Environmental Effects on Metsulfuron and Chlorsulfuron Bioactivity in Soil[1]

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ABSTRACT

Metsulfuron {2-[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-7l)amino] carbonyl]amino]sulfonyl]benzoic acid}, a sulfonylurea herbicide, is persistent in soil and may injure susceptible crops. To elucidate potential carryover situations, the effect of environment and soil factors on metsulfuron bioactivity was examined in a Platner loam (fine, montmorillonitic, mesic, Aricidic Paleustolls) and a Valent sand (mixed, mesic Ustic Torripsamments) soil with a corn (*Zea mays* L.) root bioassay. Increasing soil temperature from 8 to 24°C in the loam soil reduced the duration of metsulfuron bioactivity by 49%. The temperature effect on metsulfuron bioactivity in the sand was less pronounced, as the 16°C increase resulted in only 20% reduction of duration of bioactivity. The effect of soil water content on metsulfuron bioactivity was soil and temperature related. Increasing soil water increased metsulfuron degradation in the sand at 24°C, but not at 16°C, while in the loam, metsulfuron degradation was not affected by soil water level at either temperature. Incorporating metsulfuron in soil reduced the duration of bioactivity, while the presence of growing wheat (*Triticum aestivum* L.) plants in metsulfuron-treated soil did not affect the duration of bioactivity. Metsulfuron retained on surface straw residue was washed off by simulated rainfall, but duration of metsulfuron bioactivity was increased by this straw residue retention. Chlorsulfuron {2-chloro-*N*-[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl) amino]carbonyl]benzenesulfonamide}, another sulfonylurea herbicide, persisted slightly longer than metsulfuron at the same rate in both soils when incubated at 16°C with identical soil water levels.

Additional Index Words: **herbicide, soil temperature, soil water level, straw interception, incorporation.**

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The sulfonylureas are a new class of herbicides, exhibiting soil activity at extraordinarily low rates of application (Levitt, 1983). Chlorsulfuron {2-chloro-*N*-[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]carbonyl] benzenesulfonamide} was one of the initial sulfonylureas developed, with soil activity persisting for 518 ± 30 d when applied in the Central Great Plains at 35 g ha^{-1} (Anderson and Smika, 1983). Chlorsulfuron selectively controls broadleaf weeds in winter wheat (*Triticum aestivum* L.) and barley (*Hordeum vulgare* L.). Due to its lengthy persistence however, succeeding crops such as corn (*Zea mays* L.), sunflowers (*Helianthus annuus* L.), and safflower (*Carthamus tinctorius* L.), that are susceptible to chlorsulfuron, were injured up to 3 yr after applicataion (Zimdahl and Henson, 1983 and Dyer and Fay, 1984). Metsulfuron {2-[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino] carbonyl]amino]sulfonyl]benzoic acid}, is closely related chemically to chlorsulfuron (Levitt, 1983), and has similar soil activity and persistence (Anonymous, 1983 and Norwood, 1983). Metsulfuron also controls broadleaf weeds in small grains.

Most research on the persistence of sulfonylureas in soils has been conducted with chlorsulfuron. Anderson and Barrett (1985) reported that soil temperature, pH, and texture greatly influenced chlorsulfuron breakdown. Incorporation of chlorsulfuron by physical mixing also resulted in a greater loss of chlorsulfuron activity. Joshi et al. (1984) reported that in acidic soils, chemical hydrolysis and microbial degradation contributed to chlorsulfuron dissipation, whereas in basic soils chemical hydrolysis was less. Both chemical hydrolysis and microbial degradation slowed markedly at low temperatures. Mersie and Foy (1984) found that soil type greatly influenced chlorsulfuron activity. Over 8 μg kg^{-1} of chlorsulfuron was required to inhibit corn root growth 50% in a soil with a cation exchange capacity (CEC) of 330 mmol (+) kg^{-1}, while the 50% growth inhibition (GI) concentration for a soil with a CEC of 30 mmol (+) kg^{-1} was only 0.4 μg kg^{-1}. They also reported that the correlation between 50% GI and CEC was positive, while a negative correlation occurred between 50% GI and soil pH.

