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Effect of Temperature and Moisture on Aminocyclopyrachlor Soil Half-Life

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Aminocyclopyrachlor will control a variety of invasive weeds but may injure sensitive plant species if seeded into treated soil too soon after application. Aminocyclopyrachlor 50% dissipation time (DT_{50}) ranged from 3 to > 112 d in four soils from the Northern Great Plains. The DT_{50} was dependent on several factors including soil type, moisture content, and temperature. Across four different soil textures, aminocyclopyrachlor dissipation generally increased as soil moisture content increased, but moisture had less of an impact in sandy soils. Aminocyclopyrachlor dissipation also increased as temperature increased in the four soils. The most rapid dissipation occurred in soils with higher clay content, which also had the highest organic matter content of the soils evaluated, and an average DT_{50} of less than 20 d. Seeding sensitive pasture, range, or crop species after aminocyclopyrachlor applications should be done with caution since the herbicide has potential for long persistence in the soil.

Nomenclature: Aminocyclopyrachlor
Key words: Dissipation, invasive species, soil residual.

Aminocyclopyrachlor controlará varias malezas invasivas, pero podría dañar especies de plantas sensibles si estas son sembradas en suelo tratado poco tiempo después de la aplicación. El tiempo de disipación del 50% de aminocyclopyrachlor (DT_{50}) varió entre 3 y >112 días en cuatro suelos de las Grandes Planicies del Norte. El DT_{50} fue dependiente de varios factores incluyendo tipo de suelo, contenido de humedad, y temperatura. En las cuatro diferentes texturas de suelo, la disipación de aminocyclopyrachlor generalmente aumentó al incrementar el contenido de humedad del suelo, pero la humedad tuvo un impacto menor en suelos arenosos. La disipación de aminocyclopyrachlor también aumentó al incrementarse la temperatura en los cuatro suelos. La disipación más rápida, con un DT_{50} promedio de menos de 20 días, ocurrió en suelos con mayor contenido de arcilla, los cuales también tuvieron los mayores contenidos de materia orgánica en los suelos evaluados. La siembra de pastos, especies silvestres, o cultivos sensibles después de aplicaciones de aminocyclopyrachlor deberían realizarse con precaución en vista de que este herbicida tiene potencial de una larga persistencia en el suelo.

Aminocyclopyrachlor is the first herbicide classified as a pyrimidinecarboxylic acid (Claus et al. 2010) and has both foliar and soil activity (Lindenmayer et al. 2010; Westra et al. 2010). Aminocyclopyrachlor has been evaluated for control of invasive weed species. The herbicide is structurally similar to the pyridinecarboxylic acid herbicides aminopyralid and picloram, with the exception of a cyclopropyl side chain and a pyrimidine rather than a pyridine ring (Bukun et al. 2010). Chemically the compounds have different properties. Aminocyclopyrachlor has a water solubility of 4.2 g L^{-1} (Anonymous 2009; Finkelstein et al. 2008), which is approximately 10 times more water soluble than picloram (Senseman 2007b), and nearly twice the water solubility of aminopyralid (2.5 g L^{-1}) (Senseman 2007a). Additionally, aminocyclopyrachlor has the highest soil-binding potential, with a sorption coefficient (K_{oc}) of 28 (Anonymous 2009; Finkelstein et al. 2008) compared with 10.8 and 16 for aminopyralid and picloram, respectively (Senseman 2007a,b).

Extended soil activity of aminocyclopyrachlor could be important for long-term perennial weed control (Lindenmayer et al. 2011). Aminocyclopyrachlor applied to the soil severely limited Canada thistle (*Cirsium arvense* L.) regrowth from roots and provided moderate control of rush skeletonweed (*Chondrilla juncea* L.), which confirmed that aminocyclopyrachlor had soil activity (Lindenmayer et al. 2010; Stevens and Burke 2009). When Canada thistle roots were planted above a layer of aminocyclopyrachlor-treated soil the resultant biomass was much lower compared with when planted below a treated layer of soil (Lindenmayer et al. 2011).

