

5-31-84  
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CASE GS0187 PENDIMETHALIN STUDY 1 PM PM# 02/15/83

CHEM 108501 Pendimethalin

BRANCH EFB DISC 30 TOPIC 05

FORMULATION 00 - ACTIVE INGREDIENT

FICHE/MASTER ID 00106777-A CONTENT CAT 01

Zulalian, J., and S.K. Eisner. 1974. Prowl herbicide: A study on the behavior of <sup>14</sup>C-labeled CL 92,553 in an hydrolytic environment.

SUBST. CLASS = S.

DIRECT RVW TIME = 6 (MH) START-DATE END DATE

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CONCLUSIONS:Degradation - Hydrolysis

1. This study is scientifically valid.
2. [<sup>14</sup>C]Pendimethalin, at 0.51 and 5.1 ppm, was stable to hydrolysis for up to 4 weeks in sterile, distilled water (buffered at pH 5, 7 and 9) at 20-25 C.
3. This study fulfills EPA Data Requirements for Registering Pesticides (1983) by showing that pendimethalin is stable to hydrolysis for a minimum of 4 weeks in buffered solutions (pH 5, 7 and 9) at 20-25 C.

## MATERIALS AND METHODS:

4-Methyl-labeled [ $^{14}\text{C}$ ]pendimethalin (Prowl, 14.9  $\mu\text{Ci}/\text{mg}$ , >99% pure, Stamford Laboratories) in 100  $\mu\text{l}$  of ethanol was added to buffer solutions adjusted to pH 5.0, 7.0 and 9.0 at 0.51 and 5.1 ppm (100 and 10 ml final volume, respectively). These treatment rates corresponded to ~1x and 10x the solubility of pendimethalin in water. The buffer solutions were prepared from pHydriol buffer mixes and sterile distilled water. The flasks of solution were sealed, wrapped in aluminum foil, and stored in the dark at 20-25 C.

The buffered solutions were sampled immediately after treatment (pH 5.0 at 5.1 ppm only), and at 0.5, 1, 2, and 4 weeks after treatment. Samples were extracted with three 25-30 ml portions of ethyl acetate in a separatory funnel. The volume of the extract was then adjusted to 100 ml with ethyl acetate and triplicate 1-ml aliquots were taken for analysis by LSC.

The extracts were also analyzed by TLC. A one-dimensional TLC system using a 1,2-dichloroethylene (cis-trans mixture):nitromethane:carbon tetrachloride (210:70:70, v:v:v) solvent mixture was used to analyze samples taken at weeks 0, 0.5, 1, and 2 after treatment (volume analyzed unspecified). Solutions sampled 4 weeks after treatment were analyzed using two-dimensional TLC, with xylene:chloroform:methanol (280:70:7, v:v:v) and 1,2-dichloroethylene (cis-trans mixture):nitromethane:carbon tetrachloride (210:70:70, v:v:v) as the solvent systems. The radio-active areas on the developed plates were located by autoradiography and composed to a known pendimethalin standard.

## REPORTED RESULTS:

During the 4 week study period, between 94 and 104% of the applied radioactivity was recovered in the ethyl acetate extracts from the three buffer systems (pH 5.0, 7.0, and 9.0) at both 0.51 and 5.1 ppm (Table 1). The autoradiograms of the TLC plates indicated that pendimethalin was the only radioactive substance present in detectable amounts in all systems.

## DISCUSSION:

The solubility of pendimethalin was reported to be 0.5 ppm. Therefore, the majority of the [ $^{14}\text{C}$ ]pendimethalin at the 5.1 ppm treatment rate remained undissolved and similar concentrations of the herbicide were in solution in the 0.51 and 5.1 ppm treatments.

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Table 1. Recovery of radioactivity (%) from buffered aqueous solutions of [ $^{14}\text{C}$ ]pendimethalin stored in the dark at room temperature.

	Sampling interval (weeks)														
	0			0.5			1			2			4		
	Extract <sup>a</sup>	Aqueous	Total	Extract <sup>a</sup>	Aqueous	Total	Extract <sup>a</sup>	Aqueous	Total	Extract <sup>a</sup>	Aqueous	Total	Extract <sup>a</sup>	Aqueous	Total
pH 5 <sup>b</sup>	92.0	0.35	92.35	99.88	0.05	99.93	98.82	0.04	98.86	96.74	0.07	96.81	100.02	0.05	100.07
pH 7 <sup>b</sup>	--- <sup>c</sup>	--	--	98.71	0.07	98.78	101.20	0.06	101.26	102.04	0.31	102.35	99.12	0.16	99.28
pH 9 <sup>b</sup>	--	--	--	101.46	0.04	101.50	102.01	0.06	102.07	99.40	0.05	99.45	101.47	0.13	101.60
pH 5 <sup>d</sup>	--	--	--	99.47	0.05	99.52	99.74	0.02	99.76	98.45	0.01	98.46	96.85	0.10	96.95
pH 7 <sup>d</sup>	--	--	--	101.84	0.19	102.03	99.14	0.05	99.19	99.44	0.03	99.47	94.17	0.13	94.30
pH 9 <sup>d</sup>	--	--	--	104.41	0.07	104.48	93.82	0.04	93.86	93.87	0.10	93.97	97.01	0.12	97.13

<sup>a</sup>Represents ethyl acetate-soluble radioactivity.

<sup>b</sup>Concentration of [ $^{14}\text{C}$ ]pendimethalin dissolved in 100 ml of buffer was 0.51 ppm.

<sup>c</sup>Not determined.

<sup>d</sup>Concentration of [ $^{14}\text{C}$ ]pendimethalin in 10 ml of buffer was 5.10 ppm (only partially dissolved).

CASE GS0187      PENDIMETHALIN      STUDY 32      PM PM# 02/15/83

CHEM 108501      Pendimethalin

BRANCH EFB      DISC 30 TOPIC 05

FORMULATION 00 - ACTIVE INGREDIENT

FICHE/MASTER ID 00106777-B      CONTENT CAT 01

Moyer, M., J. Wyckoff, and G. Nzewi. 1974. Prowl (CL 92,553): Determination of CL 92,553 [N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine] and CL 202,347 [4-(1-ethylpropyl)amino)-2-methyl-3,5-dinitro-benzyl alcohol] residues in corn (foliage) and soybeans (foliage) and CL 92,553 in soil.

SUBST. CLASS = S.

DIRECT RVW TIME = 4      (MH) START-DATE      END DATE

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CONCLUSION:Field Dissipation - Terrestrial

This study is scientifically invalid because the sampling protocol was inadequate to accurately assess the dissipation of pendimethalin from soil. In addition, this study would not fulfill EPA Data Requirements for Registering Pesticides (1983) because the test soil was not completely characterized, meteorological data including rainfall data were not provided, a nonspecific analytical method was used, and no pretreatment soil samples were taken.

### MATERIALS AND METHODS:

Field plots (40 x 100 ft) of clay soil (5.5% organic matter, soil not further characterized) near Waseca, Minnesota, were treated on May 17, 1973, and again on June 4, 1974, with a single preemergent broadcast application of either 3 lb/gal EC or 4 lb/gal EC pendimethalin (Prowl, American Cyanamid Co.) at 2 lb ai/A. There was one control plot. Corn and soybeans were planted on May 28, 1974.

Twenty soil cores (0- to 3-, 3- to 6- and 6- to 9-inch depths) were taken from each treatment on July 8, 34 days after application of pendimethalin. The samples from a common treatment were combined, and analyzed for total pendimethalin residues (pendimethalin and degradates) using GC according to Method M-520, which is identical to Method M-437 (described in 00029034, Study 12), with the exception that soil (~25 g) was extracted in a 6:4:1 mixture of acidic methanol: water:soil. The detection limit was 0.05 ppm.

### REPORTED RESULTS:

Pendimethalin residues were detected as deep as 6- to 9-inches in a clay soil 34 days after application of 2 lb ai/A, although <1.0 ppm were detected below the 0- to 3-inch depth. In the 0- to 3-inch depth of soil treated with 3 lb/gal EC, 0.7 ppm of pendimethalin residues were detected under corn and 0.8 ppm under soybeans. At the same depth of soil treated with 4 lb/gal EC, 1.2 ppm of pendimethalin residues were detected under corn and 0.8 ppm under soybeans. Pendimethalin residues were slightly higher in the 3- to 6-inch depth (0.85 vs. 0.40 ppm) and 6- to 9-inch depth (0.35 vs. 0.20 ppm) of soil treated with the 3 lb/gal EC formulation than the 4 lb/gal EC formulation, respectively, regardless of crop.

### DISCUSSION:

1. The sampling schedule was inadequate (one sampling interval) to accurately establish the pattern of decline of pendimethalin and patterns of formation and decline of degradates in soil. No pretreatment soil samples were taken, and immediate posttreatment samples were not analyzed to confirm pendimethalin application rates.
  2. The analytical method used determined total pendimethalin residues in soil; parent pendimethalin and its degradates were not distinguished.
  3. Meteorological data were not provided.
  4. Complete soil characteristics, including pH and CEC, were not provided.
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Table 1. Pendimethalin residues (ppm) in soil samples from field plots of corn and soybeans treated with pendimethalin (3 lb/gal EC; 4 lb/gal EC) at 2.0 lb ai/A in both 1973 and 1974.

Formulation of pendimethalin	Crop	Sampling interval (days)	Sampling depth (inches)		
			0-3	3-6	6-9
3 lb/gal EC	Corn	34	0.75 <sup>a</sup>	0.92	0.34
	Soybeans	34	0.78	0.60	0.44
4 lb/gal EC	Corn	34	1.20	0.44	0.22
	Soybeans	34	0.81	0.40	0.16

<sup>a</sup> Detection limit is 0.05 ppm.