The objective of this study was to determine potential carryover situations by examining the effect of soil factors on duration of metsulfuron bioactivity and to compare metsulfuron with chlorsulfuron for duration of bioactivity. The soil factors tested included soil temperature, soil water content, and organic matter level. Method of incorporation, wheat straw interception, and wheat metabolism of metsulfuron were also investigated.

MATERIALS AND METHODS

General Procedures

A corn root bioassay was used to examine metsulfuron and chlorsulfuron availability in soil. Anderson and Barrett (1985) reported that this technique was capable of detecting 0.2 μg kg^{-1} of chlorsulfuron in soil. Two soils of different textures, a Platner loam (fine, montmorillonitic, mesic Aridic Paleustolls) and a Valent sand (mixed, mesic Ustic Torripsamments) were used (Table 1). Each soil was treated with 5 μg kg^{-1} of metsulfuron or chlorsulfuron by adding the herbicide suspended in 6 mL of distilled water to 300 g of air-dried soil. The herbicide was thoroughly incorporated in the soil with a twin shell soil blender (Patterson-Kelly Co., East Stroudsburg, PA). The soil was then placed in 9-cm diam by 9-cm deep plastic pots (without drainage holes). Bioassay tests indicated that the herbicides were thoroughly incorporated throughout the soil.

Corn seeds were pregerminated at 24°C for 48 h for the bioassay. Three seedlings with radicles 1 to 5 mm long were planted 5 to 10 mm deep in each pot. Soil water level in each pot was maintained at 0.20 kg kg^{-1} (67 mPa) for the loam soil and 0.08 kg kg^{-1} (67 mPa) for the sand soil, and the pots were covered with aluminum foil to reduce evaporation. After incubation for 96 h at 24°C, the corn seedlings were removed from the soil and the longest root of each corn seedling was measured to the nearest millimeter. After the corn seedlings were removed, the soil was remixed and returned to the same pot, then replanted in future bioassays. Bioassays were conducted every 3 weeks until the growth of corn roots in metsulfuron- or chlorsulfuron-treated soil did not differ from that in nontreated soil. Each treatment conducted in all studies had five replications, with a control treatment of herbicide-free soil used as the basis for determining percent growth inhibition. Analyses of variance of the data were performed according to techniques described by Steel and Torrie (1960).

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Table 1. Characteristics of the two soils used for the metsulfuron bioactivity studies.

Soil	pH†	Organic matter	Sand	Silt	Clay	Field capacity‡	Cation exchange capacity
		kg kg^{-1}					mmol (+) kg^{-1}
Loam	6.9	0.019	0.34	0.47	0.19	0.23	231
Sand	7.1	0.007	0.88	0.07	0.05	0.10	68

† Soil pH was determined by the saturated paste method.
‡ Field capacity is the soil water level at 0.03 MPa.

Soil Temperature and Soil Water Content Study

Three soil temperatures, 8, 16, and 24°C, were imposed on metsulfuron-treated soil (at 5 μg kg^{-1}) in constant temperature chambers. The soil water contents at all temperatures for the loam soil were 0.15 (100 mPa) and 0.20 kg kg^{-1}, and for the sand soil, 0.06 (100 mPa) and 0.10 (33 mPa) kg kg^{-1}. A factorial experimental design was used. To maintain the desired water contents between bioassays, the water content was adjusted to treatment level after the bioassay and each pot was covered with aluminum foil to reduce soil water evaporation. The 0.10 kg kg^{-1} pots of the sand were allowed to dry for 12 to 24 h immediately prior to planting, then adjusted to 0.08 kg kg^{-1} for the bioassay incubation period. After harvest, the 0.06 kg kg^{-1} sand pots and the 0.15 kg kg^{-1} loam pots were allowed to dry for 12 to 24 h, then adjusted to the desired water level. The pots were then covered with foil and returned to the constant temperature chamber. The control treatment was herbicide-free soil incubated at each temperature, with the soil water level maintained at 0.20 kg kg^{-1} for the soil and 0.08 kg kg^{-1} for the sand. Chlorsulfuron-treated soil (at 5 μg kg^{-1}) of both soils was incubated at 16°C, with the same soil water levels for each soil as described above, giving a comparison of duration of bioactivity for metsulfuron and chlorsulfuron.

