hydrophobic below its pK_a and so water solubility is reduced. The aqueous solubility of sulfometuron methyl at 25 °C is 8 mg·L^{-1} at pH 5 and 70 mg·L^{-1} at pH 7 (Beyer et al. 1987). Solubility of the acid is 10 mg·L^{-1} at pH 5 and 300 mg·L^{-1} at pH 7 (Harvey et al. 1985). The hydrolytic half-life for sulfometuron methyl at pH 5.0 is approximately 14 days, while at higher pH the half-life is much longer, with 87% remaining intact at pH 7 and 91% at pH 9 after 30 days (Harvey et al. 1985). Photolysis, however, occurs even under alkaline conditions, with a half-life of 1–3 days at pH 7.4–8.6 (Harvey et al. 1985). Thus, in shallow lotic or lentic systems above pH 5.2, sulfometuron methyl may be highly susceptible to degradation by photolysis and below pK_a may also degrade by hydrolysis.

The pH effect on the partition coefficients (K_d, K_{oc}, and K_{ow}) is related largely to the hydrophobicity of sulfometuron methyl at low pH and the affinity of the unionized molecule for soil organic matter. Reported K_d values range from 0.04 L·kg^{-1} (Koskinen et al. 1996) in acidic, less organic sands to 1.18 L·kg^{-1} in acidic, highly organic soils (Oliveira et al. 2001). Oliveira et al. (2001) found that in soils with pH below pK_a for sulfometuron methyl, K_d was positively correlated with the amount of soil organic matter. Similarly, reported K_{oc} values range from 5.4 L·kg^{-1} (Koskinen et al. 1996) to 50 L·kg^{-1} (Oliveira et al. 2001) and are correlated with the amount of organic matter and pH. K_{ow}, is larger when the aqueous medium is acidic than when it is basic, and reported K_{ow} values range from 15 at pH 5 to 0.31 at pH 7 (Beyer et al. 1987).

The physical characteristics and low partition coefficients (Harvey et al. 1985; Wehtje et al. 1987) of sulfometuron methyl allow for its rapid off-site movement from, or degradation in, the upper few centimetres of soil. Breakdown of sulfometuron methyl through metabolism and by chemically degradative pathways significantly reduces its persistence in the environment. Breakdown also occurs in soils, when a more complex mixture of breakdown products is formed, which include those observed during hydrolysis and photolysis (Hay 1990). The instability of sulfometuron methyl in acid aqueous solution may reduce its potential to move off site and downward through the soil profile in the acid soils of southern pine forests, but surface or subsurface movement will also be affected by the degree to which photolysis can occur.

The fate of sulfometuron methyl has been reviewed, but there has been little research on its fate and ecosystem impacts following its use in forest-vegetation management (Blair and Martin 1988; Michael 2003). Neary and Michael (1989) monitored a 4-ha watershed on deep sandy soil (pH 4.0) in Florida treated with sulfometuron methyl at the rate of 0.42 kg a.i.·ha^{-1}, and found that off-site movement occurred for only 7 days after treatment. The maximum concentration observed in water (7 μg·L^{-1}) followed 54 mm of precipitation that began 1 day after treatment and continued until 3 days after treatment. Stone et al. (1993) observed movement of sulfometuron methyl in acid, low-base-saturated sandy soils typical of the National Forests in northern Minnesota, Wisconsin, and Michigan. Sulfometuron methyl applied to soil columns dissipated within 80 days of application of Oust® and was not observed below 20 cm in the soil columns. Few data are available on sulfometuron methyl movement in forest soils in the southern United States. Michael (2003) described the off-site movement of sulfometuron methyl in stormflow and its vertical movement through soil (pH 4.5) after application of 0.42 kg a.i.·ha^{-1} to forest sites in Mississippi. In this study, sulfometuron methyl remained mostly in the upper 15 cm of soil, and was infrequently detected below the limits of quantitation for the HPLC analytical method (Wells and Michael 1987) at depths to 30 cm. Off-site movement in stormflow in the presence of streamside management zones was pulsed and ephemeral, with a maximum concentration of 44 μg·L^{-1}.

