# Field Persistence Studies with Emulsifiable Concentrate and Granular Formulations of the Herbicide Pendimethalin in Saskatchewan

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Studies were undertaken to evaluate the dissipation of pendimethalin (1.1 kg $ha^{-1}$) in two Saskatchewan field soils following applications of an emulsifiable concentrate (EC) formulation in the spring. Persistence of pendimethalin applied as a granular formulation in the fall was also investigated at one of the locations. The EC formulation was applied in May, 1993, and by late September, 0.50 ± 0.16 kg $ha^{-1}$ (41 ± 13% of that applied) remained at the Estlin site, and 0.34 ± 0.07 kg $ha^{-1}$ (31 ± 6%) remained at the Regina location. Over the winter, there was little further loss of pendimethalin at either locality. By September, 1994 (479 days after treatment), 0.34 ± 0.07 kg $ha^{-1}$ (28 ± 6% of the applied herbicide) remained at Estlin and 0.13 ± 0.05 kg $ha^{-1}$ (12 ± 4%) remained at Regina. Some overwinter dissipation of pendimethalin occurred from a granular formulation (1.51 kg a.i. $ha^{-1}$) applied at Regina on October 4, 1993, with 1.01 ± 0.15 kg $ha^{-1}$ (67 ± 10% of the initial treatment) remaining in May, 1994. By the third week in September, 1994, 350 days after application, 0.45 ± 0.08 kg $ha^{-1}$ (30 ± 5% of the initial herbicide) remained. Over the 479-day EC formulation and the 350-day granular formulation study periods, there was no leaching of the herbicide below 10 cm.

**Keywords:** *Field studies; formulations; herbicide; leaching; pendimethalin; persistence*

## INTRODUCTION

Pendimethalin (*N*-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine) is a dinitroaniline herbicide used for the selective control of grassy and broadleaf weeds in a variety of crops. The herbicide is used at rates of 0.56 to 3.36 kg $ha^{-1}$ in both emulsifiable concentrate (EC) and granular formulations (WSSA, 1994).

Pendimethalin, like other dinitroaniline herbicides, is water insoluble (0.27 $\mu g\ g^{-1}$ at 25 °C) is relatively volatile (vapor pressure $9.4 \times 10^{-6}$ mmHg at 25 °C) and is strongly adsorbed to soil colloids (Zheng et al., 1993; WSSA, 1994). The herbicide is not significantly leached in soil columns (Zheng et al., 1993), nor is leaching considered to occur under field conditions (WSSA, 1994).

In soil, pendimethalin is degraded by both chemical reactions and biological processes (Parochetti and Dec, 1978; Savage and Jordan, 1980; Barua et al., 1990; Kulshrestha and Singh, 1992). Losses through volatility may also occur (WSSA, 1994). The breakdown of pendimethalin in soil is both temperature and moisture dependent (Walker and Bond, 1977; Barrett and Lavy, 1983; Zimdahl et al., 1984; Kulshrestha and Singh, 1992). As with other dinitroaniline herbicides, degradation proceeds more rapidly under flooded anaerobic conditions than under aerobic conditions (Savage, 1978; Barrett and Lavy, 1983; Kulshrestha and Singh, 1992). Field persistence studies have indicated that the herbicide is persistent, with residues being carried over to the next crop year (Walker and Bond, 1977; Barrett and Lavy, 1983; Zimdahl et al., 1984).

Pendimethalin is currently being considered for registration in western Canada, where little is known regarding its field persistence under extreme prairie weather conditions. We evaluated the dissipation of pendimethalin in two Saskatchewan field soils, at Estlin and Regina, following spring applications of an emulsifiable concentrate (EC) formulation. In addition, the persistence of pendimethalin applied as a granular formulation in the fall was investigated at the Regina location.