CASE GS0187 PENDIMETHALIN STUDY 33

PM PM# 02/15/83

CHEM 108501 Pendimethalin

BRANCH EFB DISC 30 TOPIC 04

FORMULATION 12 - EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 00106777-C CONTENT CAT 01

Bohn, W., C. Potts, M. Moyer, et al. 1974. Prowl (CL 92,553): Determination of CL 92,553 [N-ethylpropyl-3,4-dimethyl-2,6-dinitrobenzenamine] and CL 202,347 [4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitro-benzyl alcohol] residues in beets (foliage and roots), corn (foliage), soybeans (foliage) and wheat (foliage) and CL 92,553 in soil.

SUBST. CLASS = S.

DIRECT RVW TIME = 8 (MH) START-DATE END DATE

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CONCLUSIONS:Field Accumulation - Rotational Crops

1. This study is scientifically valid.
2. Neither pendimethalin residues (uncharacterized) nor 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) were detected (<0.05 ppm) in the foliage of corn, soybean, and spring wheat grown for 42 days, and beets (foliage and roots) grown for 61 days in a silty clay loam soil treated the previous 2 years with pendimethalin (3 lb/gal EC). Pendimethalin residues were detected at 0.15-0.18 ppm in the 0- to 3-inch depth 327 days after the last pendimethalin treatment (~3 weeks before planting).
3. This study does not fulfill EPA Data Requirements for Registering Pesticides (1983) because the test soil was not completely characterized, a nonspecific analytical method was used, field test data were not provided, and immediate posttreatment and day of harvest soil samples were not analyzed.


MATERIALS AND METHODS:

Three field plots (24 x 15 feet) of silty clay loam soil (3-4% organic matter, soil not further characterized) near Rochelle, Illinois, were planted to corn, soybeans, spring wheat, and red beets on April 25-26, 1974. One plot had been previously treated with pendimethalin (Prowl, 3 lb/gal EC, American Cyanamid Co.) at 2.0 lb ai/A on May 5, 1972, and 2.0 lb ai/A June 9, 1973; a second plot had been treated with 4 lb ai/A on May 5, 1972, and 2.0 lb ai/A on June 9, 1973. The third plot received no pendimethalin treatments. Soil samples (0- to 3-, 3- to 6-, and 6- to 9-inch depth) were taken on May 2, 1974, 327 days after the last pendimethalin application. Corn, soybean, and wheat foliage were sampled June 4, 1974, 42 days after planting. Beet foliage and roots were sampled June 25, 1974, 61 days after planting.

Soil samples from a common plot and sampling date were combined and analyzed for total pendimethalin residues (pendimethalin and degradates) using GC according to Method M-520, which is identical to Method M-437 (described in 00029034, Study 12) with the exception that soil (~25 g) was extracted in 6:4:1 mixture of acidic methanol:water:soil. The detection limit was 0.05 ppm.

Corn samples from a common treatment were combined and analyzed for total pendimethalin residues (pendimethalin and degradates) according to American Cyanamid Method M-458.1. Using this method, freshly frozen, chopped plant tissue (>10 g) was mixed with a volume of aqueous acidic methanol (20 ml concentrated HCl, 200 ml distilled water, and 780 ml methanol) to produce a solvent to sample ratio of 20:1. The extract was then mixed twice with a mixture of 50 ml 0.1 N HCl and 50 ml hexane, each time drawing the hexane partition out of the funnel. The hexane extract was evaporated to dryness, then redissolved in 10 ml of hexane. This solution was eluted through a deactivated Florisil column with hexane:benzene (80:20). The eluate was evaporated to dryness and redissolved in 5 ml of benzene. The benzene solution was diluted (amount unspecified) and an aliquot (size unspecified) analyzed for total pendimethalin residues using GC with an electron capture detector. The detection limit was 0.05 ppm, and recovery from fortified samples ranged from 87.5 to 126.7%.

Determination of 4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) in corn was by American Cyanamid Method M-459.1. Freshly frozen, chopped plant tissue (>10 g) was mixed with a volume of aqueous acidic methanol (described previously) to produce a solvent to sample ratio of 20:1. The extract was then mixed twice with a mixture of 50 ml 0.1 N HCl/NaCl (8.3 ml concentrated HCl, 120 g NaCl brought to 1000 ml with distilled water) and 50 ml hexane, each time drawing the hexane partition out of the funnel. The hexane extract was evaporated to dryness, then redissolved in 10 ml of acetic anhydride containing 2 drops of pyridine. After >2 hours, the solution was mixed with 25 ml of 10% sodium carbonate and extracted three times





with 50 ml of hexane, each time drawing the hexane partition out of the funnel. The hexane extract was evaporated to dryness, redissolved in 10 ml hexane, and eluted through a deactivated Florisil column with benzene. The eluate was evaporated to dryness, redissolved in 5 ml benzene and analyzed according to Method M-458.1 (previously described). The detection limit was 0.05 ppm, and recovery from fortified samples ranged from 75.2 to 110.8%.

Soybean samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-483, which is identical to Method M-458.1 (described previously). CL 202,347 was determined in the soybean foliage using GC according to Method M-531, which is identical to Method M-459.1 (described previously) with the exception that chloroform rather than hexane was used in the separatory funnel partitioning. The detection limit for both M-483 and M-531 was 0.05 ppm. Recovery from fortified samples ranged from 92.2 to 110.1% for pendimethalin and from 79.6 to 105.8% for CL 202,347.

Wheat samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-485, which is identical to Method M-458.1 (described previously). CL 202,347 was determined in the plant tissue using GC according to Method M-522, which is identical to Method M-459.1 (described previously). The detection limit for both M-485 and M-522 was 0.05 ppm. Recovery from fortified samples ranged from 76.4 to 133.7% for pendimethalin and from 71.5 to 120.7% for CL 202,347.

Beet samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-529, which is identical to Method M-458.1 (described previously). CL 202,347 was determined in the plant tissue using GC according to Method M-530, which is identical to Method M-459.1 (described previously). The detection limit for both M-529 and M-530 was 0.05 ppm. Recovery from fortified samples ranged from 75.3 to 142.0% for pendimethalin and from 73.1 to 119.4% for CL 202,347.

#### REPORTED RESULTS:

Neither pendimethalin residues nor CL 202,347 were detected in the corn, soybean, wheat, or beet foliage, or beet roots in either the 2.0 + 2.0 or 4.0 + 2.0 lb ai/A treatments (Table 1). Pendimethalin residues were detected at ~0.2 ppm in the 0- to 3-inch depth of soil of both treatments 327 days after the last application of pendimethalin at 2.0 lb ai/A. No pendimethalin residues were detected below the 3-inch depth of soil.

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DISCUSSION:

1. Field test data, such as amount of rainfall and irrigation water, depth of water table, slope of test site, and soil and air temperature data, were not reported.
2. Complete soil characteristics, such as pH and CEC, were not provided.
3. Immediate posttreatment samples were not analyzed to confirm pendimethalin application rates.
4. The analytical methods used determined total pendimethalin residues or CL 202,347; total pendimethalin residues could not be characterized.
5. The erratic recovery of pendimethalin from fortified samples (e.g., 75.3-142.0% for beets) and CL 202,347 (e.g., 71.5-120.7% for wheat) indicates considerable interference may have occurred in the analyses.
6. Pendimethalin application rates were not confirmed by soil analysis. No immediate posttreatment or day of harvest soil samples were collected and analyzed.

Table 1. Pendimethalin residues and CL 202,347 (ppm) in soil and four rotational crops in plots treated with pendimethalin (3 lb/gal EC) the two preceding years.

Sample	Treatment rate (lb ai/A)					Days posttreatment	Pendimethalin residues	CL 202,347
	1972	+	1973	+	1974			
Soil	0	+	0	+	0	---	---	---
Beet foliage	0	+	0	+	0	---	ND <sup>a</sup>	ND
Beet root	0	+	0	+	0	---	ND	ND
Corn foliage	0	+	0	+	0	---	ND	ND
Soybean foliage	0	+	0	+	0	---	ND	ND
Wheat foliage	0	+	0	+	0	---	ND	ND
Soil (0-3 inches)	2	+	2	+	0	327	0.15	---
(3-6 inches)	2	+	2	+	0	327	ND	---
(6-9 inches)	2	+	2	+	0	327	ND	---
Beet foliage	2	+	2	+	0	382	ND	ND
Beet root	2	+	2	+	0	382	ND	ND
Corn foliage	2	+	2	+	0	363	ND	ND
Soybean foliage	2	+	2	+	0	363	ND	ND
Wheat foliage	2	+	2	+	0	363	ND	ND
Soil (0-3 inches)	4	+	2	+	0	327	0.18	---
(3-6 inches)	4	+	2	+	0	327	ND	---
(6-9 inches)	4	+	2	+	0	327	ND	---
Beet foliage	4	+	2	+	0	382	ND	ND
Beet root	4	+	2	+	0	382	ND	ND
Corn foliage	4	+	2	+	0	363	ND	ND
Soybean foliage	4	+	2	+	0	363	ND	ND
Wheat foliage	4	+	2	+	0	363	ND	ND

<sup>a</sup> Not detected, detection limit is 0.05 ppm.

CASE GS0187      PENDIMETHALIN      STUDY 34      PM PM# 02/15/83

CHEM 108501      Pendimethalin

BRANCH EFB      DISC 30 TOPIC 05

FORMULATION 12 - EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 00106777-D      CONTENT CAT 01

Boughton P., J. Wyckoff, and G. Nzewi. 1974. Prowl (CL 92,553): Determination of CL 92,553 [N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine] and CL 202,347 [4-([1-ethylpropyl]amino)-2-methyl-3,5-dinitro-benzyl alcohol] residues in corn (foliage), soybeans (foliage), and wheat (foliage) and CL 92,553 in soil.

SUBST. CLASS = S.