Sulfometuron methyl impacts on aquatic ecosystems are not well understood and there is little information beyond that required for pesticide registration under the Federal Insecticide, Fungicide, and Rodenticide Act. Growth inhibition of rooted macrophytes has been demonstrated and at least some may be very sensitive, provided exposure is sufficiently long. The concentration at which stem growth is reduced by 50% (EC_{50}) for shortspike watermilfoil, *Myriophyllum sibiricum* Komarov, is 0.3 μg·L^{-1} following

14 days of static exposure (Roshon et al. 1999), and it is 10 $\mu g \cdot L^{-1}$ for waterthyme, *Hydrilla verticillata* (L. f.) Royle (Syracuse Environmental Research Associates, Inc. 1999). The EC_{50} for swollen duckweed, *Lemna gibba* L., a floating macrophyte, is less than 0.5 $\mu g \cdot L^{-1}$ but the no-observable-effect level is only slightly less at 0.3 $\mu g \cdot L^{-1}$. Even less is known about the impacts of sulfometuron methyl on benthic macroinvertebrates. The macroinvertebrate test organism for sulfometuron methyl registration was *Daphnia magna* Straus, the no-observable-effect level for which was 2400 $mg \cdot L^{-1}$. The susceptibility range for benthic macroinvertebrates is not known.

The impacts of the increased intensity of herbicide use in short-rotation plantations have not been adequately studied in the southeastern United States, particularly with respect to water quality and aquatic organisms. Therefore, understanding the fate of applied herbicides, which are essential components of intensively managed short-rotation plantations, is fundamental to assessing the sustainability of management practices. This is particularly true as management intensity increases in both pine and hardwood plantations. This study was conducted to determine the environmental fate of sulfometuron methyl, the degree of exposure of aquatic organisms, and the impacts of that exposure on macroinvertebrate populations in drained catchments in the lower coastal plain of South Carolina.

Materials and methods

Study area

Trice Experimental Forest is an experimental tract owned and managed by International Paper and located near Sumter, South Carolina. The site is representative of the south Atlantic upper coastal plain and the study areas were established in 1997 under the Oak Ridge National Laboratory's Agenda 2020 research program. It was under agriculture for more than 150 years prior to its establishment as a research area. Detailed descriptions are given by Trettin and Davis (2002). The surface agricultural drainage network on the site was used to define the small catchments (3.4–9 ha) used in this study and surface runoff was controlled by berms surrounding each catchment and a drainage ditch in the center of each catchment. Each drainage ditch was protected from spray by a vegetative strip approximately 3 m wide on each side of the ditch. Four catchments (C1, C3, C5, C6) were used in the herbicide portion of this study. Four more catchments (C2, C4, and two additional reference catchments located in the contiguous watershed 2) were also used in the portion of this study examining the impact on invertebrates. All catchments are proximal to each other (C1–C6 in watershed 1 and two reference cathments in watershed 2) and are very similar in soils, physical characteristics, and pretreatment cover vegetation. The watersheds are flat with 0%–2% slope and sandy loam soil predominantly in the Goldsboro Series. Soil bulk density is 1.39–1.69 $g \cdot cm^{-2}$, with 0.9%–1.3% organic matter, and pH ranges from 5.6 to 6.0. Two of the catchments were planted with sweetgum, *Liquidambar styraciflua* L., and four with sycamore, *Platanus occidentalis* L.

Four of the catchments were selected for use in the herbicide-movement portion of this study. The two sweetgum catchments, C5 (5.4 ha) and C6 (5.9 ha), were selected for herbicide treatment because competing weed control was needed. The two sycamore catchments, C1 (3.4 ha) and C3 (6.8 ha), were used as reference. Pretreatment samples of fauna indicated that there were no differences between the catchments. The catchments were instrumented with flumes and pressure transducers connected to recording flowmeters. During rain events when the stream stage in drainages rose above a threshold of 5 mm, flowmeters were set to trigger automatic water samplers.