Wheat Residue Study

This experiment examined the effect of incorporating wheat residue (at 0.05 Mg kg^{-1}) in both soils on metsulfuron persistence. Wheat residue was air-dried, ground through a 0.85 mm-mesh screen, and thoroughly mixed with 300 g of soil in a plastic bag. Metsulfuron was added in 6 mL of distilled water to give the desired concentration (5 μg kg^{-1}) in the soil, then the soil was thoroughly mixed in the twin shell soil blender. Water levels were adjusted for the bioassays as described previously. Two soil water levels were maintained, 0.15 and 0.25 kg kg^{-1} (33 mPa) for the soil, and 0.06 and 0.10 kg kg^{-1} for the sand. The treated soils and controls were incubated at 24°C. Bioassays were conducted at 3-week intervals until metsulfuron activity was not detected by the bioassay. The data was analyzed as a factorial experiment.

Method of Application

Metsulfuron (5 μg kg^{-1}) was applied by two methods to the loam. To simulate a surface application, metsulfuron was mixed with the upper 3 mm of soil, resulting in 30 g of treated soil, whereas in the total incorporation method, the entire 300 g of soil in each pot was treated with metsulfuron. The soil was not remixed after each bioassay, as was done in the other studies. Also, surface-applied metsulfuron was incorporated 6 and 12 weeks after application, to determine if length of the incorporation period influenced the duration of bioactivity. All treatments were incubated at 24°C with a soil water level of 0.15 kg kg^{-1}. Bioassays were conducted at 3-week intervals until metsulfuron activity was not detected by the bioassay.

Table 3. Comparison of metsulfuron and chlorsulfuron bioactivity in two soils maintained at two soil water levels.†

	Loam soil, 252 d after application				Sand soil, 231 d after application		
	Soil water level kg kg^{-1}				Soil water level kg kg^{-1}		
Herbicide	0.15	0.20	Mean	Herbicide	0.06	0.10	Mean
	% growth inhibition				% growth inhibition		
Metsulfuron	6	4	5	Metsulfuron	6	5	6
Chlorsulfuron	11	38	25	Chlorsulfuron	28	29	29
Mean	9	21		Mean	17	17	
LSD (0.05) for soil water by herbicide interaction = 7				No significant soil water by herbicide interaction.			
LSD (0.05) for main treatments = 5				LSD (0.05) for main treatments = 2			

† Incubation temperature of the soil was 16°C.

Table 2. Length of time required at three soil temperatures before inhibition of corn root growth by metsulfuron was < 20%.†

Soil temperature	Soil: Loam	Soil: Sand
°C	d	
8	267	235
16	231	220
24	136	187
LSD (0.05)	7	4

† Soil water level was 0.15 kg kg^{-1} for the loam soil and 0.06 kg kg^{-1} for the sand soil.

Wheat Metabolism Study

Winter wheat seedlings were grown in metsulfuron-treated loam soil (at 5 μg kg^{-1}) in a glasshouse (average daily temperature of 25 ± 3°C) to determine if metabolism of metsulfuron by wheat seedlings affected the duration of metsulfuron bioactivity. Six wheat seeds were planted in each pot, thinned to four plants per pot after emergence, then removed after 1, 2, or 3 weeks of plant growth. During the glasshouse phase of the experiment, soil water level was maintained at 0.25 kg kg^{-1} by daily watering. After wheat plants were removed, pots were transferred to the 24°C constant temperature chambers with soil water level maintained at 0.20 kg kg^{-1} by use of aluminum foil covers. Bioassays were conducted every 3 weeks until metsulfuron activity was not detected by the corn seedlings.