## MATERIALS AND METHODS

**Chemicals.** Analytical samples of pendimethalin were provided by Cyanamid Canada Inc., Markham, Ontario, Canada. The commercial EC formulation PROWL 400, containing 400 g $L^{-1}$ pendimethalin, and a granular formulation containing 6.25% pendimethalin were used for field applications.

**Soils.** The two study locations were at Estlin and Regina on Dark Brown Chernozem clays. The physical characteristics of the soils were determined by the Plains Innovative Laboratory Services, Saskatoon, Saskatchewan, Canada, and are summarized in Table 1.

**Site Preparation.** The study area at Estlin had been sown to spring wheat in 1991 with difenzoquat (1,2-dimethyl-3,5-diphenyl-1*H*-pyrazolium methyl sulfate) at the recommended rate of 0.40 kg $ha^{-1}$ for weed control. During 1992, the site was in stubble fallow. The experimental location was disced the first week of May 1993 to a depth of 7.5 cm.

At Regina, the study area had been seeded to spring wheat in 1991 and 1992, with no herbicide applications either year. During the 3rd week of May, 1993, the area was disked to a depth of 7.5 cm.

Fall application of the granular pendimethalin formulation was made to wheat stubble at the Regina location adjacent to the EC formulation study. The site had been seeded to spring wheat in 1991, 1992, and 1993.

**Spring Herbicide Application.** At both sites, an area 12 × 100 m was marked out, and the herbicide was applied at a rate of 1.1 kg $ha^{-1}$ with a conventional sprayer, with a 12-m boom that had 24 80° Tee Jet XR8002 nozzles set 45 cm above the soil surface. The tank contained 1 L of the EC formulation

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0021-8561/95/1443-2988$09.00/0 Published 1995 by the American Chemical Society

**Table 1. Characteristics of Soils Used in the Study**

| soil type | soil depth (cm) | clay (%) | silt (%) | sand (%) | org matter (%) | pH | CEC (mequiv/100 g) | cond (mS/cm) | moisture 1/3 bar (%) |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | Estlin | | | | |
| clay | 0–10 | 68 | 30 | 2 | 3.9 | 6.7 | 33 | 2.7 | 70 |
| clay | 10–20 | 71 | 28 | 1 | 3.4 | 6.9 | 33 | 2.6 | 71 |
| | | | | | Regina | | | | |
| clay | 0–10 | 63 | 33 | 4 | 3.4 | 7.3 | 29 | 2.6 | 61 |
| clay | 10–20 | 65 | 32 | 3 | 3.3 | 7.3 | 30 | 2.6 | 63 |

(400 g of pendimethalin) in 36.4 L water, and the volume sprayed was equivalent to 100 L $ha^{-1}$. Spray pressure was 275 kPa, which delivered 770 mL of spray $min^{-1}$ $nozzle^{-1}$. To achieve the required application rate, a tractor speed of 9.2 km $h^{-1}$ was maintained. On May 28, 1993, pendimethalin was applied to the surface of the weed-free plots at both sites. Immediately following application, the herbicide was incorporated into the soil by discing twice, at right angles, to a depth of 7.5 cm. The centers of the sprayed areas were divided into three plots (5 × 25 m) with a 5-m strip between each plot.

**Fall Herbicide Application.** The granular formulation was applied at the Regina location with a Valmar 1220 Airflo Granular Applicator with a 5.03-m boom fitted with 12 outlets. The boom was set at a height of 50 cm above the field surface. Two areas (5.03 × 100 m) were treated, the applicator being calibrated according to the manufacturer's specifications to deliver 1.4 kg $ha^{-1}$. The actual treatment rate, as determined by weighing the granules in the hopper before and after application, was 1.51 kg $ha^{-1}$. After treatment, the granules were incorporated into the soil to a depth of 5 cm, in the same direction as application, by a single pass with a duck-foot cultivator. The center of each treated strip was divided into two plots (3.5 × 40 m) with a 5-m strip between plots. Fall treatment was made on October 4, 1993. A second incorporation, at right angles to the direction of application and to a depth of 5 cm, was made using a duck-foot cultivator on May 6, 1994, 214 days after treatment.

