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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

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OFFICE OF PREVENTION,
PESTICIDES AND
TOXIC SUBSTANCES

May 9, 2001

Memorandum

SUBJECT: Review of "Determination of Transferable Residues in Turf Treated with Pendulum® 2G or Pendulum® WDG Herbicide" MRID No.449699-01.
DP Barcode: D274769.

FROM: Shanna Recore, Industrial Hygienist
Reregistration Branch II
Health Effects Division (7509C)

Shanna Recore 5/10/2001

THRU: Al Nielsen, Branch Senior Scientist
Reregistration Branch II
Health Effects Division (7509C)

Al Nielsen 5/10/2001

TO: Mark Perry, Chemical Review Manager
Special Review and Reregistration Division (7508W)

Attached is a review of the turf dislodgeable foliar residue data submitted by American Cyanamid Company (ACCO). This review was completed by Versar, Inc. on December 22, 2000, under supervision of HED. It has undergone secondary review in HED and has been revised to reflect Agency policies.

Executive Summary

The data collected reflecting the dissipation of pendimethalin from treated turf meet most of the criteria specified by the U.S. Environmental Protection Agency's (US-EPA) OPPTS Series 875, Occupational and Residential Exposure Test Guidelines, Group B: Postapplication Exposure Monitoring Test Guidelines, 875.2100, Transferable Residue Dissipation: Lawn and Turf. The data are of sufficient scientific quality to be used to determine turf transferable residue (TTR) dissipation.

Summary

In this study, two formulations of pendimethalin, Pendulum® 2G (granular) and Pendulum® WDG (water dispersible granular), were applied once, at the maximum application rate, 3.0 lbs a.i./acre, to turf in three geographical locations: California, Pennsylvania, and Florida. Triplicate transferable residue (TTR) samples were collected using the modified California Cloth Roller technique. Samples were collected prior to treatment and at intervals from 0 to 14 days after treatment. Control and field fortified recovery samples were collected prior to treatment and 3 and 7 days after treatment. Samples were analyzed for pendimethalin only.

All of the field Pendulum® 2G residue levels were below the limit of quantitation (LOQ), at all sampling intervals at all sites, during the 14 days following treatment, with the exception of 7 days after treatment when residues averaged 17.5 ng/cm² at the Pennsylvania site. This spike occurred 1 day after mowing and 11 days after a rainfall.

In California, the maximum Pendulum® WDG residue occurred 4 hours after treatment and decreased to below the LOQ by 14 days after treatment. In Pennsylvania, the maximum Pendulum® WDG residue occurred immediately after treatment and decreased to below the LOQ by 7 days after treatment. In Florida, the maximum Pendulum® WDG residue occurred on 1 day after treatment and decreased to below the LOQ by 14 days after treatment for one of the three samples. The maximum Pendulum® WDG residue levels varied significantly from site to site: 1,280 ng/cm² in California, 846 ng/cm² in Pennsylvania, and 608 ng/cm² in Florida. In addition, a significant number (approximately 50 percent) of Pendulum® WDG TTR samples exceeded the high field fortification level of 718 ng/cm² in California, indicating that the high fortification level was not well chosen for the California data set. The following dissipation half life values were obtained: 1.35 days ($R^2 = 0.907$) for California; 1.44 days ($R^2 = 0.985$) for Pennsylvania; and 2.17 days ($R^2 = 0.940$) for California.

Conclusion

The turf transferable residue study completed in support of the regulatory requirements contained the following omissions and flaws with respect to Series 875 Group B Postapplication Exposure Monitoring Test Guidelines. The most important discrepancies and issues of concern include: (1) agronomy practices and maintenance pesticide applications (past and present), and weather data were not conducted as per the GLP regulations at any of the test sites; (2) a significant number (approximately 50 percent) of the Pendulum® WDG TTR samples exceeded the high field fortification level of 718 ng/cm² in California, indicating that the highest

fortification level was not well chosen for the California data set; (3) there was no discussion of potential breakdown products or contaminants of concern in the pendimethalin-containing formulations used and only pendimethalin residues were analyzed; (4) the product labels state that a repeat application after 6-8 weeks may be applied, if necessary, and depending on the initial application rate, but only one application of each formulation was applied in this study (5) climate conditions during the course of the study are not known because rainfall information was not available in the report for California and data presented are for 1997 and not 1998, when the field portion of this study was conducted; (6) detailed information on the validation study or the analytical methodology employed in the study is not provided in the report and, in addition, the analytical method was developed during the same time period (May 1998) that the study was initiated in California and Pennsylvania; and (7) a separate control plot was not established at any of the test sites, however, control samples of exposed cloth were collected prior to treatment and at two sampling intervals using pristine cotton sheeting.

The data collected in this study are acceptable, however, transfer efficiencies of less than 0.5% for granular formulations and less than 1% for liquid formulations should not be used with the revised Transfer Coefficients, as listed in Science Advisory Council for Exposure Policy 11 (February 22, 2001), since these revised TCs are based on TTR transfer efficiencies of ~1-5% (transfer efficiency = residue found on cloth/study application rate). The ORETF is generating transfer coefficients to be used with data with lower transfer efficiency.