Aminocyclopyrachlor can remain active in the soil for at least 2 yr (Westra et al. 2008a), and the half-life is likely dependent on environment (EPA 2010). Aminocyclopyrachlor is degraded in the soil by microbes (Anonymous 2009; Finkelstein et al. 2008) and by photolysis when deposited on the soil surface (EPA 2010). Aqueous photolysis was much faster than on soil, 1 to 8 d vs. 129 d. In turf studies, the half-life ranged from 37 to 103 d, whereas in bare soil, the half-life was longer and ranged from 80 to 164 d (Anonymous 2009; Finkelstein et al. 2008). The herbicide was stable in anaerobic aquatic and anaerobic terrestrial environments with a half-life of 1,733 and 6,932 d, respectively.

High grain prices may influence land managers to convert noncropland or land enrolled in the conservation reserve program (CRP) to cropland (Hellerstein and Malcolm 2011). Conservation reserve program land could have been treated with residual herbicides to control invasive weeds as required by law throughout enrollment, so crops planted into previous CRP land may be sensitive to herbicides

Agronomic crops sensitive to aminocyclopyrachlor include alfalfa (*Medicago sativa* L.) (Westra et al. 2008b), spring and winter wheat (*Triticum aestivum* L.) (Kniss and Lyon 2011),

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Table 1. Physical and chemical characteristics of four soils from the Northern Great Plains included in the soil dissipation experiment.

Location	Soil series	Sand	Silt	Clay	Organic matter	Field capacity gravimetric water content	pH
		% by wt					
Fargo	Fargo	5	45	50	7.0	55	7.2
Jamestown	Svea-Barnes	37	42	21	6.4	51	5.7
Medora	Glendive-Havre	5	35	60	1.2	38	8.1
Walcott	Lamoure	86	9	5	2.6	49	7.8

soybean [*Glycine max* (L.) Merr.], (Westra et al. 2008b), and sunflower (*Helianthus annuus* L.). Winter wheat planted in soil previously treated with aminocyclopyrachlor resulted in yield losses, although visual estimates of injury were low (Kniss and Lyon 2011). Yield reductions of 50% were measured when the soil was treated 2, 4, and 6 mo before planting with aminocyclopyrachlor at 20, 25, and 61 g ha^{-1}, respectively. Losses were likely due to low seed production as the result of auxin accumulation in the ovary initiating fruit development before fertilization (Gillaspy et al. 1993; Kniss and Lyon 2011).

Knowing the dissipation rate of aminocyclopyrachlor is important if treated CRP land is returned to crops or if pasture and rangeland is reseeded following a weed control program using this herbicide. The objective of this research was to evaluate the effect of temperature and moisture on soil dissipation of aminocyclopyrachlor in soils found in the Northern Great Plains. Once determined, the time required for cropping in previously treated areas can be more accurately estimated.

Materials and Methods

The effect of moisture and temperature on the dissipation of aminocyclopyrachlor was evaluated on four soils found in the Northern Great Plains using a soybean bioassay. Fargo silty clay (Fine, smectitic, frigid Typic Epiaquerts) (Soil Survey Staff 2011), Svea-Barnes loam (Fine-loamy, mixed, superactive, frigid Pachic Hapludolls; Fine-loamy, mixed superactive, frigid Calcic Hapludolls), Glendive-Havre clay (Coarse-loamy, mixed, superactive, calcareous, frigid Aridic Ustifluvents; Fine-loamy, mixed, superactive, calcareous, frigid Aridic Ustifluvents), and Lamoure loamy sand (Fine-silty, mixed, superactive, calcareous, frigid Cumulic Endoaquolls) soils were collected near Fargo, Jamestown, Medora, and Walcott, North Dakota, respectively (Table 1). Soil was obtained from 0- to 15-cm depth, screened through a 6-mm sieve, air dried for 5 d, and stored at 22 C until needed (approximately 1 to 2 mo). The field capacity (FC) was determined by weight for each soil type in a preliminary study (Conklin 2012).