DIRECT RVW TIME = 4 1/2 (MH) START-DATE      END DATE

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CONCLUSIONS:Field Accumulation - Rotational Crops

1. This study is scientifically valid.
2. Neither pendimethalin residues (uncharacterized) nor 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) accumulated (<0.05 ppm) in corn, soybean, or wheat foliage from plants grown for ~40 days in a clay loam soil treated the preceding year with pendimethalin (3 lb/gal EC). In the soil 376 days after the pendimethalin treatment [0 (corn and soybean) and ~39 (wheat) days after planting], pendimethalin residues of 0.45 ppm were detected in the 0- to 3-inch depth.
3. This study does not fulfill EPA Data Requirements for Registering Pesticides (1983) because the test soil was not completely characterized, field test data were insufficiently provided, a nonspecific analytical method was used, and immediate posttreatment and day of harvest (corn and soybeans) soil samples were not analyzed.

MATERIALS AND METHODS:

Two field plots (200 x 20 feet) of clay loam soil (5% organic matter, soil not further characterized) near Waseca, Minnesota, were planted to corn, soybeans, and beets on May 28, 1974, and to wheat on April 19, 1974. One plot had been previously treated with pendimethalin (Prowl, 3 lb/gal EC, American Cyanamid Co.) at 2 lb ai/A on May 17, 1973. The second plot received no pendimethalin treatment. Soil samples (0- to 3-, 3- to 6-, and 6- to 9-inch depth) were taken on May 28, 1974, 376 days after treatment. Wheat samples were obtained May 29, 1974, 40 days after planting, and corn and soybean samples were obtained July 8, 1974, 41 days after planting. No beet samples were obtained because of crop failure.

Soil samples from a common plot and sampling date were combined and analyzed for total pendimethalin residues (pendimethalin and degradates) using GC according to Method M-520, which is identical to Method M-437 (described in 00029034, Study 12) with the exception that soil (~25 g) was extracted in a 6:4:1 mixture of acidic methanol:water:soil. The detection limit was 0.05 ppm.

Corn samples from a common treatment were combined and analyzed for total pendimethalin residues and CL 202,347 in plant tissue using GC according to Methods M-458.1 and 459.1, respectively (described in 00106777-C, Study 33). The detection limit for both M-458.1 and M-459.1 was 0.05 ppm. Recovery from fortified samples ranged from 87.5 to 126.7% for pendimethalin and from 75.2 to 110.8% for CL 202,347.

Soybean samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-483, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the soybean foliage using GC according to Method M-531, which is identical to Method M-459.1 (described in 00106777-C, Study 33) with the exception that chloroform rather than hexane was used in the separatory funnel partitioning. The detection limit for both M-483 and M-531 was 0.05 ppm. Recovery from fortified samples ranged from 92.2 to 110.1% for pendimethalin and from 79.6 to 105.8% for CL 202,347.

Wheat samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-485, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the plant tissue using GC according to Method M-522, which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-485 and M-522 was 0.05 ppm. Recovery from fortified samples ranged from 76.4 to 133.7% for pendimethalin and from 71.5 to 120.7% for CL 202,347.

REPORTED RESULTS:

Neither pendimethalin residues nor CL 202,347 were detected in the corn, soybean, or wheat foliage in the clay loam soil treated the preceding year with pendimethalin at 2.0 lb ai/A (Table 1). In the upper 3 inches of soil in the treated plots, 0.45 ppm pendimethalin residues were recovered 376 days after the last treatment. No pendimethalin was detected (<0.05 ppm) in the 3- to 6- or 6- to 9-inch depths of soil.

DISCUSSION:

1. Field test data, such as meteorological data, irrigation practices, depth of water table, and slope of test site, were not reported.
2. Complete soil characteristics, such as pH and CEC, were not provided.
3. Immediate posttreatment soil samples were not analyzed to confirm pendimethalin application rates.
4. The analytical methods used determined total pendimethalin residues or CL 202,347; total pendimethalin residues could not be characterized.
5. The erratic recovery of pendimethalin (e.g. 76.4-133.7% for wheat) and CL 202,347 (e.g. 71.5-120.7% for wheat) from fortified samples indicates considerable interference may have occurred in the analyses.
6. Pendimethalin application rates were not confirmed by immediate post-treatment soil analyses.

Table 1. Pendimethalin residues and CL 202,347 (ppm) in soil and three rotational crops in clay loam soil treated with pendimethalin (3 lb/gal EC) at 2.0 lb ai/A the previous year.

Sample	Treatment rate (lb ai/A)	Days posttreatment	Pendimethalin residues	CL 202,347
	1973 + 1974			
Soil	0 + 0	--	--	--
Corn foliage	0 + 0	--	ND <sup>a</sup>	ND
Soybean foliage	0 + 0	--	ND	ND
Wheat foliage	0 + 0	--	ND	ND
Soil (0-3 inches)	2.0 + 0	376	0.45	--
(3-6 inches)	2.0 + 0	376	ND	--
(6-9 inches)	2.0 + 0	376	ND	--
Corn foliage	2.0 + 0	417	ND	ND
Soybean foliage	2.0 + 0	417	ND	ND
Wheat foliage	2.0 + 0	417	ND	ND
Beet foliage <sup>b</sup>	2.0 + 0	--	--	--
Beet roots	2.0 + 0	--	--	--

<sup>a</sup> Not detected, detection limit is 0.05 ppm.

<sup>b</sup> Beet crop failed, no samples analyzed.

CASE GS0187      PENDIMETHALIN      STUDY 35      PM PM# 02/15/83

CHEM 108501      Pendimethalin

BRANCH EFB      DISC 30 TOPIC 05

FORMULATION 12 - EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 00106777-E      CONTENT CAT 01

Bohn, W., C. Potts, P. Boughton, et al. 1974. Prowl (CL 92,553): Determination of CL 92,553 [N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine] and CL 202,347 [4-([1-ethylpropyl]amino)-2-methyl-3,5-dinitro-benzyl alcohol] residues in beets (foliage and roots), corn (foliage), soybeans (foliage), and oats (foliage) and CL 92,553 in soil.

SUBST. CLASS = S.

DIRECT RVW TIME = 3      (MH) START-DATE      END DATE

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CONCLUSIONS:Field Accumulation - Rotational Crops

1. This study is scientifically valid.
2. Neither pendimethalin residues (uncharacterized) nor 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) accumulated (<0.05 ppm) in corn, soybean, oat, or beet foliage, or beet roots from plants grown for ~1 month in soil treated the preceding year with pendimethalin (3 lb/gal EC). In the soil 373 days after the pendimethalin treatments [0 days (corn and oat) and ~1 month (soybean and beets) after planting], pendimethalin residues of <0.60 ppm were detected in the 0- to 9-inch depth.
3. This study does not fulfill EPA Data Requirements for Registering Pesticides (1983) because the test soil was not characterized, field test data were insufficiently provided, a nonspecific analytical method was used, and immediate posttreatment and day of harvest soil samples were not analyzed.



### MATERIALS AND METHODS:

Two field plots (size unspecified, soil not characterized) near Bement, Illinois, were planted to corn and oats on May 26, 1974, to soybeans on June 28, 1974, and to red beets on June 30, 1974. One plot had been previously treated with pendimethalin (Prowl, 3 lb/gal EC, American Cyanamid Co.) at 2.0 lb ai/A on May 18, 1973; the other had been treated with pendimethalin at 6.0 lb ai/A on the same date. Soil samples (0- to 3-, 3- to 6-, and 6- to 9-inch depth) were taken on May 26, 1974, 373 days after treatment. Corn and oat foliage samples were obtained June 27, 1974, 32 days after planting. Soybean (foliage) and red beet (foliage and roots) samples were obtained July 29, 1974, 32 and 30 days after planting, respectively.

Soil samples from a common plot and sampling date were combined and analyzed for total pendimethalin residues (pendimethalin and degradates) using GC according to Method M-520, which is identical to Method M-437 (described in 00029034, Study 12) with the exception that soil (~25 g) was extracted in a 6:4:1 mixture of acidic methanol:water:soil. The detection limit was 0.05 ppm.

Corn samples from a common treatment were combined and analyzed for total pendimethalin residues and 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) in plant tissue using GC according to Methods M-458.1 and 459.1, respectively (described in 00106777-C, Study 33). The detection limit for both M-458.1 and M-459.1 was 0.05 ppm. Recovery from fortified samples ranged from 87.5 to 126.7% for pendimethalin and from 75.2 to 110.8% for CL 202,347.

Soybean samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-483, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the soybean foliage using GC according to Method M-531, which is identical to Method M-459.1 (described in 00106777-C, Study 33) with the exception that chloroform rather than hexane was used in the separatory funnel partitioning. The detection limit for both M-483 and M-531 was 0.05 ppm. Recovery from fortified samples ranged from 92.2 to 110.1% for pendimethalin and from 79.6 to 105.8% for CL 202,347.

Oat samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-538, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the plant tissue using GC according to Method M-539, which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-538 and M-539 was 0.05 ppm. Recovery from fortified samples ranged from 88.9 to 119.3% for pendimethalin and from 73.1 to 116.5% for CL 202,347. Beet samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according

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to Method M-529, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the plant tissue using GC according to Method M-530, which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-529 and M-530 was 0.05 ppm. Recovery from fortified samples ranged from 75.3 to 142.0% for pendimethalin and from 73.1 to 119.4% for CL 202,347.

#### REPORTED RESULTS:

Neither pendimethalin residues nor CL 202,347 were detected (<0.05 ppm) in the corn, soybean, oat, or beet foliage, or beet roots in the soils treated the previous year with pendimethalin at 2.0 or 6.0 lb ai/A (Table 1). Pendimethalin residues were recovered at 0- to 3-, 3- to 6-, and 6- to 9-inch depths of soil in both treatments. In soil treated at 2.0 lb ai/A, 0.15, 0.13 and 0.09 ppm pendimethalin residues were detected at the 0- to 3-, 3- to 6-, and 6- to 9-inch depths, respectively. In soil treated at 6.0 lb ai/A, 0.23, 0.66 and 0.91 ppm were detected.

#### DISCUSSION:

1. Field test data, such as meteorological data, depth of water table, slope of test site, and irrigation practices, were not provided.
2. Soil characteristics, such as texture, pH and organic matter content, were not provided.
3. Immediate posttreatment soil samples were not analyzed to confirm pendimethalin application rates.
4. The erratic recovery of pendimethalin (e.g., 75.3-142.0% for beets) and CL 202,347 (e.g., 73.1-119.4% for beets) indicates considerable interference may have occurred in the analyses.
5. The analytical methods used determined total pendimethalin residues or CL 202,347; total pendimethalin residues could not be characterized.