The transfer efficiency that resulted from the study on Pendulum 2G was 0.0064% at all three sites. So these results will not be used by HED until the ORETF generates appropriate dermal exposure transfer coefficients. The transfer efficiencies for Pendulum WDG were above 1% at all three sites and HED will use the results in the RED.

HED would like the registrant to review our list of issues and respond appropriately.

MEMORANDUM

TO: Christina Jarvis

cc: 110082.1000.001.01

FROM: Diane Forrest/Kathy Coon

DATE: December 22, 2000

SUBJECT: Review of CL 92553 (*Pendimethalin*): *Determination of Transferable Residues in Turf Treated with Pendulum® 2G or Pendulum® WDG Herbicide* - MRID #449699-01

This report reviews *CL 92553 (Pendimethalin): Determination of Transferable Residues in Turf Treated with Pendulum® 2G or Pendulum® WDG Herbicide*, submitted by American Cyanamid Company in response to the U.S. Environmental Protection Agency's (U.S. EPA) Data Call-In (DCI) notice issued March 3, 1995 and amended February 10, 1998. The requirements for this study are specified by the U.S. EPA's OPPTS Series 875, Occupational and Residential Exposure Test Guidelines, Group B: Postapplication Exposure Monitoring Test Guidelines, 875.2100, Transferable Residue Dissipation: Lawn and Turf. The following information may be used to identify the study:

Title:	<i>CL 92553 (Pendimethalin): Determination of Transferable Residues in Turf Treated with Pendulum® 2G or Pendulum® WDG Herbicide</i> 242 pages		
Sponsor:	American Cyanamid Company P.O. Box 400 Princeton, NJ 08543		
Field Study Test Sites:	Tami Belcher Excel Research Services, Inc. 3021 W. Dakota, Suite 110 Fresno (Madera), CA 93722	Tim White Crop Management Strategies P.O. Box 510 Hereford (Germansville), PA 18056	Wayne Currey Weed Systems, Inc. 8168 Alderman Road Melrose, FL 32666
Analytical Laboratories	Terry Bixler, Principal Analyst Maxim Technologies, Inc. 140 Telegraph Road Middleport, NY 14105	Gerald Picard, Group Leader, and Benjamin Guzman, Principal Analyst American Cyanamid Company P.O. Box 400 Princeton, NJ 08543	
Study Director and Author:	Andrew Garrett Field Residue Sciences American Cyanamid Company P.O. Box 400 Princeton, NJ 08543		
Report Date:	October 19, 1999		
Identifying Codes:	MRID #449699-01; Study Numbers: RES 99-076, RES 99-077, RES 99-078		

EXECUTIVE SUMMARY

This report reviews a transferable turf residue (TTR) study submitted by American Cyanamid Company (ACCO) in response to the U.S. Environmental Protection Agency's (U.S. EPA) Data Call-In (DCI) notice issued March 3, 1995, and amended February 10, 1998. The herbicide pendimethalin was applied using two different formulations to turf in three geographical locations: California, Pennsylvania, and Florida. Both a granular formulation, Pendulum® 2G containing 2.0 percent pendimethalin, and a water dispersible granular formulation, Pendulum® WDG containing 60 percent pendimethalin, were applied at all three locations. This study was conducted to determine the levels of pendimethalin residues that can be transferred from turf to 100 percent cotton cloth material following one application of the test substance at a maximum label rate (3.0 pounds ai/acre).

Triplicate TTR samples were collected from two treated plots in Madera, California (May 29 - June 12, 1998), Germansville, Pennsylvania (May 14-29, 1998), and Melrose, Florida (November 7-21, 1998). The sampling was performed prior to treatment, 0.1 hours (immediately after), 4 and 8 hours after application, and at 1, 3, 5, 7 and 14 days after treatment (DAT) at each test site. The modified California Roller Technique was used to collect the samples. Control and field fortified recovery samples were collected prior to treatment and in DAT-3 and DAT-7. Each plot contained three replicates subplots with 10 sampling areas in each replicate. All samples were collected randomly.

All of the field Pendulum® 2G residue levels were below the limit of quantification (LOQ), at all sampling intervals at all sites, during the 14 days following treatment, with the exception of DAT-7 when residues averaged 17.5 ng/cm² at the Pennsylvania site. This spike occurred 1 day after mowing and 11 days after a rainfall.

In California, the maximum Pendulum® WDG residue occurred 4 hours after treatment and decreased to below the LOQ by DAT-14. In Pennsylvania, the maximum Pendulum® WDG residue occurred immediately after treatment (DAT-0) and decreased to below the LOQ by DAT-7. In Florida, the maximum Pendulum® WDG residue occurred on DAT-1 and decreased to below the LOQ by DAT-14 for one of the three samples. The maximum Pendulum® WDG residue levels varied significantly from site to site. The highest level was reported in California (1,280 ng/cm²). The maximum level reported in Florida (608 ng/cm²) was only approximately 50 percent of the California maximum. In addition, a significant number (approximately 50 percent) of Pendulum® WDG TTR samples exceeded the high field fortification level of 718 ng/cm² in California, indicating that the high fortification level was not well chosen for the California data set.