Straw Interception Study

Metsulfuron at 5 μg kg^{-1} in 6 mL of water was applied with a pipette to three levels of wheat straw on the surface of the loam soil to determine if wheat straw absorbed metsulfuron, thus reducing its bioactivity in the soil. The three straw levels were 0, 1, and 2 g of straw per pot, representing 0, 1680, and 3360 kg ha^{-1}. The 1680 kg ha^{-1} treatment represents the average straw cover on the soil surface after wheat harvest in the Central Great Plains (Greb et al., 1967). After herbicide application, 1 gm of straw was placed on the 0 straw level pots. The pots were watered twice a week to maintain a 0.25 kg kg^{-1} soil water level. Water was applied in two ways to simulate two levels of rainfall, the entire volume passing through the straw, or one half being applied after the straw was removed from the surface, and the remaining half applied over the straw after it was returned to the soil surface. The pots were incubated at 24°C. Bioassays were conducted every 3 weeks until metsulfuron activity was not detected by the bioassay.

RESULTS AND DISCUSSION

Effect of Soil Temperature and Soil Water Level

Increasing soil temperature significantly reduced the duration of metsulfuron bioactivity in both soils (Table 2). With the loam, corn root growth inhibition by metsulfuron incubated at 24°C was less than 20% in 136 d, while metsulfuron bioactivity at 8°C lasted for 267 d, twice as long as the 24°C treatment. A similar but less pronounced temperature effect was found with the sand, indicating that temperature effects on metsulfuron availability to corn roots was influenced by soil type.

Table 4. Effect of wheat residue and soil water level on metsulfuron bioactivity in two soils incubated at 24°C. Data expressed as number of days required before inhibition of corn root growth was less than 20%.

Loam soil				Sand soil			
	Wheat residue kg kg^{-1}				Wheat residue kg kg^{-1}		
Soil water level	0.00	0.05	Mean	Soil water level	0.00	0.05	Mean
kg kg^{-1}	—— d ——			kg kg^{-1}	—— d ——		
0.15	127	115	121	0.06	157	140	149
0.25	131	106	119	0.10	114	92	103
Mean	129	111		Mean	136	116	

LSD (0.05) for soil water by wheat residue interaction = 5
LSD (0.05) for main treatments = 3

No significant soil water by wheat residue interaction.
LSD (0.05) for main treatments = 8

Table 5. Effect of incorporation and time of incorporation after application on metsulfuron bioactivity in the loam soil incubated at 24°C. Soil water content was maintained at 0.15 kg kg^{-1}.

Method of application	Time of incorporation after application	Days after application 126	Days after application 168
	weeks	% growth inhibition	
Surface	--	54	22
Incorporation	0	34	8
Incorporation	6	34	9
Incorporation	12	41	0
LSD (0.05)		12	12

A comparison of metsulfuron and chlorsulfuron bioactivity indicated that chlorsulfuron persisted longer than metsulfuron in both soils (Table 3). However, a significant soil water by chlorsulfuron bioactivity interaction occurred in the loam. Increasing soil water reduced chlorsulfuron degradation, as 38% GI occurred 252 d after application at the 0.20 kg kg^{-1} soil water level, compared to 11% GI at 0.15 kg kg^{-1} soil water level. This interaction did not occur with the sand where soil water level did not affect bioactivity of either herbicide. The higher soil water content in the loam soil may have resulted in anaerobic microsites, possibly reducing chlorsulfuron breakdown. These results indicate that water effects on chlorsulfuron bioactivity may vary with soil type. This was previously reported by Anderson and Barrett (1985).

Wheat Residue Study

Adding wheat residue affected metsulfuron bioactivity differently in the two soils (Table 4). In the loam soil, a significant wheat residue by soil water content interaction occurred, as adding wheat residue and increasing soil water reduced the duration of metsulfuron bioactivity. Increasing the soil water level without adding wheat residue produced the opposite effect, duration of bioactivity being increased. This interaction did not occur in the sand, where either increasing soil water or adding wheat residue significantly decreased metsulfuron persistence. Soil pH greatly affects chemical hydrolysis of chlorsulfuron, with increased acidity decreasing persistence (Joshi et al., 1984). For the loam, addition of wheat residue at the high soil water level changed the pH from 6.9 to 5.8. This may have increased metsulfuron breakdown. This pH shift did not occur with the sand soil. Comparing the water effect alone in the two soils, increasing soil water affected metsulfuron persistence more in the sand soil, where a significant decrease in duration of bioactivity occurred (Table 4). This effect appeared to be temperature related, as soil water levels did not affect metsulfuron degradation in the sand soil at 16°C in the previous study (Table 3).