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Table 1. Pendimethalin residues and CL 202,347 (ppm) in soil and four rotational crops in plots treated with pendimethalin (3 lb/gal EC) at 2.0 or 6.0 lb ai/A the previous year.

Sample	Treatment rate (lb ai/A) 1973 + 1974	Days posttreatment	Pendimethalin residues	CL 202,347
Soil	0 + 0	--	--	--
Corn foliage	0 + 0	--	ND <sup>a</sup>	ND
Soybean foliage	0 + 0	--	ND	ND
Oat foliage	0 + 0	--	ND	ND
Beet foliage	0 + 0	--	ND	ND
Beet roots	0 + 0	--	ND	ND
Soil (0-3 inches)	2.0 + 0	373	0.15	--
(3-6 inches)	2.0 + 0	373	0.13	--
(6-9 inches)	2.0 + 0	373	0.09	--
Corn foliage	2.0 + 0	405	ND	ND
Soybean foliage	2.0 + 0	437	ND	ND
Oat foliage	2.0 + 0	405	ND	ND
Beet foliage	2.0 + 0	437	ND	ND
Beet roots	2.0 + 0	437	ND	ND
Soil (0-3 inches)	6.0 + 0	373	0.23	--
(3-6 inches)	6.0 + 0	373	0.66	--
(6-9 inches)	6.0 + 0	373	0.91	--
Corn foliage	6.0 + 0	405	ND	ND
Soybean foliage	6.0 + 0	437	ND	ND
Oat foliage	6.0 + 0	405	ND	ND
Beet foliage	6.0 + 0	437	ND	ND
Beet roots	6.0 + 0	437	ND	ND

<sup>a</sup> Not detected, detection limit is 0.05 ppm.

CASE GS0187      PENDIMETHALIN      STUDY 36      PM PM# 02/15/83

CHEM 108501      Pendimethalin

BRANCH EFB      DISC 30 TOPIC 05

FORMULATION 12 - EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 00106777-F      CONTENT CAT 01

Boughton, P., J. Wyckoff, and G. Nzewi. 1974. Prowl (CL 92,553): Determination of CL 92,553 [N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine] and CL 202,347 [3-([1-ethylpropyl]amino)-2-methyl-3,5-dinitro-benzyl alcohol] residues in beets (foliage and roots), corn (foliage) and wheat (foliage) and CL 92,553 in soil.

SUBST. CLASS =

DIRECT RVW TIME = 3      (MH) START-DATE      END DATE

REVIEWED BY: K. Patten

TITLE: Staff Scientist

ORG: Dynamac Corp., Enviro Control Division, Rockville, MD

TEL: 468-2500

SIGNATURE: *K. Patten*

DATE: May 24, 1984

APPROVED BY:

TITLE:

ORG:

TEL:

SIGNATURE:

DATE:

CONCLUSIONS:Field Accumulation - Rotational Crops

1. This study is scientifically valid.
2. Neither pendimethalin residues nor 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) accumulated (<0.05 ppm) in corn or wheat foliage or whole red beet plants grown for 55, 25, and 36 days, respectively, in a silt loam soil treated the preceding year with pendimethalin (3 lb/gal EC). In the 0- to 6-inch depth of soil, pendimethalin residues of <0.33 ppm were detected 349 days after the pendimethalin treatment (31, 15, and 2 days after planting of corn, wheat, and beets, respectively). No pendimethalin was detected in the soil at the 6- to 9-inch depth.
3. This study does not fulfill EPA Data Requirements for Registering Pesticides (1983) because field test data were insufficiently provided, a non-specific analytical method was used, and immediate posttreatment and day of harvest soil samples were not analyzed.

MATERIALS AND METHODS:

Three field plots (20 x 100 feet) of a silt loam soil (13.5% sand, 60% silt, 26% clay, 3.5% organic matter) near York, Nebraska, were planted to corn, wheat, and red beets on April 16, May 2, and May 15, 1974, respectively. Two of the plots had been treated the previous year, on May 3, 1973, with pendimethalin (Prowl, 3 lb/gal EC, American Cyanamid Co.) at 1.5 and 2.0 lb ai/A. The third plot remained untreated. Soil samples (0- to 3-, 3- to 6- and 6- to 9-inch depths) were obtained on May 17 and June 2, 1974, 349 and 364 days after the pendimethalin treatment, respectively. Wheat foliage was sampled May 27 (389 days after pendimethalin treatment), corn foliage was sampled June 10 (403 days after) and beet tops and roots were sampled June 20, 1974 (413 days after).

Soil samples from a common plot and sampling date were combined and analyzed for total pendimethalin residues (pendimethalin and degradation products) using GC according to Method M-520, which is identical to Method M-437 (described in 00029034, Study 12) with the exception that soil (~25 g) was extracted in a 6:4:1 mixture of acidic methanol:water:soil. The detection limit was 0.05 ppm.

Corn samples from a common treatment were combined and analyzed for total pendimethalin residues and 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) in plant tissue using GC according to Methods M-458.1 and 459.1, respectively (described in 00106777-C, Study 33). The detection limit for both M-458.1 and M-459.1 was 0.05 ppm. Recovery from fortified samples ranged from 87.5 to 126.7% for pendimethalin and from 75.2 to 110.8% for CL 202,347.

Wheat samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-485, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the plant tissue using GC according to Method M-533, which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-485 and M-522 was 0.05 ppm. Recovery from fortified samples ranged from 76.4 to 133.7% for pendimethalin and from 71.5 to 120.7% for CL 202,347.

Beet samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-529, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the plant tissue using GC according to Method M-530, which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-529 and M-530 was 0.05 ppm. Recovery from fortified samples ranged from 75.3 to 142.0% for pendimethalin and from 73.1 to 119.4% for CL 202,347.

REPORTED RESULTS:

Neither pendimethalin residues nor CL 202,347 were detected ( $<0.05$  ppm) in the corn, wheat, or beets grown in the silt loam soil treated the previous year with pendimethalin at 1.5 or 2.0 lb ai/A (Table 1). Pendimethalin residues were recovered from the upper 6 inches of soil for both treatments, but not from the 6- to 9-inch depth. Soil treated at 1.5 lb ai/A contained pendimethalin residues of 0.14 ppm in the 0- to 3-inch depth and 0.22 ppm in the 3- to 6-inch depth at 349 days after treatment. Soil treated at 2.0 lb ai/A contained pendimethalin residues of 0.17 ppm in the 0- to 3-inch depth and 0.50 ppm in the 3- to 6-inch depth at 349 days after treatment.

DISCUSSION:

1. Complete meteorological data were not provided. Rainfall and temperature data from May-October was provided, but the year of these data was not indicated. In addition, field test data, such as depth of water table, slope of test site, and irrigation practices were not reported.
2. Immediate posttreatment soil samples were not analyzed to confirm pendimethalin application rates.
3. The erratic recovery of pendimethalin (e.g. 75.3-142.0% for beets) and CL 202,347 (e.g. 71.5-120.7% for wheat) indicates considerable interference may have occurred in the analyses.
4. Although a 4.0 lb ai/A treatment was reported on the field data sheets; no results were reported in the lab data.
5. The methods used determined total pendimethalin residues or CL 202,347; total pendimethalin residues could not be characterized.
6. Complete soil characteristics, such as pH and CEC, were not provided.

Table 1. Pendimethalin residues and CL 202,347 (ppm) in soil and three rotational crops in plots treated with pendimethalin (3 lb/gal EC) at 1.5 and 2.0 lb ai/A the previous year.

Sample	Treatment rate (lb ai/A) 1973 + 1974	Days posttreatment	Pendimethalin residues	CL 202,347
Soil	0 + 0	--	--	--
Corn foliage	0 + 0	--	ND <sup>a</sup>	ND
Wheat foliage	0 + 0	--	ND	ND
Beet plant	0 + 0	--	ND	ND
Soil (0-3 inches)	1.5 + 0	349	0.14	--
(3-6 inches)	1.5 + 0	349	0.22	--
(6-9 inches)	1.5 + 0	349	ND	--
Soil (0-3 inches)	1.5 + 0	364	0.10	--
(3-6 inches)	1.5 + 0	364	0.22	--
Corn foliage	1.5 + 0	403	ND	ND
Wheat foliage	1.5 + 0	389	ND	ND
Beet plant	1.5 + 0	413	ND	ND
Soil (0-3 inches)	2.0 + 0	349	0.17	--
(3-6 inches)	2.0 + 0	349	0.50	--
(6-9 inches)	2.0 + 0	349	ND	--
Soil (0-3 inches)	2.0 + 0	364	0.17	--
(3-6 inches)	2.0 + 0	364	0.36	--
Corn foliage	2.0 + 0	403	ND	ND
Wheat foliage	2.0 + 0	389	ND	ND
Beet plant	2.0 + 0	413	ND	ND

<sup>a</sup> Not detected, detection limit is 0.05 ppm.

CASE GS0187      PENDIMETHALIN      STUDY 37      PM PM# 02/15/83

CHEM 108501      Pendimethalin

BRANCH EFB      DISC 30 TOPIC 05

FORMULATION 12 - EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 00106777-G      CONTENT CAT 01

Bohn, W., M. Moyer, J. Wyckoff, et al. 1974. Prowl (CL 92,553): Determination of CL 92,553 [N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine] and CL 202,347 [4-(1-ethylpropylamino)-2-methyl-3,5-dinitrobenzyl alcohol] residues in beets (foliage and roots), corn (foliage) and soybeans (foliage) and CL 92,553 in soil.