American Cyanamid Company (ACCO) averaged triplicate TTR values (Pendulum® WDG) at each sampling interval from each test site. Linear regressions were conducted plotting time

sampled versus the logarithm (base 10) of the pendimethalin residue values to show the dissipation of the residue over the period. ACCO considered all data from DAT-0 through DAT-7 or 14 samples when residues were below the LOQ.

ACCO's calculated dissipation half-lives and correlation coefficients were as follows:

- California - 1.67 days ($R^2 = 0.967$)
- Pennsylvania - 1 day ($R^2 = 0.941$)
- Florida - 2.23 days ($R^2 = 0.941$)

Versar used individual TTR values (Pendulum® WDG), not averages, in conducting linear regressions on the three data sets. Only TTR values above the LOQ were included with one exception. When analyzing the data, a value of one-half of the LOQ was used for data points where TTR values were below LOQ. This occurred in the Florida data set. The linear regressions were conducted using the natural logarithm of TTR values processed by Microsoft's Excel 97®.

Versar's calculated dissipation half-lives and correlation coefficients were as follows:

- California - 1.35 days ($R^2 = 0.907$)
- Pennsylvania - 1.44 days ($R^2 = 0.985$)
- Florida - 2.17 days ($R^2 = 0.940$)

The study was in compliance with the major technical aspects of OPPTS Series 875 guidelines. The most important issues of concern are identified below:

- Agronomy practices and maintenance pesticide applications (past and present), and weather data were not conducted as per the GLP regulations at any of the test sites.
- The maximum Pendulum® WDG residue levels varied significantly from site to site. The highest level was reported in California (1,280 ng/cm²). The maximum level reported in Florida (608 ng/cm²) was only approximately 50 percent of the California maximum.
- A significant number (approximately 50 percent) of the Pendulum® WDG TTR samples exceeded the high field fortification level of 718 ng/cm² in California, indicating that the high fortification level was not well chosen for the California data set.
- There was no discussion of potential breakdown products or contaminants of concern in the pendimethalin-containing formulations used, although there are regulated metabolites. Only pendimethalin residues were analyzed.

- The product labels state that a repeat application after 6-8 weeks may be applied, if necessary, and depending on the initial application rate. Only one application of each formulation was applied in this study.
- Rainfall information was not available in the report for California. Data presented are for 1997 and not 1998, when the field portion of this study was conducted. Because of this error, climate conditions during the course of the study are not known.
- Detailed information on the validation study or the analytical methodology employed in the study is not provided in the report. In addition, the analytical method was developed during the same time period (May 1998) that the study was initiated in California and Pennsylvania.
- A separate control plot was not established at any of the test sites. However, control samples of exposed cloth were collected prior to treatment and at two sampling intervals using pristine cotton sheeting.

STUDY REVIEW

Study Background

This report reviews a transferable turf residue (TTR) study submitted by American Cyanamid Company (ACCO) in response to the U.S. Environmental Protection Agency's (U.S. EPA) Data Call-In (DCI) notice issued March 3, 1995, and amended February 10, 1998. Pendimethalin, CAS No. 40487-42-1, is the active ingredient (ai) in Pendulum® 2G and Pendulum® WDG, the herbicide formulations applied in this study. Pendulum® 2G is a granular formulation containing 2.0 percent pendimethalin; Pendulum® WDG is a water dispersible granule formulation containing 60 percent pendimethalin. Pendimethalin provides preemergence control of most annual grasses and some broadleaf weeds as they germinate in turfgrass. The objective of this study was to determine the levels of pendimethalin residues that can be transferred from turf to 100 percent cotton cloth material following one application of the test substance at a maximum rate and minimum volume of carrier specified on the label.

The study was conducted at three geographical locations. All field and analytical operations were coordinated and overseen by ACCO. On-site field operations were conducted by Excel Research Services, Inc. in Madera, California; Crop Management Strategies (CMS) in Germansville, Pennsylvania; and Weed Systems, Inc. in Melrose, Florida. All cloth samples were analyzed by Maxim Technologies, Inc. in Middleport, New York.

TTR sampling was performed prior to treatment and 0.1 hour, 4 hours, 8 hours, 1 day, 3 days, 5 days, 7 days, and 14 days after treatment (DAT) utilizing the Modified California Roller Technique. Samples were collected between May 29 and June 12, 1998, in California; between May 14 and May 29, 1998, in Pennsylvania; and between November 7 and November 21, 1998, in Florida. Samples were stored frozen between 67 and 104 days before analysis.

Test Plots

The test sites were set up at Madera, California (Madera County), at Germansville, Pennsylvania (Lehigh County), and at Melrose, Florida (Putnam County) to represent the "commercial use, climatic conditions, and turf types expected in the intended-use areas." Two turf types were treated: (1) fescue, grown on the test sites since 1996 and 1991 in California and Pennsylvania, respectively, and (2) bahiagrass, grown on the test site in Florida since 1995.

The test site in California consisted of two treated plots with three replicates each (10 x 20 feet). A buffer of 8 feet was used to separate the replicates and a buffer of 250 feet was used to separate the two treatment plots. The test site had a slope of less than 1 percent. A control plot was not established; however, control samples of exposed cloth were collected before treatment and at two sampling intervals using pristine cotton sheeting. No soil preparation or additional cultivation

was performed on the plots. The treated plots were mowed to a height of 2 inches one day prior to application of the test substances and 7 days after application. The grass clippings were bagged and removed after each mowing. During the course of the study, no fertilizer was applied to the treated plots.