Method of Application Study

Incorporating metsulfuron in the loam soil significantly reduced its persistence compared to surface application (Fig. 1), which was also reported for chlorsulfuron (Anderson and Barrett, 1985). This decreased bioactivity was not affected by length of the incorporation period as incorporating metsulfuron at 0, 6, or 12 weeks after application did not significantly alter metsulfuron bioactivity (Table 5). These data indicate that the incorporation effect resulted from a reduction of metsulfuron concentration in the planting zone of the soil by dilution. From a management perspective, incorporating metsulfuron might reduce its potential for carryover damage.

Wheat Metabolism Study

Sweetser et al. (1982) reported that winter wheat metabolizes chlorsulfuron and Caseley (1982) found that the presence of growing wheat in chlorsulfuron-treated soil reduced its persistence. If wheat-metsulfuron interactions are the same as for wheat-chlorsulfuron and metsulfuron was applied for residual weed control where volunteer winter wheat was growing, metabolism by winter wheat could reduce the persistence of metsulfuron. In this study, the presence of winter wheat did not reduce metsulfuron persistence (Fig. 2). Within the growing wheat treatments, the duration of bioactivity decreased with length of wheat growth, but the presence of growing wheat did not

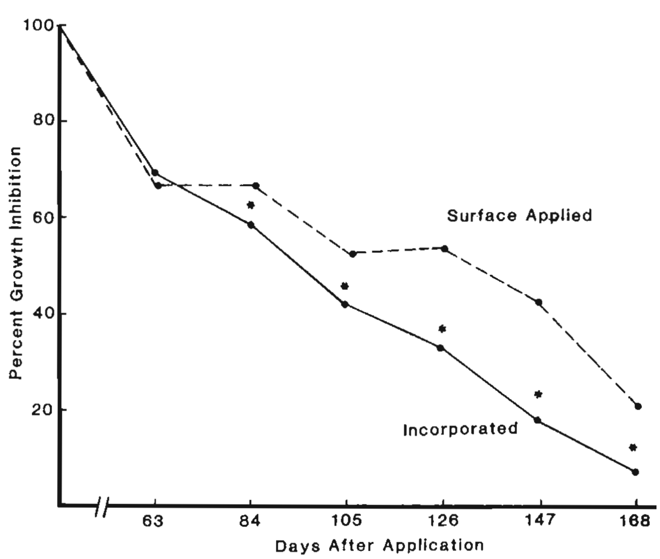

Fig. 1. Effect of method of application on metsulfuron persistence in a loam soil. Asterisk indicates that the incorporated treatment was significantly different at the 0.05 level on that day of bioassay.

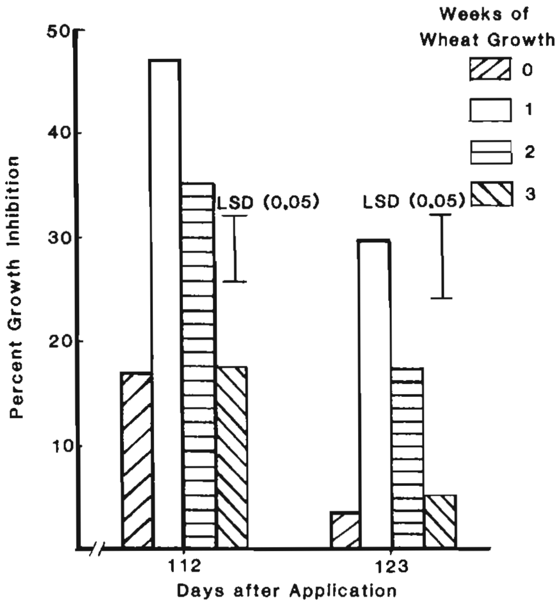

Fig. 2. Effect of wheat metabolism on metsulfuron bioactivity in the loam soil. Incubation temperature was 24°C, and soil water level was 0.20 kg kg^{-1}.