The remaining catchments, C2 and C4, received operational treatments (aerially applied) with sulfometuron methyl at the same rates as C5 and C6 and were subsequently used in sampling invertebrates. C2 and C4 and the two reference catchments in watershed 2 were included to increase the statistical power of the invertebrate sampling. Pretreatment samples indicated that no differences in benthic macroinvertebrate fauna existed among the eight catchments.

Treatments

Sulfometuron methyl was applied in the Oust® 75 DG formulation in 235 $L \cdot ha^{-1}$ water by means of a tractor-mounted boom sprayer to catchments 5 and 6 on 14 March 2001. The application rate was 0.053 kg $a.i. \cdot ha^{-1}$. Catchments 2 and 4 were treated at the same rate but the herbicide was aerially applied. Oust® 75 DG contains 75% sulfometuron methyl and was obtained from E.I. DuPont de Nemours Chemical Company, Wilmington, Delaware, USA.

Collection and storage of water samples

Samples of storm-runoff water were collected in 1-L wedge-shaped high-density-polypropylene autosampler bottles with Styrofoam®-lined screw caps, using Isco Model 3700 automatic samplers (Isco Inc., Lincoln, Neb.). Samples of neat water from the treated sites had pH values ranging from 6.19 to 6.21. Each bottle was dosed with 1 mL of 1M potassium phosphate buffer (pH 7.0) prior to mounting in the sampler to preserve the samples and insure that the sample pH would not drop below pK_a. Samplers were set to collect 800–900 mL per bottle to leave space for expansion on freezing. Samples were retrieved from the Isco samplers within 0–4 h after the completion of a sampling cycle (i.e., 6–10 h after sampler initiation, since samplers ran for 6 h to collect 24 samples at 15-min intervals). All samples were immediately transferred to freezers at the site for transport to the George W. Andrews Forestry Science herbicide-analysis laboratory in Auburn, Alabama. Samples were kept frozen at or below –15 °C until analyzed. Pretreatment water samples were used in developing analytical methods, and pre- and post-treatment samples from the catchments, including the controls, were used in preparing spike and blank pairs for quality control in the analytical process.

Analytical methods

There were no commercially available ELISA kits for sulfometuron methyl at the time of this study. A kit for the related herbicide metsulfuron methyl was, however, available on special order from Strategic Diagnostics Inc. (Newark, Delaware) as Part No. 74600. That kit exhibited considerable cross-reactivity for other sulfonylurea herbicides, including sulfometuron methyl. We calibrated our kits

(Lot 1F1129) using standards made with sulfometuron methyl (99 ± 0.5%, Lot 255–73A) obtained from Chem Service, Inc. (West Chester, Pennsylvania) in place of the metsulfuron methyl standards supplied. Based on results from the kits tested with analytical-grade sulfometuron methyl, conservative calibration standard levels for sulfometuron methyl of 0.00, 0.100, 0.500, and 2.0 $\mu g \cdot L^{-1}$ were chosen for routine sample analyses. In a typical 96-well ELISA plate analysis, 12 wells were used for blanks and calibration standards, each in triplicate. Two wells were reserved for a blank and spiked quality-control pair using pretreatment water from catchments C5 and C6 and an untreated control site (C3), leaving 82 wells for actual runoff samples from treated catchments. Plate absorbances were measured using a Ceres 900 plate reader (Bio-Tek, Winooski, Vermont). Response curves and sample concentrations were calculated using Kineticalc II software installed on the plate reader's data system. Samples were returned to freezer storage promptly after analysis. Samples were first analyzed by direct-injection (no clean-up) HPLC to determine which were within range of the ELISA kit. Any samples that were out of range, i.e., above 2 $\mu g \cdot L^{-1}$, were diluted and analyzed by ELISA. Using this technique we established the ELISA lower limit of detection at 0.075 $\mu g \cdot L^{-1}$ and the lower limit of quantitation at 0.20 $\mu g \cdot L^{-1}$. Any values between the lower limit of detection and the lower limit of quantitation were recorded as trace values, possibly present but not reliably quantifiable. All data presented are from the ELISA analyses.