The test site in Pennsylvania consisted of two treated plots with three replicates each (3 x 50 feet). A buffer of 3 feet was used to separate the replicates and a buffer of 15 feet was used to separate the two treatment plots. The test site had a slope of 2 to 4 percent. A control plot was not established; however, control samples of exposed cloth were collected before treatment and at two sampling intervals using pristine cotton sheeting. No soil preparation or additional cultivation was performed on the plots. The treated plots were mowed to a height of 3 inches three days prior to application of the test substances, to a height of 2 inches the day prior to application, and to a height of 2.5 inches 6 days after application. The grass clippings were bagged and removed after each mowing, except on the mowing three days prior to application. Fertilizer was applied to the treated plots approximately 1 month prior to application of the test substances.

The test site in Florida consisted of two treated plots with three replicates each (10 x 35 feet). A buffer of 3 feet was used to separate the replicates and a buffer of 10 feet was used to separate the two treatment plots. The test site had a slope of 2 percent. A control plot was not established; however, control samples of exposed cloth were collected prior to treatment and at two sampling intervals using pristine cotton sheeting. No soil preparation or additional cultivation was performed on the plots. The treated plots were mowed 1 day prior to application to a height of 3 inches. Previous mowings had occurred 12 days and 5 days prior to the application date to a height of 3 to 3.5 inches. Fertilizer was applied at the earliest mowing.

Pesticide Use History

The report states that no maintenance pesticides were applied to any of the test sites during the course of the study. Historical maintenance practices and pesticides use data were not provided in the report. The Statement of GLP Compliance prepared for each test site states that "agronomic practices and maintenance pesticide applications (past and present) were not conducted as per the GLP regulations."

Materials and Equipment

No pesticide labels were provided for review in the report. For the purpose of this review, Versar obtained label information for Pendulum® 2G (EPA Reg. No. 241-375) and Pendulum® WDG (EPA Reg. No. 241-340) from EPA's 1998 Pesticide Product Label System (PPLS). Both the granular and water dispersible granular product were used at all test sites.

According to the product labels reviewed by Versar, the maximum application rate for Pendulum® 2G is 150 pounds of formulated product per acre of turfgrass (either cool season or warm season) or 3.0 pounds ai/acre. The label recommends that Pendulum® 2G be applied with a calibrated spreader to well established turf grass prior to weed germination in the spring. The application should be followed by a light watering to obtain best results. The efficacy of Pendulum® 2G will improve if the application is followed by ½-inch of rainfall or its equivalent in sprinkler irrigation within 30 days. A repeat application of 75 pounds per acre (1.5 pounds ai/acre) may be made 6 to 8 weeks later, if necessary. For cool season grasses, this repeat application is allowed only if no more than 100 pounds per acre was applied initially. No such restrictions exist for warm season grasses. In this study, one application of Pendulum® 2G was applied using a granular applicator at all three test sites in California, Pennsylvania, and Florida. The report specified the type of applicator used only at the Florida test site (Vigoro Model VHD 125 Hand Spreader). In California, the actual rate of application was determined by the application pass time. The actual application rate at this test site was 2.99 pounds ai/acre. In Pennsylvania and Florida, the actual rate of application was determined by weighing the amount of test substance in the applicator hopper prior to and after application. The actual application rates in Pennsylvania and Florida were 3.15 pounds ai/acre and 3.0 pounds ai/acre, respectively.

The maximum application rate for Pendulum® WDG is 5.0 pounds of formulated product per acre of turfgrass (either cool season or warm season) or 3.0 pounds ai/acre. The label recommends that Pendulum® WDG be applied with calibrated ground equipment in at least 40 gallons of water per acre to well established turfgrass prior to weed germination in the spring. Low pressure (e.g., 20-40 pounds per square inch (psi)) sprayers are recommended. The efficacy of Pendulum® WDG will improve if the application is followed by ½-inch of rainfall or its equivalent in sprinkler irrigation. A repeat application of 5.0 pounds per acre may be applied after 6 to 8 weeks to warm season grasses, if necessary. A single repeat application of up to 2.5 pounds per acre may be applied to cool season grasses, but only if no more than 3.4 pounds per acre was applied during the first application. In this study, one application of Pendulum® WDG was applied using a calibrated (i.e., volume/time) 40 psi and 21 psi tractor mounted sprayer at the California and Pennsylvania test sites, respectively. A calibrated 18 psi back pack sprayer was used at the Florida test site. The actual application rates ranged from 3.0 to 3.1 lbs ai/acre in a spray volume of 40.4 to 41.2 gallons per acre at the three test sites.

Meteorology

The Statement of GLP Compliance prepared for each test site states that “weather data were not collected per the GLP regulations.” Air temperature readings, relative humidity, wind speed and direction, and soil moisture and temperature were summarized in the report for the day of application at each test site. However, Versar noted problems with the daily weather data presented in Appendix C of each site report.