SUBST. CLASS =

DIRECT RVW TIME = 3      (MH) START-DATE      END DATE

REVIEWED BY: K. Patten and L. Lewis

TITLE: Staff Scientists

ORG: Dynamac Corp., Enviro Control Division, Rockville, MD

TEL: 468-2500

SIGNATURE: *K. Patten L. Lewis*

DATE: May 24, 1984

APPROVED BY:

TITLE:

ORG:

TEL:

SIGNATURE:

DATE:

CONCLUSIONS:Field Accumulation - Rotational Crops

1. This study is scientifically valid.
2. Neither pendimethalin residues (uncharacterized) nor 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) accumulated (<0.05 ppm) in the foliage of corn, soybean, oat, or beet (foliage and roots) plants grown for 48, 32, 37, and 72 days, respectively, in a silty clay loam soil treated the preceding year with pendimethalin (3 lb/gal EC). Maximum pendimethalin residues of 0.43 ppm were detected in the 0- to 9-inch depth of soil 345 days after application of pendimethalin (1-16 days after planting).
3. This study does not fulfill EPA Data Requirements for Registering Pesticides (1983) because field test data were not provided, immediate posttreatment and day of harvest soil samples were not analyzed, and a nonspecific analytical method was used.



### MATERIALS AND METHODS:

Three field plots (10 x 90 feet) of silty clay loam soil (17.2% sand, 54.6% silt, 28.2% clay, 1.7 organic matter) near London, Ohio, were planted to corn, oats, and red beets on April 27 and to soybeans on May 13, 1974. Two of the plots had been treated on May 16, 1973, with pendimethalin (Prowl, 3 lb/gal EC, American Cyanamid Co.) at 2.5 and 4.5 lb ai/A. Before the 1974 planting, the soil had been plowed and disced (10-inch depth). A third plot remained as an untreated control. Soil samples (0- to 3-, 3- to 6- and 6- to 9-inch depth) were obtained on April 26, 1974, 345 days after the pendimethalin treatment. Oat foliage was sampled June 3, corn and soybean foliage June 14, and beet foliage and roots July 8, 1974, 383, 394, and 418 days after the pendimethalin treatments, respectively.

Soil samples from a common plot and sampling date were combined and analyzed for total pendimethalin residues (pendimethalin and degradation products) using GC according to Method M-520, which is identical to Method M-437 (described in 00029034, Study 12) with the exception that soil (~25 g) was extracted in a 6:4:1 mixture of acidic methanol:water:soil. The detection limit was 0.05 ppm.

Corn samples from a common treatment were combined and analyzed for total pendimethalin residues and 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) in plant tissue using GC according to Methods M-458.1 and M-459.1, respectively (described in 00106777-C, Study 33). The detection limit for both M-458.1 and M-459.1 was 0.05 ppm. Recovery from fortified samples ranged from 87.5 to 126.7% for pendimethalin from 75.2 to 110.8% for CL 202,347.

Soybean samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-483, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the soybean foliage using GC according to Method M-531, which is identical to Method M-459.1 (described in 00106777-C, Study 33) with the exception that chloroform rather than hexane was used in the separatory funnel partitioning. The detection limit for both M-483 and M-531 was 0.05 ppm. Recovery from fortified samples ranged from 92.2 to 110.1% for pendimethalin and from 79.6 to 105.8% for CL 202,347.

Oat samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-538, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the plant tissue using GC accord-

ing to Method M-539, which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-538 and M-539 was 0.05 ppm. Recovery from fortified samples ranged from 88.9 to 119.3% for pendimethalin and from 73.1 to 116.5% for CL 202,347.

Beet samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-529, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the plant tissue using GC according to Method M-530 which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-529 and M-530 was 0.05 ppm. Recovery from fortified samples ranged from 75.3 to 142.0% for pendimethalin and from 73.1 to 119.4% for CL 202,347.

#### REPORTED RESULTS:

Neither pendimethalin residues nor CL 202,347 were detected (<0.05 ppm) in corn, soybeans, oats, or beets grown in the silty clay loam soil treated the previous year with pendimethalin at 1.5 or 4.5 lb ai/A (Table 1). Pendimethalin residues were not detected in the upper 9 inches of soil treated with pendimethalin at 1.5 lb ai/A. In soil treated with pendimethalin at 4.5 lb ai/A, pendimethalin residues of 0.41 ppm were recovered from the 0- to 3-inch depth, 0.68 ppm from the 3- to 6-inch depth, and 0.21 ppm from the 6- to 9-inch depth.

#### DISCUSSION:

1. Complete meteorological data including rainfall were not provided.
2. Two mechanical analyses of the soil were reported: 17.2% sand, 54.6% silt, 28.2% clay, a silty clay loam soil; and 42.2% sand 33.6% silt, 24% clay, a loam soil.
3. Field data but not laboratory analyses of a treated crop study was included in the report.
4. Field data reports were unintelligible because of the poor copies provided.
5. Soil in the control plot was not analyzed for pendimethalin residues.
6. Immediate posttreatment soil samples were not analyzed to confirm pendimethalin application rate.
7. The erratic recovery of pendimethalin from fortified beets (75.3-142.0%) indicates considerable interference may have occurred in the analyses.
8. The methods used determined total pendimethalin residues or CL 202,347; total pendimethalin residues could not be characterized.

Table 1. Pendimethalin residues and CL 202,347 (ppm) in soil and four rotational crops grown in plots treated with pendimethalin (3 lb/gal EC) at 1.5 and 4.5 lb ai/A the previous year.

Sample	Treatment rate (lb ai/A) 1973 + 1974	Days posttreatment	Pendimethalin residues	CL 202,347
Soil	0 + 0	--	--	--
Beet foliage	0 + 0	--	ND <sup>a</sup>	ND
Beet root	0 + 0	--	ND	ND
Oat foliage	0 + 0	--	ND	ND
Soil (0-3 inches)	1.5 + 0	345	ND	--
(3-6 inches)	1.5 + 0	345	ND	--
(6-9 inches)	1.5 + 0	345	ND	--
Beet foliage	1.5 + 0	418	ND	ND
Beet root	1.5 + 0	418	ND	ND
Corn foliage	1.5 + 0	394	ND	ND
Soybean foliage	1.5 + 0	394	ND	ND
Oat foliage	1.5 + 0	383	ND	ND
Soil (0-3 inches)	4.5 + 0	345	0.41	--
(3-6 inches)	4.5 + 0	345	0.68	--
(6-9 inches)	4.5 + 0	345	0.21	--
Beet foliage	4.5 + 0	418	ND	ND
Beet root	4.5 + 0	418	ND	ND
Corn foliage	4.5 + 0	394	ND	ND
Soybean foliage	4.5 + 0	394	ND	ND
Oat foliage	4.5 + 0	383	ND	ND

<sup>a</sup> Not detected, detection limit is 0.05 ppm.

CASE GS0187 PENDIMETHALIN STUDY 38 PM PM# 02/15/83

CHEM 108501 Pendimethalin

BRANCH EFB DISC 30 TOPIC 05

FORMULATION 00 - ACTIVE INGREDIENT

FICHE/MASTER ID 00106777-H CONTENT CAT 01

Bohn, W., J. Wyckoff, and G. Nzewi. 1974. Prowl (Cl 92,553): Determination of CL 92,553 [N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine] and CL 202,347 [4-([1-ethylpropyl]amino)-2-methyl-3,5-dinitro-benzyl alcohol] residues in corn (foliage) and soybeans (foliage) and CL 92,553 in soil..

SUBST. CLASS = S.

DIRECT RVW TIME = 3 (MH) START-DATE END DATE

REVIEWED BY: K. Patten

TITLE: Staff Scientist

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TEL: 468-2500

SIGNATURE: *K. Patten*

DATE: May 24, 1984

APPROVED BY:

TITLE:

ORG:

TEL:

SIGNATURE:

DATE:

CONCLUSION:Field Accumulation - Rotational Crops

This study is scientifically invalid because no soil samples were analyzed to confirm the application of pendimethalin to the soil. Additionally, this study would not fulfill EPA Data Requirements for Registering Pesticides (1983) because the test soil was not characterized, field test data including rainfall were not provided, and a nonspecific analytical method was used.

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MATERIALS AND METHODS:

A field plot (15 x 90 feet) of silt loam soil (soil not further characterized) near London, Ohio, was treated with pendimethalin (Prowl, 3 lb/gal EC, American Cyanamid Co.) at 2.0 lb ai/A on April 27, 1974, and planted to corn. On May 20, 1974, the plot was disced to destroy the corn and was replanted to corn and soybeans. Soybean foliage was sampled on June 13, 1974, 47 days after treatment, and corn foliage was sampled on July 7, 1974, 81 days after treatment.

Corn samples from a treatment date were combined and analyzed for total pendimethalin residues (pendimethalin and its degradates) and 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) in plant tissue using GC according to Methods M-458.1 and M-459.1, respectively (described in 00106777-C, Study 33). The detection limit for both M-458.1 and M-459.1 was 0.05 ppm. Recovery from fortified samples ranged from 87.5 to 126.7% for pendimethalin and from 75.2 to 110.8% for CL 202,347.

Soybean samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-483, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the soybean foliage using GC according to Method M-531, which is identical to Method M-459.1 (described in 00106777-C, Study 33) with the exception that chloroform rather than hexane was used in the separatory funnel partitioning. The detection limit for both M-483 and M-531 was 0.05 ppm. Recovery from fortified samples ranged from 92.2 to 110.1% for pendimethalin and from 79.6 to 105.8% for CL 202,347.

REPORTED RESULTS:

Neither pendimethalin residues nor CL 202,347 were detected (<0.05 ppm) in the foliage of corn and soybeans grown in soil treated 23 days prior to planting with pendimethalin at 2.0 lb ai/A.

DISCUSSION:

1. Field test data such as meteorological data, slope of the test site, depth of water table, and irrigation practices were not provided.
2. No supporting soil analyses are provided. There is no confirmation of the application of pendimethalin to the soil.
3. Complete soil characteristics, including organic matter content, pH, and CEC, were not provided.
4. The methods used determined total pendimethalin residues and CL 202,347; total pendimethalin residues could not be characterized.