**Soil Sampling from Spring Treatments.** Pre-application check plots adjacent to the treated plots were sampled on May 19 (Estlin) and May 20 (Regina) by taking eight random cores (7.62 cm i.d. and 10 cm deep; total surface area 365 $cm^2$). Samples were also taken from the 10–20-cm depth. All samples from each plot were pooled in paper bags.

Immediately following herbicide application, two sets of eight cores (7.62 × 10 cm) were randomly taken from each of the three treated plots. Each of the six sets of samples was placed in separate paper bags. Further samples (eight cores per plot) were taken from the treated sites at regular intervals following application. Samples were similarly taken from the 0–10-cm depths of the control plots adjacent to the treated areas at 6–10 weekly intervals to confirm the absence of compounds that might interfere with the gas chromatographic analyses.

At ~4–10-week intervals, soil from the 10–20-cm soil depths was removed for analysis to determine if leaching of pendimethalin from the top soil had occurred. To avoid contamination by soil from the top 0–10 cm, a 20 × 20-cm (400-$cm^2$) metal frame, 10 cm in height and made of 1.5 mm sheet steel, was inserted into the soil to a depth of 10 cm, and the enclosed soil was carefully removed with a trowel and discarded. Then, the soil from the 10–20-cm level was carefully excavated. One sample was taken from each plot. After sampling, the holes were filled with untreated soil, and corners of sampled areas were marked with four pegs.

Sampling of the 0–10-cm and 10–20-cm treated plots and of the 0–10-cm control areas, as described, was also continued at regular intervals throughout 1994.

Immediately after collection, all soil samples were taken to the laboratory, weighed (~3–4 kg), and poured through a Riffle sampler to mix and divide the soil into two approximately equal parts. The parts were recombined and remixed five times. Finally, the soil was successively divided by repouring through the sampler until there was ~100 g of soil remaining. From this soil, a portion (40 g) was extracted (the day of sampling) and analyzed for pendimethalin.

**Soil Sampling from Fall Treatment.** Sampling of the four granular plots at Regina was not initiated until May 10, 1994, four days after completion of the second incorporation. Pre-application check plots adjacent to the treated plots were sampled on October 4, 1993, as just described. Samples (eight cores per plot) were taken from the 0–10-cm and 10–20-cm soil depths at regular intervals throughout 1994. Samples were similarly taken from the 0–10-cm depths of the control plots adjacent to the treated areas. Sample preparation prior to extraction and analysis was identical to that just described for the spring treatment.

**Soil Extraction and Analysis.** Soil extraction and analysis of pendimethalin residues were very similar to those described for trifluralin (Grover et al., 1988). The 40-g soil samples were placed in a 250-mL glass-stoppered flask with 100 mL of extraction solution containing acetonitrile, water, and glacial acetic acid (80:20:2.5, v/v/v). The samples were shaken for 1 h on a wrist-action shaker. The soil sample was then allowed to remain in contact with the extraction solvent overnight before being shaken for another 1-h period. The soil extracts were then centrifuged at 2000*g* for 4 min, and 25 mL of the supernatant was shaken with 100 mL of 5% aqueous sodium carbonate and 25 mL of *n*-hexane in a 250-mL separatory funnel. After separation, the hexane layer was collected in a 50-mL glass-stoppered flask, dried for 5 min over 5 g of anhydrous sodium chloride, and analyzed for pendimethalin by gas chromatography.

Following analysis, the concentration of pendimethalin in the total soil from each plot was calculated. The weight of the soil samples was based on an area of 365 $cm^2$, so amounts of herbicide remaining could be determined as kilograms per hectare.