In California, the report states that daily weather data were collected from an on-site Excel Weather Station, and complete weather data are presented in Appendix C. However, the data presented in this appendix are for 1997 and not 1998, when the field portion of this study was conducted. Because of this error, climatic conditions during the course of the study are not known. (Versar is reasonably certain this was not a simple typographical error.) The climatic conditions reported for the test site on the day of application show an air temperature of 76°F, moist soil, relative humidity of 68 percent, and winds 1 to 2 mph. Supplemental irrigation (type was not identified) was used four times during the course of the study. Approximately 0.5 inch of irrigation was applied on DAT-4 and DAT-5 and approximately 1 inch of irrigation was applied on DAT-7 and DAT-12. It is not known if the irrigation applied on DAT-5 and DAT-7 was applied before or after sampling was completed for that day.

In Pennsylvania, daily weather data (temperature and rainfall) were collected from an on-site CMS Weather Station (Appendix C). No other daily weather data were presented. A total of 0.61 inches of rain fell over a period of 14 days after application. This included 0.49 inches on DAT-14, the final day of sampling. Minimum air temperatures during the course of the study ranged from 46°F to 60°F; maximum air temperatures ranged from 69°F to 98°F. Climatic conditions reported for each test plot at the test site on the day of application show air temperatures of 74°F and 86°F, moist surface soils, relative humidities of 47 and 54 percent, and winds 1 to 4 mph. Supplemental irrigation was not used at the Pennsylvania test site.

In Florida, daily weather data (rainfall only) were collected on-site from a Weed Systems Weather Station. Other daily weather data were collected from the University of Florida Agronomy Department and NOAA Cooperative Weather Station, located 18 miles from the test site in Gainesville, Florida. A total of 0.32 inches of rain fell over a period of 14 days after application (Appendix C). All of this rainfall occurred on DAT-12. No rain fell on any of the sampling days. Minimum air temperatures during the course of the study ranged from 38°F to 67°F; maximum air temperatures ranged from 68°F to 80°F. Climatic conditions reported for each test plot on the day of application show air temperatures of 67°F and 71°F, moist soils, relative humidities of 51 and 55 percent, and winds 1 to 3 mph. Prior to the date of application (12 days and 5 days prior), the test plots were sprinkler irrigated with 0.75 and 0.30 inches of on-site well water, respectively. No further supplemental irrigation was used at the Florida test site.

Sampling of Turf Transfer Residue Samples

Sampling was performed prior to treatment (1 day prior in Pennsylvania and just before application in California and Florida) and 0.1 hours (immediately after), at 4 hours and 8 hours, and on DAT-1, 3, 5, 7, and 14 at each test site. The Modified California Roller Technique was used. Control and field fortified recovery samples were collected prior to treatment and on DAT-3 and DAT-7. Triplicate samples were collected at each sampling interval. Details on the sampling technique were not provided in any of the study site reports. However, no deviations from this

standardized technique were noted. Each plot contained three replicate subplots with 10 sampling areas in each replicate. Samples were collected randomly, with no area being sampled more than once. The categorization scheme was determined by computer in California and Pennsylvania. This information was not reported for the Florida test site. The cotton cloth samples were folded and placed in clean 1-gallon containers. All the samples were stored in freezers within 4 hours of sampling in California and Pennsylvania and within 1 hour in Florida.

QA/QC

Sample Handling and Storage

At the test sites, all of the cotton cloth samples were placed in freezers within 1 to 4 hours of sampling, where they remained until shipment. Daily freezer temperatures were monitored and the temperature of the storage freezers remained below freezing for the sample storage period. All samples were shipped from the test sites within 3 to 19 days of the last day of sampling.

Control and treated cotton cloth samples were packed separately in corrugated cardboard boxes. All boxes were appropriately labeled. The samples were then transported frozen to ACCO in Princeton, New Jersey, via a freezer truck. Upon arrival at ACCO, having been anywhere from <1 to 27 days in transport, the samples were placed in a walk-in freezer maintained at $\leq 10^{\circ}\text{C}$ for 21 to 42 days until they were prepared for analysis. Samples were then shipped via Federal Express to Maxim Technologies in Middleport, New York, for analysis.

Tank Mix and Product Analyses

On the day of application, samples of the test substances (Pendulum® 2G and WDG) and a sample of the Pendulum® WDG tank mix were collected at all three test sites. The samples were placed in a freezer 1 to 4 hours after collection, where they remained until shipment to ACCO in Princeton, New Jersey, via freezer truck. The product and tank mix samples were packed separately from the field TTR samples in styrofoam containers. Samples were analyzed to monitor the stability of each test substance from the time of characterization to application. The analyses were performed at ACCO (For the California data set, this information was provided in the sample tracking summary. Duplicate analyses of the product samples, collected at the three test sites, determined that the formulations contained from 1.98 to 2.01 percent (Pendulum® 2G) and from 62.20 to 62.89 percent (Pendulum® WDG) active ingredient. Results of the tank mix analyses show that the concentration of product in the samples of the tank mix averaged 93 percent of expected in California, 105 percent of expected in Pennsylvania, and 87 percent of expected in Florida.

Deposition Plates

Deposition plate samples were not prepared.