Moisture Study. Aminocyclopyrachlor was applied at 36 μg kg^{-1} in 10 ml of solution to 500 g air-dry soil in wax paper bags and allowed to dry for 24 h. Soil was mixed in the bags by inverting 20 times and poured into individual 10-cm-diam by 8-cm plastic pots with five 0.25-cm-diam holes in the bottom that had been covered with filter paper. Each pot was placed in a separate 13- by 13- by 4-cm-deep tray to collect leachate, and soil water contents were established at 22.5, 45, or 90%. The pots were then placed in dark chambers with a constant temperature of 16 ±2 C for 8 wk. The desired FC was maintained by weighing pots twice weekly and adding water as needed. Upon removal the soil was frozen to reduce or eliminate microbial activity until the bioassay. Soil with water contents of 22.5% and 45% FC was warmed 5 d at 16 ± 2 C. Soil with a water content of 90% FC was allowed to warm 2 d at 16 ± 2 C and 3 d at 21 ± 2 C to reduce water content to approximately 45% FC. The soil in each pot was individually mixed before planting.

Aminocyclopyrachlor concentration remaining in the soil was determined by a soybean bioassay. A standard curve with four replications per treatment of each soil type was prepared with aminocyclopyrachlor concentrations of 0, 4.5, 9, 18, and 36 μg kg^{-1} soil. The soil was air dried, mixed, and placed into plastic pots as previously described. Eight 'Traill' soybean seeds were planted 1 cm into each pot of soil. Soil was moistened by adding water alternately to the surface and subsurface throughout the bioassay as needed to maintain approximately 50% FC. After emergence soybeans were thinned to four per pot and water-soluble fertilizer (Jack's Classic All Purpose Water Soluble Plant Food, Analysis: 20-20-20, J. R. Peters, Inc., 6656 Grant Way, Allentown, PA 18106) was applied at 85 kg nitrogen ha^{-1}. Pots were rotated every 4 d to reduce environmental variability in the greenhouse. The greenhouse was maintained at 21 C, and natural sunlight was supplemented with metal halide lights with an intensity of 450 μmol m^{-2} s^{-1} for a 16-h photoperiod. Soybeans were cut at the soil surface 15 to 17 d after planting and stem height was measured to the apical meristem. Preliminary studies found that the soybean bioassay was sensitive to aminocyclopyrachlor from < 1 to approximately 50 parts per billion.

Temperature Study. Soil was weighed, mixed, and placed in pots as previously described, except water content was maintained at 45% FC for the duration of the temperature study. The soil was stored in dark chambers with temperature held constant at 8 ± 2, 16 ± 2, or 24 ± 2 C. Four pots of each soil type were removed 0, 2, 4, 8, and 16 wk after treatment. Soil was frozen until the soybean bioassay was initiated. Soil was warmed at 16 ± 2 C for 5 d and then placed in the greenhouse and prepared for planting. The moisture and temperature bioassays were conducted at the same time and utilized the same standard curve.

Data Analysis. The moisture and temperature studies were a randomized complete block with four replicates. Soybean stem height was analyzed with SAS (Statistical Analysis Software 2003, version 9.1, SAS Institute, Inc., 100 SAS Campus Drive, Cary, NC 27513) ANOVA using PROC

Table 2. Effect of moisture on aminocyclopyrachlor dissipation to 50% (DT_{50}) in four soils from the Northern Great Plains 56 d after treatment with 36 μg kg^{-1} and held at 16 C.

Moisture content	Soil series			
	Fargo	Glendive-Havre	Lamoure	Svea-Barnes
% Field capacity	DT_{50}			
22.5	> 50[a] a[b]	20 a	44 a	7 a
45	17 b	54 b	30 a	3 a
90	5 c	19 a	21 a	11 a
R^2	99[c]	94	95	97

[a] Actual DT_{50} exceeds the sensitivity of the test; however, means were separated using the estimated value.

[b] Numbers followed by the same letter within each soil are not significantly different according to probability of difference ($P \leq 0.05$).

[c] R^2 values from the equation for the standard curve used in each soil series to estimate herbicide concentration.

GLM. Experiments were repeated and runs were homogeneous and data combined. Regression analysis was used to develop curves on the basis of soybean stem height from standard curve soils.