**Gas Chromatographic Analysis.** A Hewlett-Packard Model 5890 gas chromatograph equipped with a $^{63}Ni$ electron-capture detector was operated at 350 °C. The megabore column (30 m × 0.53 mm i.d.) was of fused silica coated with 0.88 μm of HP-1. Carrier gas was helium at a flow rate of 5 mL $min^{-1}$; for the detector, a nitrogen makeup flow of 30 mL $min^{-1}$ was used. All samples (2 μL) were injected directly onto the column at 70 °C, with a Hewlett-Packard Model 7673A automatic sampler. This temperature was maintained for 1 min. The column temperature was then increased at a rate of 25 °C $min^{-1}$ and held at 270 °C for 4 min. Under these conditions, the retention time for pendimethalin was 10.4 min. Data from unknown samples and standards of known pendimethalin concentrations, in hexane, were plotted and integrated with a Hewlett-Packard 300 Series chromatography work station.

**Soil Recovery Studies.** Replicate air dry soil samples (40 g) collected from the top 10 cm at each site and sieved to pass a 2-mm screen were treated with the appropriate amounts of pendimethalin solution (containing 1.0 or 0.1 mg $mL^{-1}$ methanol). After thorough mixing, these samples were incubated in the dark at 20 °C for various time intervals (cf. Table 2) prior to extraction and analysis as described. Over a variety of concentrations and incubation periods (Table 2), pendimethalin recoveries were all in excess of 100%, averaging ~115% for both soils (Table 2). The reported field data were not corrected for any recovery factor. Recoveries of pendimethalin from soils collected from the 10–20-cm soil depths and fortified at the 0.2-μg $g^{-1}$ level were the same as those from fortified top soil (Table 2). Extraction and analysis of untreated control soils indicated that there were no interfering substances with the same gas chromatographic retention time as pendimethalin.

**Table 2. Recoveries of Pendimethalin from Fortified Soils**

| depth (cm) | added ($\mu$g g$^{-1}$) | replicates | fortification time before extraction | recovered[a] (%) Regina | Estlin |
|---|---|---|---|---|---|
| 0–10 | 2.0 | 3 | 24 h | 112 ± 1 | —[b] |
| 0–10 | 2.0 | 3 | 10 weeks | 116 ± 8 | — |
| 0–10 | 1.5 | 6 | 15 min | 113 ± 4 | 115 ± 4 |
| 0–10 | 1.5 | 3 | 72 h | 122 ± 6 | 116 ± 4 |
| 0–10 | 1.0 | 3 | 24 h | 119 ± 6 | — |
| 0–10 | 1.0 | 3 | 10 weeks | 115 ± 6 | — |
| 0–10 | 0.75 | 6 | 48 h | 113 ± 3 | 117 ± 7 |
| 0–10 | 0.20 | 4 | 72 h | 117 ± 4 | 117 ± 4 |
| 0–10 | 0.20 | 3 | 10 weeks | 122 ± 4 | — |
| 10–20 | 0.20 | 6 | 48 h | 112 ± 9 | 114 ± 5 |
| overall mean and standard deviation | | | | 116 ± 7 | 115 ± 4 |

[a] Mean and standard deviation. [b] Not determined.

**Table 3. Pendimethalin Residues Remaining with Time in a Clay Field Soil at Estlin Following Treatment with an Emulsifiable Concentrate Formulation at 1.21 kg ha$^{-1}$ on May 28, 1993**