Analytical Methodology

A copy of the analytical methodology used by Maxim Technologies was not included for review in the study report. The cotton sheet samples were analyzed using American Cyanamid Method M 3208 (Draft Date 5/19/98) entitled *CL 92553 (pendimethalin): GC Method for the Determination of CL 92553 Residues on Cotton Cloth*. The method was validated at Maxim Technologies, Inc. and the data were reported in American Cyanamid Study Number PR98PT05 (Maxim Study No. A011.279). The method was developed during the same time period (May 1998) that the study was initiated in California and Pennsylvania. The average recovery from the validation study from fortified cotton sheets was 94 percent, with a standard deviation of ± 8 percent (N=8). No additional information on the validation study was provided in this report.

Samples were analyzed using Hewlett Packard 5890 gas chromatograph equipped with a nitrogen/phosphorus detector. The run time and retention time were 16.8 and 7.6 minutes, respectively. A complete listing of instrument conditions is provided in the report. Four analytical standards were used for the analyses and standard linearity curves were generated. Regression statistics indicate an excellent correlation ($R^2 = 0.99$). Representative chromatograms of pendimethalin standards and fortified control cotton cloth samples are provided in the report. Chromatograms show good peak separation and sharpness of peaks.

One minor modification made to Method 3208 was reported in the site reports for California and Pennsylvania. Cotton sheets for control and fortification samples were extracted in 1-gallon glass jars instead of 1-gallon paint cans. In addition, the residues were reconstituted in 1.0 mL of ethyl acetate.

Limit of Detection (LOD) and Limit of Quantification (LOQ)

For this study, the report states that the limit of quantitation (LOQ) was 4.31 ng/cm² for all study sites. The limit of detection (LOD) varied and was approximately 0.75 ng/cm² for the California samples, 0.666 ng/cm² for the Pennsylvania samples, and 0.639 ng/cm² for the samples collected in Florida.

Laboratory Recovery

Concurrent laboratory fortification samples containing pendimethalin were analyzed with each set of field samples. Four or five fortification levels were prepared, but only one to three levels were analyzed with each set of samples. For the analysis of the California samples, fortification levels were 4.31, 8.61, 17.9, 718, and 1,525 ng/cm²; Pennsylvania samples were 4.31, 8.61, 17.9, 718, and 1,005 ng/cm²; and Florida samples were 4.31, 17.9, 718, and 1,005 ng/cm². The concurrent recoveries averaged 104 ± 5.5 (N=16) percent for California, 101 ± 5.6 (N=16) percent for

Pennsylvania, and 104 ± 10.2 (N=13) for Florida. Chromatograms showed good peak separation and sharpness of peaks.

Field Fortification Recovery

Field fortification samples were generated on three days (pre-application, DAT-3, and DAT-7) at each site. Cotton sheeting was fortified with either a low level (17.9 ng/cm^2) or a high level (718 ng/cm^2) of pendimethalin. The fortifications were performed in triplicate. At the pre-application sampling, the cotton cloth used for the control samples and the fortifications was exposed to untreated turf using the Modified California Roller Technique. At the DAT-3 and DAT-7 sampling, pristine cotton sheeting was used for the controls and fortifications. Table 1 summarizes the overall average field fortified sample recovery results. The field recovery values were within guidelines (90 to 110 percent recovery) for the California and Florida samples. However, there was a significant positive bias for the Pennsylvania samples (131 to 133 percent).

Table 1. Summary of Fortified Field Sample Recoveries for Pendimethalin

Test Substance	Fortification Level (ng/cm^2)	California (percent)	Pennsylvania (percent)	Florida (percent)
Pendimethalin	17.9	104 ± 5.1 (N=9)	133 ± 5.5 (N=9)	104 ± 4.7 (N=9)
	718	98.9 ± 4.4 (N=9)	131 ± 6.2 (N=9)	105 ± 4.3 (N=9)

Storage Stability Recovery

No designated storage stability samples were prepared. ACCO relied on the fortified field samples instead. The sample storage interval for the California and Florida TTR samples ranged from 80 to 104 days. TTR samples collected in Pennsylvania were stored between 67 and 83 days. Field fortification samples collected in California and Florida were stored between 90 and 104 days. Field fortification samples collected in Pennsylvania were stored between 74 and 76 days.

Results

Pendimethalin TTR data are summarized in Table 2. All of the field Pendulum® 2G residue levels were below the LOQ, at all sampling intervals at all sites, during the 14 days following treatment, with the exception of DAT-7 when residues averaged 17.5 ng/cm^2 at the Pennsylvania site. This spike occurred 1 day after mowing and 11 days after a rainfall.

In California, the maximum Pendulum® WDG residue occurred 4 hours after treatment and decreased to below the LOQ by DAT-14. In Pennsylvania, the maximum Pendulum® WDG residue occurred immediately after treatment (DAT-0) and decreased to below the LOQ by DAT-7. In Florida, the maximum Pendulum® WDG residue occurred on DAT-1 and decreased to below the LOQ by DAT-14 for one of the three samples. The maximum Pendulum® WDG residue levels varied significantly from site to site. The highest level was reported in California (1,280 ng/cm²). The maximum level reported in Florida (608 ng/cm²) was only approximately 50 percent of the California maximum. In addition, a significant number (approximately 50 percent) of Pendulum® WDG TTR samples exceeded the high field fortification level of 718 ng/cm² in California, indicating that the high fortification level was not well chosen for the California data set.