Sample preparation and analysis for sulfometuron methyl

Water samples were retrieved from freezers the day before analysis and thawed by immersion in lukewarm water. Bottles were first shaken and then allowed to settle for at least 1 h. Approximately 10 mL (±0.2 mL) subsamples were decanted into 17 mm × 100 mm graduated polystyrene culture tubes with polyethylene caps (USA Scientific Inc., Ocala, Florida) and used for analysis. For 1:9 or 1:19 dilutions, 1.0 or 0.5 mL, respectively, was transferred using an Eppendorf 100- to 1000-mL adjustable pipette (Brinkmann, Westbury, N.Y.) and diluted to 10 mL with high-purity water from a Milli-Q® Plus system (Millipore, Bedford, Mass.). Dissolved organic substances (humic acids) were found to interfere with the ELISA test. To remove these humic acids each quality control pair and all field samples were treated with divalent lead prior to analysis. Undiluted water samples were treated by adding 50 µL of 1.0 $mol \cdot L^{-1}$ lead (II) acetate dihydrate (ACS Reagent, J.T.Baker, Phillipsburg, N.J.) using an Eppendorf repeater pipette (Brinkmann). Diluted samples were treated with 50 µL of diluted lead acetate (0.1 $mol \cdot L^{-1}$ for 1:9 dilutions and 0.05 $mol \cdot L^{-1}$ for 1:19 dilutions). All tubes were capped immediately, inverted 2–3 times to homogenize the contents, and allowed to stand for at least 45 min. The tubes were then centrifuged at 1066*g* for 30 min to settle out any precipitates and analyzed as above.

Aquatic invertebrates

Aquatic invertebrates were sampled in (*i*) the two ditches (C5 and C6) that drained the tree stands experimentally treated with Oust®, (*ii*) two other drainage ditches at Trice Experimental Forest that had been treated operationally with Oust® (C2 and C4), and (*iii*) four nearby reference drainage ditches where Oust® was not applied (including C1 and C3). Samples were collected on (*i*) 2 March, 12 days prior to Oust® applications, (*ii*) 27 March, 13 days after experimental applications, and (*iii*) 2 May, 7 weeks after application. In each of the eight drainage ditches on each sampling date, invertebrates were collected at three or four randomly selected locations using a standardized 1 m sweep of a D-frame net (30 cm width, 1 mm mesh). This sampler was used because the ditches had wetland characteristics (minimal flow, heavily vegetated) and a sweep net is considered the optimal device for characterizing invertebrate assemblages in freshwater wetlands (Rader et al. 2001). Samples from the ditches treated with sulfometuron methyl were collected in the immediate vicinity of the gauging stations. Samples from reference catchments were collected in a similar manner. Samples were preserved in 95% ethanol and transferred to the laboratory for processing. Invertebrates were manually removed from mud and plant debris using dissecting scopes, identified to family or genus using keys in Pennak (1989), Thorp and Covich (1991), and Merritt and Cummins (1996), and quantified. Invertebrate family richness and the abundances of common organisms from the four Oust®-treated and four reference watershed ditches were compared using one-way ANOVA. Subsamples collected in each ditch were pooled for analysis, so the units for comparison were whole ditches.

Table 1. Precipitation recorded on catchments C5 and C6 at Trice Experimental Forest during the study of sulfometuron methyl (Oust®) dissipation in 2001.