Table 1. Pendimethalin residues and CL 202,347 (ppm) in corn and soybeans planted in a silt loam soil 23 days after application of pendimethalin (3 lb/gal EC) at 2.0 lb ai/A.

Sample	Days posttreatment	Days postplanting	Pendimethalin residues	CL 202,347
Corn foliage	81	58	ND <sup>a</sup>	ND
Soybean foliage	47	24	ND	ND

<sup>a</sup> Not detected, detection limit is 0.05 ppm.

CASE GS0187      PENDIMETHALIN      STUDY 39      PM PM# 02/15/83

CHEM 108501      Pendimethalin

BRANCH EFB      DISC 30 TOPIC 05

FORMULATION 12 - EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 00106777-I      CONTENT CAT 01

Potts, C., J. Wyckoff, and G. Nzewi. 1974. Prowl (CL 92,553): Determination of CL 92,553 [N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine] and CL 202,347 [4-([1-ethylpropyl]amino)-2-methyl-3,5-dinitro-benzyl] residues in beets (foliage and roots), corn (foliage), and wheat (foliage), and CL 92,553 in soil.

SUBST. CLASS = S.

DIRECT RVW TIME = 3      (MH) START-DATE      END DATE

REVIEWED BY: K. Patten

TITLE: Staff Scientist

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DATE: May 24, 1984

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CONCLUSIONS:Field Accumulation - Rotational Crops

1. This study is scientifically valid.
2. Neither pendimethalin residues (uncharacterized) nor 4-[1-(ethylpropyl)-amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) accumulated (<0.05 ppm) in the foliage of corn, wheat, or beet (foliage and roots) plants grown for 41 days in sandy loam soil treated the previous year with pendimethalin (3 lb/gal EC). In the upper 9 inches of soil 360 days after application (at planting) pendimethalin residues were <0.24 ppm.
3. This study does not fulfill EPA Data Requirements for Registering Pesticides (1983) because field test data including rainfall were not provided, the test soil was not completely characterized, a nonspecific analytical method was used, and immediate posttreatment and day of harvest soil samples were not analyzed.

MATERIALS AND METHODS:

Three field plots (8 rows x 100 feet) of a sandy loam soil (soil not further characterized) near Greeley, Colorado, were planted to corn, wheat, and red beets on May 7, 1974. Two of the plots had been treated on May 12, 1973, with pendimethalin (Prowl, 3 lb/gal EC, American Cyanamid Co.) at 1.5 and 6.0 lb ai/A. The third plot remained untreated. Soil samples (0- to 3-, 3- to 6-, and 6- to 9-inch depth) were obtained on May 7, 1974, 360 days after the pendimethalin treatment. Foliage samples from the corn, wheat, and beets, and root samples from the beets were obtained on June 17, 1974, 401 days after the pendimethalin treatment.

Soil samples from a common plot and sampling date were combined and analyzed for total pendimethalin residues (pendimethalin and degradates) using GC according to Method M-520, which is identical to Method M-437 (described in 00029034, Study 12) with the exception that soil (~25 g) was extracted in a 6:4:1 mixture of acidic methanol:water:soil. The detection limit was 0.05 ppm.

Corn samples from a common treatment were combined and analyzed for total pendimethalin residues (pendimethalin and its degradates) and 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) in plant tissue using GC according to Methods M-458.1 and M-459.1, respectively (described in 00106777-C, Study 33). The detection limit for both M-458.1 and M-459.1 was 0.05 ppm. Recovery from fortified samples ranged from 87.5 to 126.7% for pendimethalin and from 75.2 to 110.8% for CL 202,347.

Wheat samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-485, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the plant tissue using GC according to Method M-522, which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-485 and M-522 was 0.05 ppm. Recovery from fortified samples ranged from 76.4 to 133.7% for pendimethalin and from 71.5 to 120.7% for CL 202,347.

Beet samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-529, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the plant tissue using GC according to Method M-530, which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-529 and M-530 was 0.05 ppm. Recovery from fortified samples ranged from 75.3 to 142.0% for pendimethalin and from 73.1 to 119.4% for CL 202,347.



REPORTED RESULTS:

Neither pendimethalin nor CL 202,347 residues were detected (<0.05 ppm) in corn, wheat, and beet foliage, or beet roots grown in a sandy loam soil treated with pendimethalin at 1.5 or 6.0 lb ai/A (Table 1). In the soil treated with pendimethalin at 1.5 lb ai/A, pendimethalin residues of 0.11, 0.14, and 0.07 ppm were detected from the 0- to 3-, 3- to 6-, and 6- to 9-inch depths, respectively. In soil treated with pendimethalin at 6.0 lb ai/A, pendimethalin residues of 0.24 ppm were detected in the 0- to 3-inch depth and 0.12 ppm in the 6- to 9-inch depth. Pendimethalin residues were not detected in the 3- to 6-inch depth of soil treated at 6.0 lb ai/A.

DISCUSSION:

1. Complete meteorological data including rainfall were not reported.
2. Complete soil characteristics, such as pH, CEC, and organic matter content, were not provided.
3. Field data reports are unintelligible because of the poor copies provided.
4. The soil in the control plot was not analyzed for pendimethalin residues.
5. Immediate posttreatment soil samples were not analyzed to confirm pendimethalin application rates.
6. The erratic recovery of pendimethalin (e.g., 75.3 to 142.0% from beets) and CL 202,347 (e.g., 71.5 to 120.7% in wheat) suggests considerable interference may have occurred in the analyses.
7. The methods used determined total pendimethalin residues or CL 202,347; total pendimethalin residues could not be characterized.

Table 1. Pendimethalin residues and CL 202,347 (ppm) in soil and three rotational crops grown in a sandy loam soil treated with pendimethalin (3 lb/gal EC) at 1.5 and 6.0 lb ai/A the previous year.

Sample	Treatment rate (lb ai/A) 1973 + 1974	Days posttreatment	Pendimethalin residues	CL 202,347
Soil	0 + 0	--	--	--
Corn foliage	0 + 0	--	ND <sup>a</sup>	ND
Wheat foliage	0 + 0	--	ND	ND
Beet foliage	0 + 0	--	ND	ND
Beet roots	0 + 0	--	ND	ND
Soil (0-3 inches)	1.5 + 0	360	0.11	--
(3-6 inches)	1.5 + 0	360	0.14	--
(6-9 inches)	1.5 + 0	360	0.07	--
Corn foliage	1.5 + 0	401	ND	ND
Wheat foliage	1.5 + 0	401	ND	ND
Beet foliage	1.5 + 0	401	ND	ND
Beet roots	1.5 + 0	401	ND	ND
Soil (0-3 inches)	6.0 + 0	360	0.24	--
(3-6 inches)	6.0 + 0	360	ND	--
(6-9 inches)	6.0 + 0	360	0.12	--
Corn foliage	6.0 + 0	401	ND	ND
Wheat foliage	6.0 + 0	401	ND	ND
Beet foliage	6.0 + 0	401	ND	ND
Beet roots	6.0 + 0	401	ND	ND

<sup>a</sup> Not detected, detection limit is 0.05 ppm.

CASE GS0187      PENDIMETHALIN      STUDY 40      PM PM# 02/15/83

CHEM 108501      Pendimethalin

BRANCH EFB      DISC 30 TOPIC 05

FORMULATION 12 - EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 00106777-J      CONTENT CAT 01

Boughton, P., J. Wyckoff, and C. Kust. 1974. Prowl (CL 92,553): Determination of CL 92,553 [N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine] and CL 202,347 [4-([1-ethylpropyl]amino)-2-methyl-3,5-dinitro-benzyl alcohol] residues in wheat (foliage) and CL 92,553 in soil.

SUBST. CLASS = S.

DIRECT RVW TIME = 3 1/2 (MH) START-DATE      END DATE

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CONCLUSIONS:Field Accumulation - Rotational Crops

1. This study is scientifically valid.
2. Neither pendimethalin residues (uncharacterized) nor 4-[1-(ethylpropyl)-amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) accumulated (<0.05 ppm) in foliage of wheat grown for 32 days in a sandy loam soil treated the previous year with pendimethalin (3 lb/gal EC). In the 0- to 3-inch depth of soil at the time of planting, pendimethalin residues of ~0.11 ppm were detected. Pendimethalin residues were not detected below the 3-inch depth.
3. This study does not fulfill EPA Data Requirements for Registering Pesticides (1983) because the test soil was not completely characterized, immediate posttreatment and day of harvest soil samples were not analyzed, a nonspecific analytical method was used, and complete field test data were not provided.

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### MATERIALS AND METHODS:

Two field plots (24 x 230 feet) of a sandy loam soil (soil not further characterized) near Jackson, North Carolina, were planted to wheat and red beets on May 11, 1974. On May 9, 1973, one plot had been treated with pendimethalin (Prowl, 3 lb/gal EC, American Cyanamid Co.) at 0.5 lb ai/A, and the second plot with pendimethalin at 1.0 lb ai/A. The pendimethalin treatments were immediately incorporated to a depth of 3-4 inches. The soil was plowed and disced (depth not specified) before the soil was sampled (0- to 3-, 3- to 6-, and 6- to 9-inch depth) on May 1, 1974, 357 days after the pendimethalin treatment. Samples of the wheat foliage were obtained on June 12, 1974, 389 days after pendimethalin treatment. Red beets were not sampled because they were too small.


Soil samples from a common plot and sampling date were combined and analyzed for total pendimethalin residues (pendimethalin and degradates) using GC according to Method M-520, which is identical to Method M-437 (described in 00029034, Study 12) with the exception that soil (~25 g) was extracted in a 6:4:1 mixture of acidic methanol:water:soil. The detection limit was 0.05 ppm.

Wheat samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-485, which is identical to Method M-458.1 (described in 00106777-C, Study 33). 4-[1-(Ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202, 347) was determined in the plant tissue using GC according to Method M-522, which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-485 and M-522 was 0.05 ppm. Recovery from fortified samples ranged from 76.4 to 133.7% for pendimethalin and from 71.5% to 120.7% for CL 202,347.