Table 2. Summary of Pendimethalin Turf-Transferable Residue Data

Study Sample Set	Maximum Uncorrected Residue (ng/cm ²) and Day Attained	Minimum Uncorrected Residue (ng/cm ²)	Number of Samples <LOQ	Number of Samples >LOQ and <17.9 (ng/cm ²)	Number of Samples >718 (ng/cm ²)
<i>California</i> Pendulum® 2G	<LOQ at all sampling intervals	<LOQ at all sampling intervals	24/24	0/24	0/24
Pendulum® WDG	1,280 at 4 hours (DAT-0.167)	<LOQ at all DAT-14	3/24	1/24	11/24
<i>Pennsylvania</i> Pendulum® 2G	22.7 at DAT-7*	14.8 at DAT-7* <LOQ at all other sampling intervals	21/24*	2/24*	0/24
Pendulum® WDG	846 at 0.1 hours (DAT-0)	<LOQ at all DAT-7 and DAT-14	6/24	0/24	6/24
<i>Florida</i> Pendulum® 2G	<LOQ at all sampling intervals	<LOQ at all sampling intervals	24/24	0/24	0/24
Pendulum® WDG	608 at DAT-1	7.70 at DAT-14 <LOQ at DAT-14 for 1 sample	1/24	2/24	0/24

* All but three samples (DAT-7) were <LOQ. These spikes occurred 1 day after mowing and 11 days after a rainfall.
 Low fortification level = 17.9 ng/cm².
 High fortification level = 718 ng/cm².
 LOQ = 4.31 ng/cm².

Sample Calculations

Residue analysis results calculated by Maxim Technologies were based on the peak height responses of the samples and standards. Versar verified these calculations for a select number of samples. The following equation was used:

$$\text{ng/sq. cm} = \frac{R(\text{samp}) \times (V1) \times (V3) \times (V5) \times C(\text{std}) \times DF}{[(R(\text{std})A + R(\text{std})B / 2] \times (A) \times (V2) \times (V4)} \times 1,000 \quad (\text{Eq. 1})$$

where:

- R(samp) = peak height response of sample
- R(std) = peak height response of working standards, R(std)A and R(std)B
- A = area of sample taken for analysis in cm² (5,574 cm²)
- V1 = volume in mL of extracting solvent (1,000 mL)
- V2 = volume in mL of aliquot taken for analysis (10 mL)
- V3 = volume in mL of final solution used for analysis (1 mL)
- V4 = volume in μL of sample solution injected (1 μL)
- C(std) = concentration in μg/mL of standard solution (0.50 μg/mL)
- V5 = volume in μL of standard solution injected (1 μL)
- DF = dilution factor, if needed, of final solution
- FV = fortification volume in mL
- FC = fortification concentration (of standard solution added) in μg/mL

No adjustments in the raw data were made for the field fortification recoveries, which averaged 104 and 99 percent in California, 133 and 131 percent in Pennsylvania, and 104 and 105 percent in Florida for the low and high level field fortifications, respectively.

ACCO averaged triplicate TTR values (Pendulum® WDG) at each sampling interval from each test site. Linear regressions were conducted plotting time sampled versus the logarithm (base 10) of the pendimethalin residue values to show the dissipation of the residue over the test period. The correlation coefficients for the regressions were high, ranging from 0.941 to 0.967 for all test sites (Table 3).

Table 3. Pendimethalin TTR Half-lives as Estimated by ACCO and Versar

Data Used for Regression	California (Pendulum® WDG)		Pennsylvania (Pendulum® WDG)		Florida (Pendulum® WDG)	
	Half-life (days)	R ²	Half-life (days)	R ²	Half-life (days)	R ²
<i>ACCO</i> DAT-0 to DAT-7/14	1.67	0.967	1	0.941	2.23	0.941
<i>Versar</i> DAT-0 to DAT-5, 7, 14 where TTR values >LOQ	1.35	0.907	1.44	0.985	2.17	0.940

The equation for the linear regressions was:

$$\text{Log } Y = b[0] - b[1] X \quad (\text{Eq. 2})$$

where:

b[0] = intercept

b [1] = slope

The half-life was calculated as follows:

$$\text{Half-life} = \text{Log}0.50/\text{slope} \quad (\text{Eq. 3})$$

Versar used individual TTR values (Pendulum® WDG), not averages, in conducting linear regressions on the three data sets. Only TTR values above the LOQ were included with one exception. When analyzing the data, a value of one-half of the LOQ was used for data points where TTR values were below LOQ. This occurred in the Florida data set. The linear regressions were conducted using the natural logarithm of TTR values processed by Microsoft's Excel 97®. The TTR half-lives as estimated by Versar are presented in Table 3. Versar's values differed very little from ACCO's. The correlation coefficients for the regressions were again high, ranging from 0.907 to 0.985 for all test sites.

Data Variability

Versar examined data variability as part of the linear regression exercise. Coefficients of variance for the replicate sample data, up to the first post application day where all replicate residue values were above the LOQ (with one exception), ranged from 4.24 to 70 percent for California, 4.04 to 22.9 percent for Pennsylvania, and 3.67 to 55 percent for Florida. There are no specific requirements concerning the variability of replicate samples in the Guidelines.