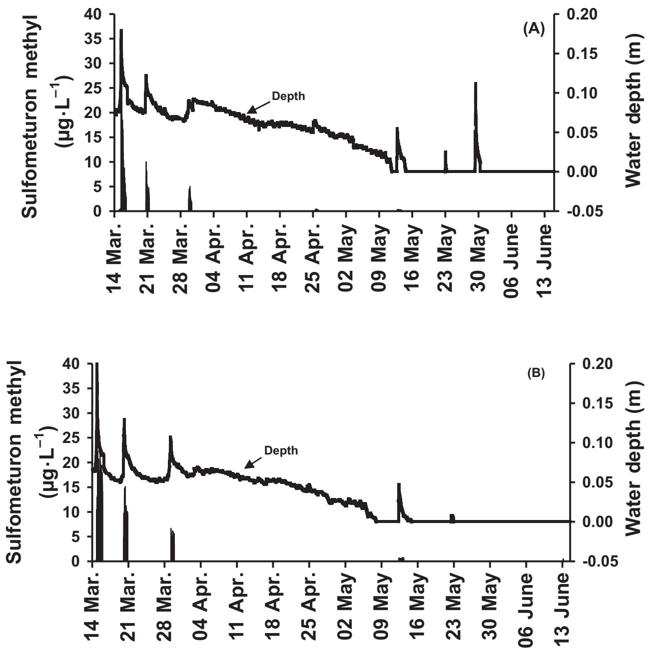
	Precipitation (mm)
15 March 2001	32.2
20 March 2001	19.4
29 March 2001	17.5
25 April 2001	08.9
12 May 2001	15.3
22 May 2001	11.1
29 May 2001	39.9
8 June 2001	11.9
13 June 2001	28.2

Results and discussion

Precipitation

Precipitation was recorded continuously for the period of the study. Nine events of sufficient intensity and duration to cause a rise in stage of at least 5 mm occurred during the 92 days of this study (Table 1 and Fig. 1). The smallest of these, 8.9 mm, occurred in the first half of the day on 25 April 2001 and resulted in water-sample initiation on catchment C5 but did not provide sufficient flow for sample initiation on C6. Once samplers were initiated, sampling continued, with samples collected at 15-min intervals. The largest event occurred on 29 May, but did not initiate sampling on the droughtier C6.

Fig. 1. Movement of sulfometuron methyl off catchments C5 (A) and C6 (B) in stormflow following ground application of 0.053 kg a.i.·ha^{-1} in the Oust® herbicide formulation.



Analysis of sulfometuron methyl

Pretreatment storm samples from C3, C5, and C6 were collected and analyzed. They did not contain quantifiable amounts (>0.2 μg·L^{-1}) of sulfometuron methyl. These samples were then used in preparation of the quality-control samples (a blank and spiked pair on each ELISA plate) as part of the quality-assurance program. A total of 14 quality-control blank and spike pairs were analyzed. The quality-control blanks did not contain measurable levels of sulfometuron methyl (mean = 0.061 μg·L^{-1}, SD = 0.054 μg·L^{-1}, n = 14). Standard curves were computed for each of the plates, based on triplicate analysis of each of four sulfometuron methyl concentrations (0.0, 0.1, 0.5, and 2.0 μg·L^{-1}). R^2 values for the calibration curves (one for each 96-well plate) ranged from 0.955 to 0.99.

Dissipation of sulfometuron methyl

Dissipation from the treated catchments began immediately, as a rain event started within minutes after application to C5 was completed. Over the following 11.5 h a total of 32.2 mm fell on the catchments. There were nine precipitation events over the course of this study that were sufficient to initiate sampling. Figure 1 shows the details of sulfometuron methyl dissipation in this study. In general, the dissipation of sulfometuron methyl followed the patterns observed for other watersheds and chemicals in the southern USA (Michael and Neary 1993). Pulsed inputs of herbicide are typically seen at their highest concentration in the first storm, with peak concentrations continuing to decrease with each new precipitation event. Maximum observed concentrations of sulfometuron methyl in flow from these catchments lasted no more than 15 min, with subsequent samples containing generally decreasing concentrations. The sulfometuron methyl concentration dropped below 0.50 μg·L^{-1} by 25 April on C5 and by 13 May on C6.

The short duration of peak herbicide concentrations in stormflow has been reported in numerous papers and is the result of a combination of factors (Michael and Neary 1993; Michael et al. 1999; Michael 2003, 2004). The variable-source-area watershed concept — that the source of runoff expands in area through an increase in flow from intermittent and ephemeral channels — is of primary importance in sites with complex topography and to a lesser extent in other sites. In addition, where overland flow and macropore flow occur, they potentially carry high concentrations of herbicide to streams and drainage ditches. The greater the capacity for movement of water (and therefore soluble herbicides) through the soil profile, the lower the potential for overland flow, but this does not preclude pulsed input into streams and drainage ditches. Where soil partition coefficients are low and herbicide concentration is high, advective dispersion is much less effective in retarding herbicide movement. Pulsed inputs occur as the soil acts virtually as a chromatographic medium in which the solvent front (water in this case) carries with it the highest possible amount of soluble herbicide to streams and drainage ditches. Subsequent peak stormflow concentrations decrease because there is less available herbicide to contribute to these peak stormflow events (Michael 2004).