### REPORTED RESULTS:

From May 1973 through May 1974, air temperatures ranged from 11 to 94 F, and ~56.8 inches of rainfall were received. Neither pendimethalin residues nor CL 202,347 were detected (detection limit 0.05 ppm) in wheat grown in sandy loam soil treated the previous year with pendimethalin at 0.5 and 1.0 lb ai/A (Table 1). Respectively, pendimethalin residues in the 0- to 3-inch depth of soil were 0.10 and 0.11 ppm, but were not detected below the 3-inch depth.

### DISCUSSION:

1. Complete soil characteristics, such as pH, CEC, and organic matter content, were not provided.
  2. Although the beets were reportedly too small to harvest at the same time as the wheat, there was no indication why the beets were not harvested at a later date.
- 

3. No data was reported from the control plot, although the field data retrieval forms indicated a control plot existed.
4. Immediate posttreatment soil samples were not analyzed to confirm pendimethalin application rates, although field data retrieval sheets indicated they were obtained.
5. The method used determined total pendimethalin residues and CL 202,347; total pendimethalin residues could not be characterized.
6. The erratic recovery of pendimethalin (e.g., 76.4-133.7%) and CL 202,347 (e.g., 71.5-120.7%) from fortified wheat samples indicates considerable interference may have occurred in the analyses.
7. Field test data, such as slope of the test site, depth of the water table, and irrigation practices, were not reported.

Table 1. Pendimethalin residues and CL 202,347 (ppm) in sandy loam soil and wheat grown in soil treated with pendimethalin (3 lb/gal EC) at 0.5 and 1.0 lb ai/A the previous year.

Sample	Treatment rate (lb ai/A)	Days posttreatment	Pendimethalin residues	CL 202,347
	1973 + 1974			
Soil (0-3 inches)	0.5 + 0	357	0.10	--
(3-6 inches)	0.5 + 0	357	ND <sup>a</sup>	--
(6-9 inches)	0.5 + 0	357	ND	--
Wheat foliage	0.5 + 0	389	ND	ND
Soil (0-3 inches)	1.0 + 0	357	0.11	--
(3-6 inches)	1.0 + 0	357	ND	--
(6-9 inches)	1.0 + 0	357	ND	--
Wheat foliage	1.0 + 0	389	ND	ND

<sup>a</sup> Not detected, detection limit is 0.05 ppm.

CASE GS0187      PENDIMETHALIN      STUDY 41      PM PM# 02/15/83

CHEM 108501      Pendimethalin

BRANCH EFB      DISC 30 TOPIC 05

FORMULATION 12 - EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 00106777-K      CONTENT CAT 01

Bohn, W., P. Boughton, J. Wyckoff, et al. 1974. Prowl (CL 92,553): Determination of CL 92,553 [N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine] and CL 202,347 [4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitro-benzyl alcohol] residues in cotton (foliage) and soybeans (foliage) and CL 92,553 in soil.

SUBST. CLASS = S.

DIRECT RVW TIME = 3      (MH) START-DATE      END DATE

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DATE: May 30, 1984

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DATE:

CONCLUSIONS:Field Accumulation - Rotational Crops

1. This study is scientifically valid.
2. Neither pendimethalin residues (uncharacterized) nor 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) accumulated (<0.05 ppm) in soybean or cotton foliage from plants grown for 33 days in a sandy clay loam soil treated the preceding 2 years with pendimethalin (3 lb/gal EC). In the 3- to 6-inch soil depth at the time of planting, pendimethalin residues were 0.25 ppm. No pendimethalin residues were detected at the 0- to 3-inch depth.
3. This study does not fulfill EPA Data Requirement for Registering Pesticides (1983) because the test soil was not completely characterized, field test data were insufficiently provided, a nonspecific analytical method was used, and immediate posttreatment and day of harvest soil samples were not analyzed.

MATERIALS AND METHODS:

Two field plots (13 x 30 feet) of Decatur sandy clay loam soil (soil not further characterized) near Town Creek, Alabama, were planted to cotton and soybeans on April 26, 1974. One plot had been treated on May 3, 1972, and again on May 4, 1973, with pendimethalin (Prowl, 3 lb/gal EC, American Cyanamid Co.) at 1.0 lb ai/A. Before the 1974 planting, the soil had been disced (depth unspecified). The second plot remained as an untreated control. Soil samples (0- to 3- and 3- to 6-inch depth) were taken on April 26, 1974, 357 days after the pendimethalin treatment. Cotton and soybean foliage were sampled on May 29, 1974, 390 days after the pendimethalin treatment and 33 days after planting.

Soil samples from a common plot and sampling date were combined and analyzed for total pendimethalin residues (pendimethalin and degradates) using GC according to Method M-520, which is identical to Method M-437 (described in 00029034, Study 12) with the exception that soil (~25 g) was extracted in a 6:4:1 mixture of acidic methanol:water:soil. The detection limit was 0.05 ppm.

Cotton samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-516, which is identical to Method M-458.1 (described in 00106777-C, Study 33), with the exception that after the acidic methanol:hexane partitioning, the hexane fraction was partitioned a second time in 25 ml acetonitrile:water (80:20) before evaporation to dryness. 4-[1-(Ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) was determined in the plant tissue using GC according to Method M-517, which is identical to Method M-459.1 (described in 00106777-C, Study 33) with the exception that chloroform rather than hexane was used in the separatory funnel partitioning. The detection limit for both M-516 and M-517 was 0.05 ppm. Recovery from fortified samples ranged from 71.2 to 90.1% for pendimethalin and 75.4 to 138.6% for CL 202,347.

Soybean samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-483, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the soybean foliage using GC according to Method M-531, which is identical to Method M-459.1 (described in 00106777-C, Study 33) with the exception that chloroform rather than hexane was used in the separatory funnel partitioning. The detection limit for both M-483 and M-531 was 0.05 ppm. Recovery from fortified samples ranged from 92.2 to 110.1% for pendimethalin and from 79.6 to 105.8% for CL 202,347.

REPORTED RESULTS:

From April 1972 through May 1974, air temperatures ranged from 15-97 F and ~155 inches of rainfall were received. Neither pendi-



methalin residues nor CL 202,347 were detected (<0.05 ppm) in soybeans or cotton grown in sandy clay loam soil treated the previous two years with pendimethalin at 1.0 lb ai/A (Table 1). Pendimethalin residues were not detected in the upper 3 inches of soil 357 days after the last pendimethalin application, but were found at 0.25 ppm at the 3- to 6-inch depth.

#### DISCUSSION:

- 1.. Field test data, such as irrigation practices, depth of water table, and slope of test site, were not provided.
2. Complete soil characteristics, such as pH, CEC, and organic matter content, were not provided.
3. Immediate posttreatment soil samples were not analyzed to confirm pendimethalin application rates.
4. The analytical methods used determined total pendimethalin residues or CL 202,347; total pendimethalin residues could not be characterized.
5. The erratic recovery of CL 202,347 (e.g. 75.4-138.6 for cotton) from fortified samples indicates considerable interference may have occurred in the analyses.

Table 1. Pendimethalin residues and CL 202,347 (ppm) in soil, cotton, and soybeans grown in plots treated with pendimethalin (3 lb/gal EC) at 1.0 lb ai/A the previous two years.

Sample	Treatment rate (lb ai/A)					Days posttreatment	Pendimethalin residues	CL 202,347
	1972	+	1973	+	1974			
Cotton foliage	0	+	0	+	0	--	ND <sup>a</sup>	ND
Soil (0-3 inches)	1	+	1	+	0	357	ND	--
(3-6 inches)	1	+	1	+	0	357	0.25	--
Cotton foliage	1	+	1	+	0	390	ND	ND
Soybean foliage	1	+	1	+	0	390	ND	ND

<sup>a</sup> Not detected, detection limit is 0.05 ppm.

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CASE GS0187 PENDIMETHALIN STUDY 42

PM PM# 02/15/83

CHEM 108501 Pendimethalin

BRANCH EFB DISC 30 TOPIC 05

FORMULATION 12 - EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 00106777-L CONTENT CAT 01

Bohn, W., C. Potts, P. Boughton, et al. 1974. Prowl (CL 92,553): Determination of CL 92,553 [N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine], and CL 202,347 [4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol] residues in cotton (foliage), soybeans (foliage), beets (foliage and roots) and wheat (foliage) and CL 92,553 in soil.

SUBST. CLASS = S.

DIRECT RVW TIME = 3 1/2 (MH) START-DATE END DATE

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DATE: June 1, 1984

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DATE:

CONCLUSIONS:Field Accumulation - Rotational Crops

1. This study is scientifically valid.
2. Neither pendimethalin residues (uncharacterized) nor 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) accumulated (<0.05 ppm) in cotton, soybean, wheat, or beet foliage, or in beet roots from plants grown for ~30 days in a loam soil treated the previous 2 years with pendimethalin (3 lb/gal EC) at 1.0 lb ai/A. At the time of planting, pendimethalin residues in the 0- to 3- and 3- to 6-inch soil depths were <0.13 ppm.
3. This study does not fulfill EPA Data Requirements for Registering Pesticides (1983) because the test soil was not completely characterized, field test data were insufficiently provided, a nonspecific analytical method was used, and immediate posttreatment and day of harvest soil samples were not analyzed.

MATERIALS AND METHODS:

Two field plots (333 ft<sup>2</sup>) of a loam soil (50% sand, 30% silt, 18% clay, 1.8% organic matter) near Cheneyville, Louisiana, were planted to cotton, soybeans, and beets on April 24, 1974, and to wheat on May 10, 1974. One plot had been treated with pendimethalin (Prowl, 3 lb/gal EC, American Cyanamid Co.) at 1.0 lb ai/A on May 11, 1972, and on May 23, 1973. Before the 1974 planting, the soil was plowed and disced (10 inch depth). The second plot remained untreated. Soil samples (0- to 3- and 3- to 6-inch depth) were obtained April 24, 1974, 338 days after the pendimethalin treatment. Cotton, soybean, and beet foliage and beet roots were sampled May 24, 1974, ~1 month after planting. Wheat foliage samples were taken on June 10, ~1 month after planting.

