

Health & Safety

Report

Worker Health and Safety Branch

HS-1860

Transferable Turf Residue Following Imidacloprid Application

March 1, 2005

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Study Dates

Study initiation	September 9, 2004
Sample collection training	September 9, 2004
Experimental start date	September 22, 2004
Experimental termination date	October 14, 2004
Laboratory sample analysis started	September 23, 2004
Laboratory sample analysis completed	October 20, 2004
Study completed	March 3, 2005

Study Report Approvals

Approved by: [original signed by Susan Edmiston] March 3, 2005
 Susan Edmiston, Study Supervisor Date

[original signed by Chuck Andrews] March 3, 2005
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Quality Assurance Statement

Field Activities:

<u>Audit Date</u>	<u>Phase</u>	<u>Study Director Notified</u>	<u>Management Notified</u>
Aug. 16, 17, 2004	Protocol	Aug. 18, 2004	Aug. 18, 2004
Sept. 23, 2004	In process field audit	Oct. 4, 2004	Oct. 4, 2004
Feb. 10, 14, 15, 2004	Raw Data/Final report	Feb. 16, 2004	Feb. 16, 2004

[original signed by Kathy Orr] March 3, 2005
 M. Kathy Orr, Quality Assurance Officer Date

Laboratory Activities:

<u>Audit Date</u>	<u>Phase</u>	<u>Study Director Notified</u>	<u>Management Notified</u>
Unknown	Method validation report	August 31, 2004	August 31, 2004
Not completed	Records inspection	NA	NA
Not completed	Sample analysis	NA	NA
Not completed	Raw data	NA	NA

Compliance Statement

Based upon all information supplied to me including the California Department of Food and Agriculture, Center for Analytical Chemistry, (Laboratory Statements of Compliance), I hereby confirm that all aspects of this study, Project0404, were conducted in compliance with the US Environmental Protection Agency, Good Laboratory Practice standards (GLP, 40 CFR 160), with the following exceptions:

The test substance characterization was not conducted before its use in the study as required in 40 CFR 160.105(a).

Supplemental and support data such as weather data were not collected in compliance with GLP.

Not all required quality assurance SOPs were in place at the time of study conduct and other required SOPs may not have been in place at the time of study conduct.

[original signed by Angélica M. Welsh]
Angélica Welsh, Assoc. Environmental Research
Scientist, Study Director

March 3, 005
Date

Protocol and standard operating procedure deviations were documented and can be found in Appendix 1.

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Transferable Turf Residue Following Imidacloprid Application

Abstract

This study was conducted to determine transferable turf residue (TTR) with two techniques, Modified California Roller (MCR) and a specific variation of the California Roller (CR) method (several variations of the CR method exist). Turf was treated with Merit[®] 0.5 G Insecticide (granular imidacloprid formulation) and Merit[®] 2 Insecticide (liquid imidacloprid formulation).

The test site was a sod farm in Yolo County, California. The applications for the liquid-treated plot were made with a commercial ground-boom sprayer; the granular applications were made with a metered-feed drop spreader on dry grass. The low and high application rates were approximately 0.1 and 0.4 lb active ingredient/acre for both the liquid and granular applications. The study consisted of five trials with two replicates per formulation and rate application.

The study found that both MCR and CR had significantly greater sensitivity (detection of residues at a particular application rate) to liquid than granular formulations. There was no evidence that the sensitivity of either TTR method varied depending on how much active ingredient was applied, nor that the sensitivity varied more with one measurement method or one formulation than the other. This difference in sensitivity suggests that it *may* not be appropriate to pool data from liquid and granular formulations. Data are needed to characterize the relationship of dermal exposure to TTR for granular formulations; dermal exposure was not monitored in this study. If the relationship of dermal exposure to TTR differs significantly between liquid and granular formulations, then data from liquid and granular formulations should not be pooled.

No previous study has directly compared TTR results from the MCR method to results from any CR method. The current study found that the CR method tested gave TTR values averaging 2-3 times higher than the MCR method in side-by-side samples. Within each application method, the variance in method sensitivity was not significantly greater for either formulation nor for the high or low application rates. The difference in sensitivity between the two methods suggests that it is *not* appropriate to pool data from samples collected with MCR and the CR method tested; furthermore, other CR methods should be compared with MCR before pooling data.

Introduction

Pesticides are periodically used to protect turf from undesirable plants, fungi, mollusks and insects. Pesticide applications to turf normally leave residues on soil, thatch, and grass blades. These residues can be the source of human exposure resulting from contact transfer to clothing and skin. The population considered to have the greatest exposure potential from contact with pesticide-treated turf is young children who may play on residential turf soon after pesticide applications. Knowledge of the fate, transport, and availability of turf residues in the public literature is limited.