The highest concentrations observed in runoff from the catchments were 24 μg·L^{-1} on C5 and 23 μg·L^{-1} on C6. Runoff from C6 was more rapid and it was also a droughtier catchment, as demonstrated by the lack of runoff from precipitation events of 29 May and 8 June (Fig. 1). Michael and Neary (1993) and Michael (2003) report maximum streamflow concentrations in the range from 7 μg·L^{-1} (deep sand) to 44 μg·L^{-1} (acidic silty clay loam to clay loam, poorly drained) from an application rate of 0.42 kg a.i.·ha^{-1}, 8 times higher than those used in this study. However, the Mississippi soils were typically around pH 4.5 (Michael 2003), while those at Trice Experimental Forest were around pH 5.8. Under the acidic conditions in the Mississippi soils, which contained 4% organic matter, sulfometuron methyl would be more strongly absorbed into the soil and exist primarily in the non-ionized form, making it less soluble in water and less available for movement off site than under the conditions at Trice Experimental Forest.

Aquatic invertebrates

The invertebrate fauna of the watershed ditches (Table 2) was dominated numerically by midges (Diptera: Chironomidae), mosquitoes (Diptera: Culicidae), predaceous diving beetles (Coleoptera: Dytiscidae), snails (Gastropoda: Physidae), water fleas (Anopomoda: Daphniidae), and aquatic worms (Oligochaeta: Tubificidae). Pretreatment collections were composed almost exclusively of mosquitoes (which colonized or hatched quickly after ditches flooded), and ditches assigned for treatment had similar numbers of invertebrates to those assigned as references (P > 0.05). Post treatment, neither invertebrate family richness nor total invertebrate abundance (Fig. 2) varied significantly among the four Oust®-treated and four reference watersheds, either 13 days post treatment or 7 weeks post treatment (all P > 0.10). The abundances of all individual taxa were also simi-

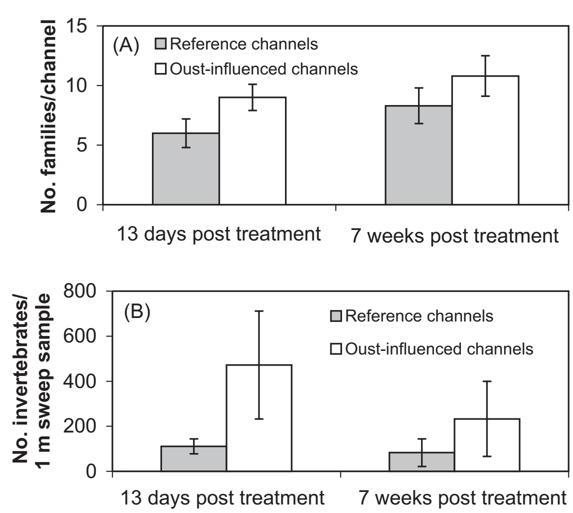
Table 2. The invertebrate fauna of watershed channels (drainage ditches) in six experimental catchments at Trice Experimental Forest.