Soil samples from a common plot and sampling date were combined and analyzed for total pendimethalin residues (pendimethalin and degradates) using GC according to Method M-520, which is identical to Method M-437 (described in 00029034, Study 12) with the exception that soil (~25 g) was extracted in a 6:4:1 mixture of acidic methanol:water:soil. The detection limit was 0.05 ppm.

Cotton samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-516, which is identical to Method M-458.1 (described in 00106777-C, Study 33), with the exception that after the acidic methanol:hexane partitioning, the hexane fraction was partitioned a second time in 25 ml acetonitrile:water (80:20) before evaporation to dryness. CL 202,347 was determined in the plant tissue using GC according to Method M-517, which is identical to Method M-459.1 (described in 00106777-C, Study 33), with the exception that chloroform rather than hexane was used in the separatory funnel partitioning. The detection limit for both M-516 and M-517 was 0.05 ppm. Recovery from fortified samples ranged from 71.2 to 90.1% for pendimethalin and 75.4 to 138.6% for CL 202,347.

Soybean samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-483, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the soybean foliage using GC according to Method M-531, which is identical to Method M-459.1 (described in 00106777-C, Study 33) with the exception that chloroform rather than hexane was used in the separatory funnel partitioning. The detection limit for both M-483 and M-531 was 0.05 ppm. Recovery from fortified samples ranged from 92.2 to 110.1% for pendimethalin and from 79.6 to 105.8% for CL 202,347.

Wheat samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-485, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL

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202,347 was determined in the plant tissue using GC according to Method M-522, which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-485 and M-522 was 0.05 ppm. Recovery from fortified samples ranged from 76.4 to 133.7% for pendimethalin and from 71.5 to 120.7% for CL 202,347.

Beet samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-529, which is identical to Method M-458.1 (described in 00106777-C, Study 33). CL 202,347 was determined in the plant tissue using GC according to Method M-530, which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-529 and M-530 was 0.05 ppm. Recovery from fortified samples ranged from 75.3 to 142.0% for pendimethalin and from 73.1 to 119.4% for CL 202,347.

#### REPORTED RESULTS:

Neither pendimethalin residues nor CL 202,347 were detected (<0.05 ppm) in cotton, soybean, wheat, or beet foliage, or in beet roots grown in loam soil treated the previous two years with pendimethalin at 1.0 lb ai/A (Table 1). At the time of planting, pendimethalin residues were not detected in the soil at the 0- to 3-inch depth, but were detected in the 3- to 6-inch depth at 0.13 ppm.

#### DISCUSSION:

1. Field test data, such as meteorological data, irrigation practices, depth of water table, and slope of test site, were not reported.
2. The test soil reported to be a sandy silt loam is a loam according to the USDA soil textural classification system.
3. Soil in the control plot was not analyzed for pendimethalin residues.
4. Immediate posttreatment soil samples were not analyzed to confirm pendimethalin application rates.
5. The erratic recovery of pendimethalin (e.g. 75.3-142.0% for beets) and CL 202,347 (e.g. 75.4-138.6% for cotton) from fortified samples indicates considerable interference may have occurred in the analyses.
6. The analytical methods used determined total pendimethalin residues or CL 202,347; total pendimethalin residues could not be characterized.
7. Complete soil characteristics, such as pH and CEC, were not provided.

Table 1. Pendimethalin residues and CL 202,347 (ppm) in soil and four rotational crops grown in a loam soil treated with pendimethalin (3 lb/gal EC) at 1.0 lb ai/A the two previous years.

Sample	Treatment rate (lb ai/A)					Days posttreatment	Pendimethalin residues	CL 202,347
	1972	+	1973	+	1974			
Soil	0	+	0	+	0	--	--	--
Cotton foliage	0	+	0	+	0	--	ND <sup>a</sup>	ND
Soybean foliage	0	+	0	+	0	--	ND	ND
Beet foliage	0	+	0	+	0	--	ND	ND
Beet roots	0	+	0	+	0	--	ND	ND
Wheat foliage	0	+	0	+	0	--	ND	ND
Soil (0-3 inches)	1.0	+	1.0	+	0	338	ND	--
(3-6 inches)	1.0	+	1.0	+	0	338	0.13	--
Cotton foliage	1.0	+	1.0	+	0	368	ND	ND
Soybean foliage	1.0	+	1.0	+	0	368	ND	ND
Beet foliage	1.0	+	1.0	+	0	368	ND	ND
Beet roots	1.0	+	1.0	+	0	368	ND	ND
Wheat foliage	1.0	+	1.0	+	0	385	ND	ND

<sup>a</sup> Not detected, detection limit is 0.05 ppm.

CASE GS0187      PENDIMETHALIN      STUDY 43      PM PM# 02/15/83

CHEM 108501      Pendimethalin

BRANCH EFB      DISC 30 TOPIC 05

FORMULATION 12 - EMULSIFIABLE CONCENTRATE (EC)

FICHE/MASTER ID 00106777-M      CONTENT CAT 01  
Boughton, P., J. Wyckoff, C. Kust. 1974. Prowl (CL 92,553): Determination of  
CL 92,553 [N-(1-ethylpropyl)-3,4-dimethyl-2,6-dinitrobenzenamine] and CL 202,347  
[4-[(1-ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol] residues in wheat  
(foliage) and CL 92,553 in soil.

SUBST. CLASS = S.

DIRECT RVW TIME = 4      (MH) START-DATE      END DATE

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CONCLUSIONS:Field Accumulation - Rotational Crop

1. This study is scientifically valid.
2. Neither pendimethalin residues (uncharacterized) nor 4-[1-(ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) accumulated (<0.05 ppm) in wheat foliage from plants grown for ~4 months in a silty clay loam soil treated the preceding year with pendimethalin at 1.0 or 2.0 lb ai/A. Pendimethalin residues in the 0- to 3- and 3- to 6-inch soil depth of both treatments were <0.15 ppm on the day the wheat was harvested, 429 days after the pendimethalin application.
3. This study does not fulfill EPA Data Requirements for Registering Pesticides (1983) because the test soil was not completely characterized, field test data were insufficiently provided, a nonspecific analytical method was used, and immediate posttreatment and day of harvest soil samples were not analyzed.

### MATERIALS AND METHODS:

Three field plots (550 x 56 feet) of silty clay loam soil (<1% organic matter, soil not further characterized) near Somerton, Arizona, were planted to wheat on December 15, 1973, and irrigated at 2 to 3 week intervals. Two of the plots had been treated on February 14, 1973, with pendimethalin (Prowl, 3 lb/gal EC, American Cyanamid Co.) at 1.0 and 2.0 lb ai/A. Before the 1973 planting, the soils had been disced and plowed (>6 inch depth). A third plot remained untreated. Soil samples (0- to 3- and 3- to 6-inch depths) were obtained on April 18, 1974, 429 days after the pendimethalin treatment. Wheat foliage was sampled on the same date, ~4 months after planting.

Soil samples from a common plot and sampling date were combined and analyzed for total pendimethalin residues (pendimethalin and degradation products) using GC according to Method M-520, which is identical to Method M-437 (described in 00029034, Study 12) with the exception that soil (~25 g) was extracted in a 6:4:1 mixture of acidic methanol: water:soil. The detection limit was 0.05 ppm.

Wheat samples from a common treatment were combined and analyzed for total pendimethalin residues using GC according to Method M-485, which is identical to Method M-458.1 (described in 00106777-C, Study 33). 4-[1-(Ethylpropyl)amino]-2-methyl-3,5-dinitrobenzyl alcohol (CL 202,347) was determined in the plant tissue using GC according to Method M-522, which is identical to Method M-459.1 (described in 00106777-C, Study 33). The detection limit for both M-485 and M-522 was 0.05 ppm. Recovery from fortified samples ranged from 76.4 to 133.7% for pendimethalin and from 71.5 to 120.7% for CL 202,347.

### REPORTED RESULTS:

From February 1973 through May 1974, air temperatures ranged from 31-110 F and ~2.6 inches of rainfall were received (no additional data provided).

Neither pendimethalin residues nor CL 202,347 were detected (<0.05 ppm) in wheat grown in silty clay loam soil treated the previous year with pendimethalin at 1.0 or 2.0 lb ai/A (Table 1). Pendimethalin residues in soil (0- to 3- and 3- to 6-inch depths) at the time of harvest were <0.15 ppm in both treatments.

### DISCUSSION:

1. The soil in the control plot was not analyzed for pendimethalin residues.
2. Immediate posttreatment soil samples were not analyzed to confirm pendimethalin application rates.



3. Complete soil characteristics, such as pH and CEC, were not provided.
4. The erratic recovery of pendimethalin (76.4 to 133.7%) and CL 202,347 (71.5 to 120.7%) from fortified samples of wheat foliage indicates considerable interference may have occurred in the analyses.
5. Field test data, such as irrigation practices, depth of water table, and slope of test site, were not reported.
6. The analytical methods used determined total pendimethalin residues or CL 202,3471; total pendimethalin residues could not be characterized.

Table 1. Pendimethalin residues and CL 202,347 (ppm) in soil and wheat grown in plots treated with pendimethalin (3 lb/gal EC) at 1.0 and 2.0 lb ai/A the previous year.

Sample	Treatment rate (lb ai/A)	Days posttreatment	Pendimethalin residues	CL 202,347
	1973 + 1974			
Soil	0 + 0	--	--	--
Wheat foliage	0 + 0	--	ND <sup>a</sup>	ND
Soil (0-3 inches)	1.0 + 0	429	0.15	--
(3-6 inches)	1.0 + 0	429	0.07	--
Wheat foliage	1.0 + 0	429	ND	ND
Soil (0-3 inches)	2.0 + 0	429	0.10	--
(3-6 inches)	2.0 + 0	429	0.10	--
Wheat foliage	2.0 + 0	429	ND	ND

<sup>a</sup> Not detected, detection limit is 0.05 ppm.

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