reduce the duration of bioactivity when compared to the control (0 treatment). Growing wheat may produce compounds allelopathic to corn root growth, thus, confounding the metabolic effect resulting from the different periods of wheat growth. However, over the period of this study, the presence of growing wheat did not reduce metsulfuron persistence.

Straw Interception Study

When applied in field situations, herbicides can be retained on plant material covering the soil surface. Ghadiri et al. (1984) reported that 60% of atrazine (6-chloro-*N*-ethyl-*N'*-(1-methylethyl)-1,3,5-triazine-2,4-diamine) applied to the soil surface was retained on wheat stubble, but that rainfall following application would wash the atrazine off the stubble and deposit it on the soil surface. Banks and Robinson (1984) found that over 50% of oryzalin(4-(dipropylamino)-3,5-dinitrobenzenesulfonamide) that was applied to the soil surface was intercepted by wheat straw, but rainfall did n not wash oryzalin off the stubble. Oryzalin is less water soluble than atrazine which might explain why oryzalin

Table 6. The interaction of straw level and water treatment on metsulfuron bioactivity in a loam soil.

	Water treatment		
Straw level	A†	B	Mean
kg ha^{-1}	% growth inhibition		
1680	16‡	40	28
3360	38	42	40
Mean	27	41	

LSD (0.05) for the significant water × straw interaction = 6
LSD (0.05) for main effects = 4

† Water was applied twice weekly; A = water applied over the straw; B = 1/2 of water applied to the soil surface, 1/2 through the straw.
‡ Bioassay was conducted 168 d after application.

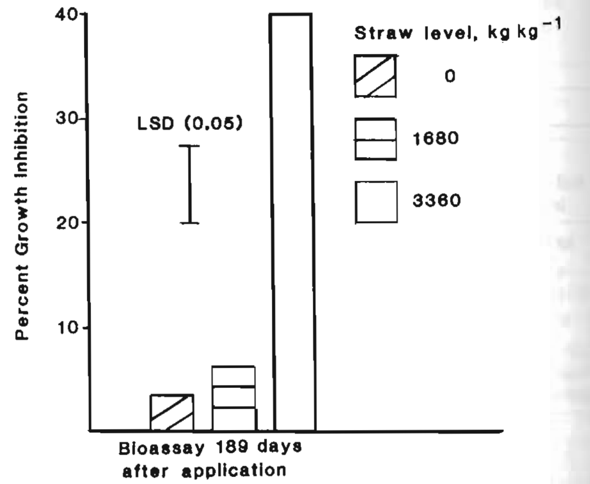

Fig. 3. The effect of straw mulch on metsulfuron bioactivity in a loam soil.

remained attached to the straw. In our study, greater metsulfuron bioactivity 189 d after application was noted in the treatments containing the higher straw treatments (Fig. 3). This might be caused by temporary metsulfuron retention on the straw which would have been washed off the straw with the later simulated-rainfall treatments. Metsulfuron temporarily retained on the straw would have been unavailable for early degradation, thus increasing its persistence compared to metsulfuron applied to bare soil. When watering intensity treatments were imposed on the straw treatments (Table 6), a significant interaction resulted, indicating that the rainfall intensity needed to wash metsulfuron off the straw was influenced by the level of straw present.