	No. per sweep sample[a]
Mollusca	
Gastropoda	
Physidae	3.9 (1.5)
Physa	
Planorbidae	1.3 (0.8)
Gryaulus	
Promenetus	
Bivalvia	
Sphaeriidae	4.3 (2.1)
Annelida	
Oligochaeta	
Tubificidae	5.0 (1.7)
Hirudinea	
Glossophoniidae	*
Arthropoda	
Crustacea	
Copepoda	
Cyclopidae	0.5 (0.2)
Anopomoda	
Daphniidae	19.0 (5.7)
Conchostraca	
Lynceidae	0.2 (0.2)
Amphipoda	
Crangonyctidae	*
Decapoda	
Cambaridae	0.4 (0.2)
Insecta	
Ephemeroptera	
Baetidae	0.3 (0.1)
Callibaetis	
Odonata	
Coenagrionidae	*
Enallagma	
Ishnura	
Libellulidae	0.1 (0.1)
Libellula	
Sympetrum	
Hemiptera	
Corixidae	0.3 (0.2)
Trichocorixa	
Gerridae	0.2 (0.1)
Gerris	
Limnoporus	
Trichoptera	
Polycentropodidae	*
Cernotina	
Coleoptera	
Dytiscidae	4.3 (0.6)
Agabus	
Copelatus	
Coptotomus	
Hydroporus	
Hygrotus	
Laccophilus	
Liodessus	
Rhantus	
Thermonectes	
Uvarus	
Haliplidae	0.2 (0.1)
Peltodytes	
Hydrophilidae	0.9 (0.2)
Berosus	
Enochrus	
Helocombus	
Helophorus	
Tropisternus	
Diptera	
Chironomidae	72.7 (20.1)
Chironomus	
Polypedilum	
Dixidae	0.1 (0.1)
Ceratopogonidae	0.2 (0.1)
Culicidae	3.8 (0.8)
Aedes	
Anopheles	
Culex	
Culiseta	
Chaoboridae	0.3 (0.2)
Chaoborus	
Ephydridae	*
Sciomyzidae	*

Note: Genera identified in each family are listed, but generic abundance was not quantified because many individuals (early instars) could not be reliably classified to genus. The community was quantified by family. An asterisk indicates a rare taxon (<0.1 individual /sample).

[a]Values are given as the mean with standard error in parentheses.

Fig. 2. Invertebrate family richness (A) and total invertebrate abundance (B) (mean ± 1 SE) in the Oust®-treated and non-treated channels at Trice Experimental Forest.



lar among treatments on both post-treatment dates (all $P > 0.05$), with the exception of physid snails, whose numbers were greater in the Oust®-treated channels than in the reference channels on both sampling dates. The value of family- versus species-level identification in aquatic invertebrate bioassessment is being debated (Bailey et al. 2001; Lenat and Resh 2001). However, in this study it is unlikely that the use of family classification masked any negative impacts of Oust® because family richness was modestly greater in Oust®-treated channels (Fig. 2), and greater taxonomic resolution should not dramatically reverse that trend. There was no evidence that Oust® negatively influenced invertebrates.

Although watershed drainage ditches such as those studied in this project are sometimes referred to as streams, the invertebrate community residing in the ditches was clearly wetland in nature. Mayflies (Ephemeroptera), stoneflies (Plecoptera), and caddisflies (Trichoptera), typically the most common, diverse, and environmentally sensitive insects in streams, were rare or absent in the watershed drainage ditches. Instead, communities were dominated by a diversity of invertebrates typical of wetland habitats such as midges, mosquitoes, water beetles, physid snails, and water fleas (Table 2; Batzer et al. 1999).

Wetland faunas are adapted to environmental stress and in this study they did not respond to Oust® treatment, even though exposure levels were relatively high. The few taxa that occur in both these watersheds and regional streams are all classified as being highly tolerant in the North Carolina Stream Biotic Index (Lenat 1993). However, most taxa from the watershed ditches are not even listed in that index because they do not occur in streams. If invertebrates are to be used as bioassessment organisms in drainage ditches of Coastal Plain plantations, it should be recognized that researchers will be dealing with a wetland fauna. Invertebrate metrics that are useful for detecting environmental impacts are just beginning to be used for wetlands (see Rader et al. 2001), although none has been developed for aquatic invertebrates residing in ditches or ephemeral wetland channels.

Environmental consequences

The environmental consequences of the use of sulfometuron methyl in forest vegetation management have been reviewed (Michael 2003). Two issues of special concern in connection with the use of herbicides in forest vegetation management are the potential for impacts on aquatic ecosystems and the potential for impacts on microbial populations encountered during movement through the soil column (Michael 2003). This study has examined impacts of sulfometuron methyl on aquatic ecosystems.