| date | days after application | rainfall since last sampling (mm) | pendimethalin[a] (kg of ai ha$^{-1}$) 0–10-cm soil depth | 10–20-cm soil depth |
|---|---|---|---|---|
| May 28, 1993 | 0 | 0 | 1.21 ± 0.21[b,c] | —[d] |
| June 3, 1993 | 6 | 7 | 0.96 ± 0.16 | — |
| June 10, 1993 | 13 | 4 | 0.92 ± 0.28 | — |
| June 23, 1993 | 26 | 27 | 0.77 ± 0.18 | <0.05 |
| July 19, 1993 | 52 | 153 | 0.82 ± 0.04 | <0.05 |
| Aug 13, 1993 | 77 | 86 | 0.65 ± 0.06 | <0.05 |
| Sept 20, 1993 | 115 | 107 | 0.50 ± 0.16 | <0.05 |
| total rainfall | | 384 | | |
| May 10, 1994 | 347 | — | 0.29 ± 0.10 | <0.05 |
| July 18, 1994 | 416 | 117 | 0.38 ± 0.12 | <0.05 |
| Sept 19, 1994 | 479 | 39 | 0.34 ± 0.07 | <0.05 |
| total rainfall | | 156 | | |

[a] Mean and standard deviation from the analysis of three replicate plots; data are uncorrected for extraction recovery. [b] Mean and standard deviation from the analysis of two sets of samples from each of three replicate plots. [c] Analysis of soil samples from 0–10 cm of three control plots adjacent to treatment area after −7, 52, 73, 115, 347, 416, and 479 days indicated <0.05 kg ha$^{-1}$ of pendimethalin. [d] Not determined.

## RESULTS AND DISCUSSION

Although an application rate of 1.1 kg ha$^{-1}$ was intended, mean rates of 1.21 and 1.11 kg ha$^{-1}$ were measured at Estlin and Regina, respectively (Tables 3 and 4). Following application, there was a loss of pendimethalin from the EC treatments at Estlin and Regina (Tables 3 and 4) so that by the third week in September, 1993, pendimethalin remaining at the Estlin site was 0.50 ± 0.16 kg ha$^{-1}$ (41 ± 13% of that applied) and at the Regina site was 0.34 ± 0.07 kg ha$^{-1}$ (31 ± 6%). Pendimethalin residues detected during the second week of May, 1994, at the individual sites, were not significantly different ($p < 0.05$, according to Duncan's multiple range test) from those observed in September, 1993 (Tables 3 and 4). During 1994, at Estlin, there was little further dissipation of pendimethalin (Table 3). In contrast, there was residue loss at the nearby Regina location (Table 4).

By the end of the 479-day study during the third week of September, 1994, the amounts of pendimethalin remaining at Estlin (Table 3) and Regina (Table 4) were 0.34 ± 0.07 (28 ± 6% of that applied) and 0.13 ± 0.05 kg ha$^{-1}$ (12 ± 4% of that applied), respectively. Although the two sites were only 15 km apart, the rainfall during the summer of 1994 (cf. Tables 3 and 4) was lower at Estlin than Regina. The higher rainfall at the latter site may have been responsible for the greater herbicide dissipation, possibly though increased microbiological breakdown or through volatility losses, given the relatively high vapor pressure of pendimethalin.

**Table 4. Pendimethalin Residues Remaining with Time in a Clay Field Soil at Regina Following Treatment with an Emulsifiable Concentrate Formulation at 1.11 kg ha$^{-1}$ on May 28, 1993**

| date | days after application | rainfall since last sampling (mm) | pendimethalin[a] (kg of ai ha$^{-1}$) 0–10-cm soil depth | 10–20-cm soil depth |
|---|---|---|---|---|
| May 28, 1993 | 0 | 0 | 1.11 ± 0.18[b,c] | —[d] |
| June 3, 1993 | 6 | 9 | 0.87 ± 0.20 | — |
| June 10, 1993 | 13 | 3 | 1.10 ± 0.46 | — |
| June 23, 1993 | 26 | 37 | 0.86 ± 0.22 | <0.05 |
| July 19, 1993 | 52 | 146 | 0.52 ± 0.09 | <0.05 |
| Aug 13, 1993 | 77 | 53 | 0.36 ± 0.04 | <0.05 |
| Sept 24, 1993 | 119 | 130 | 0.34 ± 0.07 | <0.05 |
| total rainfall | | 378[e] | | |
| May 10, 1994 | 347 | — | 0.32 ± 0.06 | <0.05 |
| July 18, 1994 | 416 | 167 | 0.16 ± 0.13 | <0.05 |
| Sept 19, 1994 | 479 | 79 | 0.13 ± 0.05 | <0.05 |
| total rainfall | | 246[f] | | |