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Herbicide Residues from Winter Wheat Plots: Effect of Tillage and Crop Management[1]

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ABSTRACT

Data on the magnitude and persistence of residues of metribuzin (4-amino-6-*t*-butyl-3-(methylthio-1,2,4-triazine-5-(4H)-one and bromoxynil octanoate (2,6-dibromo-4-cyanophenyl octanoate) in soils and runoff water from winter wheat (*Triticum aestivum* L.) fields were collected on plots managed with different tillage and crop rotation systems during three seasons from 1978 to 1982. On steeply-sloped runoff plots, the greatest runoff, erosion, and losses of herbicides occurred on plots that were planted in winter wheat after summer fallow and were tilled conventionally. The lowest erosion and herbicide losses were observed on plots directly seeded in winter wheat stubble without tillage, whereas the relative effectiveness of the other systems depended on the year, the volume of runoff, and the type of runoff events. Wheat residue from the previous crop was more effective than pea (*Pisum sativum* L.) residue in reducing erosion and herbicide losses, mainly because of the greater quantities of wheat residues produced. Compared to runoff of herbicides under summer conditions in the southeastern USA, runoff of herbicides under winter conditions in Eastern Washington was greatly extended, and there was very little movement or degradation of the herbicides while the ground was frozen. The half-life of metribuzin in soil was 102 to 112 d (k = 0.0069 − 0.0062 d^{-1}), but insufficient data were obtained to calculate the half-life of bromoxynil octanoate. The disappearance of bromoxynil octanoate and bromoxynil (2,6-dibromo-4-cyanophenol) from soil plots was rapid; only traces of bromoxynil octanoate or bromoxynil were present after 72 d under winter conditions. Small amounts of metribuzin and desaminodiketo metribuzin (DADK) moved down as far as 21 cm in the soil. Less than 0.060 μmol kg^{-1} of metribuzin and less than 0.015 μmol kg^{-1} DADK remained in the top 7 cm of soil 192 d after application of 0.43 to 0.56 kg ha^{-1} of metribuzin.

Additional Index Words: **soil erosion, high-performance liquid chromatography, no-tillage, soil conservation, herbicide in runoff water.**

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Wheat (*Triticum aestivum* L.) and other small grain production in the intensively farmed Palouse region of the Pacific Northwest depends heavily on herbicide and fertilizer use. This region is characterized by steeply sloped hillsides, extended periods of winter precipitation and runoff from saturated and partially thawed soils. As a result, the region suffers winter runoff and some of the highest erosion rates from croplands in the USA. In conventional management of winter wheat, most of the herbicides are applied to the fields during the active growing season, i.e., April through June, when there is little or no runoff. However, fall application of herbicides is increasing or likely to become more prevalent with the shift to reduced tillage or no-till management systems, which markedly reduces erosion but not necessarily runoff.

Considerable information has accumulated over the past decade on pesticide losses in runoff water from croplands and on factors affecting carry off of pesticides in runoff water under summer conditions. Most of the studies indicate that, for the majority of commercial pesticides, total losses are 0.5% or less of the amounts applied unless heavy rainfall occurs within 1 to 2 weeks after application (Wauchope, 1978). Losses may also be greater with the more persistent materials or with wettable powder formulations of herbicides because powders are easily washed off of the soil surface (Wauchope, 1978). These findings are based on data from the central and southeastern states where pesticide usage and runoff amounts are both relatively high (Wauchope, 1978), and where the chemicals are applied during warm growing conditions. Low soil temperatures, as would be the case for fall- or winter-applied materials, are known to increase the persistence of many herbicides in soil (Hyzak and Zimdahl, 1974; Hormann et al., 1979) and could lead to increased runoff losses of these chemicals in comparison to warm weather applications.

Metribuzin (4-amino-6-*t*-butyl-3-(methylthio-1,2,4-triazine-5(4H)-one) and bromoxynil octanoate (2,6-dibromo-4-cyanophenyl octanoate) are two herbicides used for weed control in wheat and are potentially useful as late fall pre- and postplant herbicides for winter wheat lands in reduced tillage management systems. Both chemicals have relatively short half-lives under spring and summer conditions (Stewart et al., 1975), but little is known about their persistence and

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Names of companies or commercial products are given solely for the purpose of providing specific information; their mention does not imply recommendation or endorsement by the U.S. Department of Agriculture over others not mentioned.

[2] Research chemist, Yakima; agricultural engineer, Pullman; supervisory soil scientist, Pullman; and supervisory research chemist, Yakima, respectively.