The time to 50% dissipation (DT_{50}) of aminocyclopyrachlor was calculated individually for each replicate in every soil. The first-order rate Equation 1 was used to describe

$$\ln\left(\frac{A_t}{A_0}\right) = -kt \qquad (1)$$

aminocyclopyrachlor dissipation (Walker 1987). A_t was the concentration of aminocyclopyrachlor in the soil at time t, A_0 was the initial aminocyclopyrachlor soil concentration, and k was the dissipation rate constant. Equation 2 was used to calculate DT_{50}

$$DT_{50} = \frac{0.693}{k} \qquad (2)$$

where k was the rate constant from Equation 1.

When the DT_{50} value for a replicate could not be determined because it was greater than 56 or 112 d for the moisture study or temperature study, respectively, the value was considered missing. When four or more DT_{50} values were missing within one treatment 56 or 112 d were used for the moisture or temperature studies, respectively. The DT_{50} values of aminocyclopyrachlor in each soil were subjected to ANOVA and compared using least-squares means. Treatment means were separated by probability of difference ($P \leq 0.05$). Treatment means calculated with more than four missing DT_{50} values are preceded with > to indicate that actual dissipation could be longer than the mean.

Results and Discussion

Moisture Study. The effect of moisture on aminocyclopyrachlor dissipation was dependent on soil type. In Fargo silty clay soil, aminocyclopyrachlor DT_{50} values decreased from > 50 to 5 d as moisture content increased from 22.5 to 90% FC, respectively (Table 2). Although not significant, the DT_{50} in the Lamoure loamy sand decreased from 44 d at 22.5% FC to 21 d at 90%. Similarly, in sandy loam soil the rate of picloram degradation was influenced by moisture (Meikle et al. 1973). As soil moisture content increased from 18 to 192% of 1/3 bar tension, picloram decomposition increased from 17 to 40% of applied herbicide.

Aminocyclopyrachlor dissipation in Fargo and Lamoure soils would be more rapid during a wet spring or summer than in dry conditions. Long periods of moist soil conditions could reduce the period of weed control in these soils; however, if the treated land is converted into cropland wet conditions could reduce the rotation restriction for planting sensitive crops.

In Glendive-Havre clay soil, aminocyclopyrachlor DT_{50} values averaged 20 d at 22.5 and 90% FC, compared with 54 d when moisture was held at 45% FC (Table 2). The decrease in DT_{50} values from 45 to 90% FC was consistent with the trend in Fargo and Lamoure soils; however, rapid dissipation at 22.5% FC was not expected. Since aminocyclopyrachlor is degraded by microbes in the soil (Anonymous 2009; Finkelstein et al. 2008) and aerobic microbial activity is greatest at intermediate soil moisture of 50 to 60% water-filled pore space (Franzluebbers 1999; Linn and Doran 1984), the DT_{50} value at 22.5% FC was expected to be greater than at 45 or 90%.

Sorption properties of the Glendive-Havre soil could explain why the aminocyclopyrachlor DT_{50} value was higher at 45% than at 22.5% FC (Table 2). The Glendive-Havre soil had a high clay content (60%) and low organic matter (1.2%) (Table 1), which would decrease aminocyclopyrachlor sorption compared with the Fargo and Lamoure soils. A study in Brazil found that aminocyclopyrachlor sorption was correlated with both organic matter and clay content in 14 soils (Oliveira et al. 2011). However, once sorbed aminocyclopyrachlor did not readily desorb and was affected by hysteresis. No significant correlation was found between pH and sorption but the pH of the tested soils was fairly narrow (4.9 to 7.2). Green and Obien (1969) concluded that although soils with weak herbicide adsorption have an increased concentration of herbicide in solution, the herbicide will not always be more phytotoxic in dry conditions. At low soil moisture hydrophilic adsorptive sites become more available to the herbicide, and can result in reduced herbicide toxicity (Grover 1966).

Aminocyclopyrachlor dissipation in the Svea-Barnes loam soil was not affected by moisture and the average DT_{50} was 11 d (Table 2). The rapid decrease in plant-available herbicide observed in this soil could be due to more rapid microbial degradation of aminocyclopyrachlor. The Svea-Barnes soil

Table 3. Effect of temperature on aminocyclopyrachlor dissipation to 50% (DT_{50}) in four soils from the Northern Great Plains 56 d after treatment with 36 $\mu g\ kg^{-1}$ and held at 45% field capacity.