Compliance Checklist

Compliance with OPPTS Series 875, Occupational and Residential Exposure Test Guidelines, Group B: Postapplication Exposure Monitoring Test Guidelines, 875.2100, Transferable Residue Dissipation: Lawn and Turf, is critical. The itemized checklist below describes compliance with the major technical aspects of OPPTS 875.2100, and is based on the "Checklist for Residue Dissipation Data" used for study review by the U.S. EPA/OPP/HED.

- *Typical end use products of the active ingredient used.* This criterion was met.
- *Transferable turf residue (TTR) data to be collected from at least three geographically distinct locations for each formulation.* This criterion was met. TTR data were collected in California, Pennsylvania, and Florida.
- *The production of metabolites, breakdown products, or the presence of contaminants of concern, should be considered in the study design on a case-by-case basis.* This criterion was not met. There was no discussion of potential breakdown products or contaminants of concern in the pendimethalin-containing formulations used, although there are regulated metabolites. Only pendimethalin residues were analyzed.
- *The choice of end-use product to test should be made considering the following factors: (1) availability in several different formulations; (2) liquid formulations (e.g., emulsifiable concentrates) are preferred; (3) watering-in is to be avoided; (4) product to be applied in a minimal amount of water; (5) product to be applied after mowing and watering; (6) application of the product should not be made within 24 hours of an expected rain event.* These criteria were partially met. Both a granular and a water dispersible granular formulation were used at three test sites. Watering-in of the products was not performed following application. However, the product labels for both formulations states that "the efficacy will improve if the application is followed by one-half inch of rainfall or its equivalent in sprinkler irrigation." The water dispersible granule was applied at all three test sites using the minimal volume of water recommended on the product label. Both formulations were applied to well-established turf which had been mowed 1 day prior to application to a height of 2-3 inches. No rainfall events occurred within 24 hours prior to the

applications at two of the test sites. Rainfall information was not available for California because of the incorrect data (reporting year) presented in this report.

- *Site(s) treated representative of reasonable worst-case climatic conditions expected in intended use areas. This criterion was partially met.*
- *End use product applied by application method recommended for the crop. Application rate given and should be at the least dilution and highest, label permitted, application rate. These criteria were partially met. According to the product labels for Pendulum® 2G (EPA Reg. No. 241-375) and Pendulum® WDG (EPA Reg. No. 241-340), the maximum label application rates were used. The application methods were acceptable. The product labels state that a repeat application after 6-8 weeks may be applied, if necessary, and depending on the initial application rate. Only one application of each formulation was applied in this study.*
- *Applications occurred at time of season that the end-use product is normally applied to achieve intended pest control. This criterion was partially met, however, it is acceptable. The study was conducted during the late spring growing season and the label recommends the product be applied earlier in the spring prior to weed germination.*
- *If multiple applications are made, the minimum allowable interval between applications should be used. This criterion was not applicable. Only one application was made at each site.*
- *Turf TTR samples should be collected via one of the following sampling methods: (1) dislodging residues from grass clippings; (2) Cheese-cloth wipes; (3) PUF Roller; (4) California Cloth or PUF Roller (32 lbs); (5) Drag Sled. This criterion was met. The turf TTR samples were collected utilizing the Modified California Roller Technique.*
- *Sampling should be sufficient to cover three half-lives and establish a dissipation curve. Recommended sampling intervals are 1 hour, 4 hours, 8 hours, 12 hours, 1, 2 and 3 days after application. This criterion was met. TTR samples were collected up to 1 day prior to treatment and 0.1 (immediately after), at 4 hours and 8 hours, and on DAT-1, 3, 5, 7, and 14. Triplicate samples were collected at each sampling interval. Average TTR field levels for Pendulum® 2G were below the LOQ, at all sampling intervals at all sites, with one exception which appears to be an anomaly. Average TTR field levels for Pendulum® WDG decreased to below the LOQ by DAT-7 or DAT-14 at two of the test sites. At the third test site, one of the triplicate samples was below the LOQ by DAT-14. The dissipation curves generated for all three test sites showed good correlation.*

- *Grass clipping samples to be dislodged within four hours of collection. Correlation established between surface area sampled and sample weight.* These criteria were not applicable because grass clippings were not collected. Turf transferable residues were collected on pieces of cloth.
- *Meteorological conditions including temperature, wind speed, daily rainfall and humidity provided for the duration of the study.* This criterion was partially met. The statement of GLP Compliance prepared for each test site states “weather data were not collected per the GLP regulations.” Air temperature readings, relative humidity, wind speed and direction, and soil moisture and temperature were reported for the day of application at each test site. However, problems with the daily weather data presented for each site were noted.
- *Residue storage stability, method efficiency (residue recovery), and limit of quantitation (LOQ) provided.* These criteria were met. Laboratory recovery and field fortification recovery values were provided in the report. Storage stability was demonstrated using the fortified field samples. The report states the LOQ to be 4.31 ng/cm².
- *Triplicate, randomly collected samples to be collected at each sampling interval.* This criterion was met. Triplicate samples were collected at each sampling interval. Samples were collected randomly with no area being sampled more than once.
- *Field recovery sample values.* The criterion was partially met. The field recovery sample values were within guidelines (90 to 110 percent recovery) for the California and Florida samples. However, there was a significant positive bias for the Pennsylvania samples (131 to 133 percent).