Herbicides are specifically designed to affect plant growth processes: cell division, photosynthesis, respiration, etc. They can also affect non-plant organisms when exposures are sufficiently high. Sulfometuron methyl can kill fish when water concentrations exceed 12 500 $\mu g \cdot L^{-1}$for 96 h continuously; this is more than 500 times higher than the highest concentration observed for a 15-min period in this study. Lower concentrations have no observable impact on fish. Hatching of fathead minnow embryos, and larval survival and growth, are not affected by concentrations of 1200 $\mu g \cdot L^{-1}$ (>50 times higher than the concentrations observed in this study) for extended periods of time.

Sulfometuron methyl concentrations that impact alga species like those of the genus *Cladophora* (a floating green alga) are more than 200 times higher than those observed on these study catchments (USDA Agricultural Research Service 1982). Other aquatic species may be more or less susceptible. For example, growth of Eurasian watermilfoil (*Myriophyllum spicatum* L.), a non-native invasive submersed, rooted perennial that prefers water 0.5–3.5 m in depth, was reduced in water with sulfometuron methyl concentrations of 1 $\mu g \cdot L^{-1}$ (USDA Agricultural Research Service 1982), and Roshon et al. (1999) found that 0.22 $\mu g \cdot L^{-1}$ reduced root dry mass of *M. sibiricum* by 50% (EC_{50} or IC_{50}) exposed to a static concentration for 14 days. Somewhat less sensitive are *L. gibba* (EC_{50} = 0.50 $\mu g \cdot L^{-1}$), *Selenastrum capricornutum* Printz (120-h EC_{50} = 4.6 $\mu g \cdot L^{-1}$), and *H. verticillata* (EC_{50} = 10 $\mu g \cdot L^{-1}$); Syracuse Environmental Research Associates, Inc. 1999). While these are species not normally found in ditches and canals of drained agricultural fields in the southern United States, it must be remembered that these drains connect to ephemeral streams. At Trice Experimental Forest, the ephemeral streams are approximately 0.5–1 km from the treated stands and the processes of degradation, absorption, and dilution do serve to reduce the sulfometuron methyl concentration that could reach ephemeral streams and their inhabitants.

The field sensitivity of most aquatic invertebrates to sulfometuron methyl has not been established. In this study, we did not detect any negative effects of sulfometuron methyl on invertebrates either in the short term (<2 weeks) or over a somewhat longer term (7 weeks). Even longer term impacts on invertebrates are unlikely to occur because aquatic communities are remarkably resistant to catastrophic disturbance. Gray and Fisher (1981) reported macroinvertebrate recolonization in Sycamore Creek following elimination of 80%–90% of benthic invertebrates. Recolonization proceeded principally by aerial pathways and nearly 66% of all taxa recolonized in 9 weeks. Recolonization can be very rapid, particularly when the affected area is relatively small. It is reasonable to conclude that recolonization rates may be similar or more rapid under considerably lower levels of impact. Such is the case with sulfometuron methyl in the catchments in this study. Sulfometuron methyl 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. The potential for adverse impacts on aquatic-ecosystem functioning in this treated watershed in the long term is low, owing to the low sulfometuron methyl concentrations observed and the documented ability of macroinvertebrates and fishes to rapidly recolonize following catastrophic extirpation. Downstream impacts are also unlikely because of the dilution of sulfometuron methyl, already at low concentrations in the stream flow.

Clearly, it is difficult to assess the potential impacts of sulfometuron methyl on all aquatic organisms from the available toxicological data and the contamination levels we observed. However, no significant impacts on the taxa identified in this study were detected. The results indicate that in forest sites in the South where soil and water conditions are less acidic than the pK_a for sulfometuron methyl and where soil organic-matter content is low, sulfometuron methyl may move off site in stormflow at concentrations that could affect some rooted aquatic macrophytes. The magnitude and duration of exposure are likely to be insufficient to generate significant long-term effects on these sensitive nontarget species.

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