Temperature C	Soil series			
	Fargo	Glendive-Havre	Lamoure	Svea-Barnes
	DT_{50}			
8	37 b[a]	> 112[b] b	> 80 b	> 88 a
16	18 a	54 a	52 b	22 a
24	11 a	51 a	13 a	13 a
R^2	99[c]	94	95	97

[a] Numbers followed by the same letter within each soil are not significantly different according to probability of difference ($P \leq 0.05$).

[b] Actual DT_{50} exceeds the sensitivity of the test; however, means were separated using the estimated value.

[c] R^2 values from the equation for the standard curve used in each soil series to estimate herbicide concentration.

was collected from a field used for corn production and had higher N–P–K values than the other soils in the study.

The most rapid dissipation of aminocyclopyrachlor in Fargo soil will likely occur when the soil moisture content is greater than 45% FC; however, in soils with higher sand content such as the Lamoure and Svea-Barnes soils low moisture did not affect aminocyclopyrachlor dissipation (Table 2). In field dissipation studies submitted to the EPA (2010) for herbicide registration, the half-life of aminocyclopyrachlor ranged from 22 to 126 d, generally higher than the range of 3 to > 50 d reported here (Table 2). The shorter half-life observed in this study could be due to limitations of the bioassay method, which reports only plant-available herbicide.

Temperature Study. Aminocyclopyrachlor DT_{50} values generally decreased as temperature increased regardless of soil type (Table 3). Aminocyclopyrachlor DT_{50} values were lowest in Fargo soil and values decreased from 37 to 11 d as temperature increased from 8 to 24 C. Aminocyclopyrachlor dissipation followed a similar trend in the Glendive-Havre soil, but DT_{50} values were longer, and ranged from 51 to > 112 d. Again, the Glendive-Havre soil had only 1.2% organic matter but a clay content of 69% (Table 1), which likely decreased aminocyclopyrachlor desorption rates and could make the herbicide available for plant uptake for a longer time period. A similar study indicated that aminopyralid dissipation was also more rapid in Fargo-Ryan soil than Glendive-Havre soil, and reasoned that high organic matter content may support greater microbial populations (Mikkelson 2010).

Aminocyclopyrachlor dissipation increased as soil temperature increased, which was similar to the response of other growth-regulator herbicides. Aminopyralid dissipation increased with increased temperature, and was approximately eight times faster at 24 C than 8 C in Lamoure and Svea-Barnes soil (Mikkelson 2010). In this study, the DT_{50} values of aminocyclopyrachlor in Lamoure and Svea-Barnes soil at 8 C were > 80 and > 88 d, respectively, but decreased to 13 d each when temperature was increased to 24 C (Table 3). Similarly, picloram (Guenzi and Beard 1976) and clopyralid (Ahmad et al. 2003) degradation were maximized when soil was incubated at ≥ 30 C and were influenced by microbial activity.

The DT_{50} values in the temperature study ranged from 11 to > 112 d (Table 3). The DT_{50} values that exceeded the sensitivity of the test could be much longer than 112 d, and in a variety of environments (conditions not given) aminocyclopyrachlor has been persistent, with a half-life as long as 373 d (EPA 2010). The potential of aminocyclopyrachlor to persist is a concern since herbicide in the soil or plant material could harm nontarget species (EPA 2011).

In summary, the rate of aminocyclopyrachlor dissipation generally increased as soil moisture content or temperature increased. The results suggest that aminocyclopyrachlor degradation may be slower in soils with low organic matter or high clay content. These data combined with those of Oliveira et al. (2011) suggest that the DT_{50} of this herbicide is complex and not easily predicted by a single soil characteristic. Rather, a combination of clay content, organic matter levels, or other soil parameters will affect aminocyclopyrachlor DT_{50}. Aminocyclopyrachlor persistence in the soil will be beneficial for long-term weed control; however, land managers should be cautious when reseeding pasture and rangeland with sensitive forb species or converting CRP to cropland where aminocyclopyrachlor was applied.

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