[a] Mean and standard deviation from the analysis of three replicate plots; data are uncorrected for extraction recovery. [b] Mean and standard deviation from the analysis of two sets of samples from each of three replicate plots. [c] Analysis of soil samples from 0–10 cm of three control plots, adjacent to treatment area after −7, 52, 73, 119, 347, 409, and 479 days indicated <0.05 kg ha$^{-1}$ of pendimethalin. [d] Not determined. [e] Thirty year av rainfall June 1–Sept 30 = 215 mm. [f] Thirty year av rainfall May 1–Sept 30 = 255 mm.

**Table 5. Pendimethalin Residues Remaining with Time in a Clay Field Soil at Regina Following Treatment with a Granular Formulation at 1.51 kg ha$^{-1}$ on October 4, 1993**

| date | days after application | rainfall since last sampling (mm) | pendimethalin[a] (kg of ai ha$^{-1}$) 0–10-cm soil depth | 10–20-cm soil depth |
|---|---|---|---|---|
| May 10, 1994 | 218 | —[b] | 1.01 ± 0.15[c] | 0.05 ± 0.05 |
| July 11, 1994 | 280 | 154 | 0.58 ± 0.12 | <0.05 |
| Sept 19, 1994 | 350 | 92 | 0.45 ± 0.08 | <0.05 |
| total rainfall | | 246[d] | | |

[a] Mean and standard deviation from the analysis of four replicate plots; data are uncorrected for extraction recovery. [b] Not determined. [c] Analysis of soil samples from the 0–10 cm of three control plots, adjacent to treatment area after 0, 218, 280, and 350 days indicated <0.05 kg ha$^{-1}$ of pendimethalin. [d] Thirty year av rainfall May 1–Sept 30 = 255 mm.

Dissipation of pendimethalin (1.11 kg ha$^{-1}$) from the granular formulation at Regina is summarized in Table 5. Following treatment in early October, 1993, 1.01 ± 0.15 kg ha$^{-1}$ (67 ± 10% of that applied) was recovered from the 0–10 cm of soil from the treated plots in May 1994. This result indicated that about two-thirds of that applied remained 218 days after application. During the spring and summer of 1994, there was a dissipation of the herbicide so that by the third week of September, 350 days after application, 0.45 ± 0.08 kg ha$^{-1}$ (30 ± 5%) of the initial treatment remained.

Over the 479-day study period, there was no movement of pendimethalin from the EC formulation to depths below 10 cm at either site (Tables 3 and 4). Similarly, there was no leaching of the herbicide from the granular treatment below 10 cm over 350 days (Table 5). During the study period, rainfall at Regina

was greater than the 30-year average during 1993, whereas for 1994, the rainfall was close to the average (Table 4).

It is concluded that residues of pendimethalin, following spring treatments at two sites, were carried over in Saskatchewan soils to the next crop year in amounts between 12 and 28% of that applied. Following fall treatment with a granular formulation at one site, 30% of that applied remained the following fall. However, it must be remembered that both 1993 and 1994 were years of adequate moisture. In drier years, the dissipation could be slower. Pendimethalin was not leached under western Canadian conditions.

ACKNOWLEDGMENT

We thank B. Caldwell for site preparation and herbicide application and incorporation L. Goulet for technical help, and L. Drew for advice and for obtaining the weather data at the Estlin site.

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Received for review March 30, 1995. Accepted August 18, 1995.[⊗] The financial assistance of Cyanamid Canada Inc. is gratefully acknowledged.

JF950188J

[⊗] Abstract published in *Advance ACS Abstracts,* October 15, 1995.