The amount of residue transferred from treated turf to humans is a critical parameter for conducting exposure assessments of people who reenter pesticide-treated areas. Generic exposure estimates can be derived if both the amount of transferable turf residue (TTR) and the rate of transfer to humans during contact are known. Several techniques are available for measuring TTR. Five techniques were evaluated in a study conducted by the Outdoor Residential Exposure Task Force (ORETF) in 1996, described in detail by Klonne *et al.* (2001). Briefly the five techniques included the California Roller (CR) (Ross *et al.*, 1991), the drag sled (Camann *et al.*, 1993), the polyurethane foam (PUF) roller (Lewis *et al.* 1994), shoe shuffling (Thompson *et al.*, 1984), and the foliar wash (Hurto and Prinster, 1993). The study suggested that some of the methods gave similar results and that modifications might improve performance (Klonne *et al.*, 2001). A second study conducted by ORETF in 1997 evaluated the “Modified California Roller” (MCR), the “Modified Shoe”, and the ORETF Roller techniques (Rosenheck *et al.*, 2001). In this study, the MCR produced the most consistent results and was selected for future use by ORETF (Rosenheck *et al.*, 2001).

Data used by ORETF to derive a generic estimate of transfer from treated turf relied on studies using the CR (all with some modification to the original design) as well as the MCR studies. There is some question as to whether the various CR methods (Bernard *et al.*, 2001, Eberhart and Ellisor, 1994; Eberhart, 1993; Rosenheck and Schimelfining, 1994; Ross *et al.*, 2001) are equivalent and whether the MCR and CR method variants result in truly equivalent results. In addition, the data set used by ORETF to develop the generic transfer estimates has insufficient data for granular formulations to determine whether the relationship between TTR and exposure is the same for both liquid and granular formulations.

The Department of Pesticide Regulation (DPR), Worker Health and Safety Branch (WH&S) use exposure and environmental concentration estimates to develop realistic exposure assessments. These exposure assessments are used to set appropriate health-protective standards. Previous WH&S turf research includes dislodgeable foliar residue and TTR studies (Maddy *et al.*, 1984; Maddy *et al.*, 1986a; Maddy *et al.*, 1986b; Schneider, et al. 1998).

The objective of this study was to compare the transfer of pesticide residues from turf to a cotton cloth using two sampling methods, a CR method vs. the MCR method. Four combinations of formulation and application rate were used in the comparison.

Materials and Methods

Two techniques were evaluated in this study. The CR is a method of collecting residue samples using a cloth sheet and a roller (Ross *et al.*, 1991). Bernard *et al.*, (2001) describes the specific variant of the CR method used in our study. The MCR is a method of collecting residue samples

using a cloth sheet mounted on a frame and a roller (Fuller *et al*, 2001). The techniques were evaluated across formulation type (granular and liquid) for sensitivity (amount of residue transferred to the matrix). The study was conducted under the federal Good Laboratory Practices (GLP) standards (40 CFR Part 160).

Test and Reference Substances

Imidacloprid (1-[(6-chloro-3-pyridinyl)methyl]-N-nitro-2-imidazolidinimine; CAS No. 138261-41-3) was applied to turf in both liquid and granular formulations. The liquid formulation used was Merit[®] 2 Insecticide, EPA Registration No. 3125-418, manufactured by Bayer Corporation. It is an aqueous concentrate in Toxicity Category III. The granular formulation applied was Merit[®] 0.5 G Insecticide, EPA Registration No. 432-1328, also manufactured by Bayer Corporation. The product is a Toxicity Category III pesticide.

The study director purchased the test substances; the receipts are on file. The lot number, storage location, and container size were documented. The test substances, Merit[®] 2 Insecticide and Merit[®] 0.5 G Insecticide, were stored in a secure storage facility, and documented according to standard operating procedures.

Reference substances were prepared from commercially available 99+% purity material.

Test System

The test system consisted of turf and white, woven, 100% pima cotton cloth; thread count is unknown.

Experimental Design

The study consisted of five trials involving five different plots treated between September 22, 2004 and October 13, 2004. Each trial consisted of four imidacloprid treatments to previously untreated sod: granular, high application rate; granular, low application rate; liquid, high application rate and liquid, low application rate. The high application rate was the maximum label rate (0.4 lb active ingredient (ai)/acre (A)) while the low application rate (0.1 lb ai/A) was set, based Eberhart and Ellisor (1994), low enough to detect a difference in residue, but high enough to be able to detect residue at 18 hours post application. For each trial, each of four subplots was treated once with one of the treatments. Subplot dimensions were 10 ft' x 400 ft for liquid applications and 3 ft x 100 ft for granular applications. The subplots were separated by a minimum of 20 ft to prevent cross contamination. The subplots were identified and marked prior to application to prevent sampling the same area twice or unintentional contact with the treated areas by study personnel. Each subplot was divided into two blocks from which two random samples were taken, one with the CR and one with the MCR.

Test Site

The test site was a sod farm located in Yolo County, California. The turf was a blend of tall fescue and Kentucky bluegrass mowed to approximately 3 inches in height prior to testing. The turf appeared healthy. Irrigation was performed approximately two to three days before each application. The turf was dry when the test substances were applied as applications were done in the afternoon, when no dew is present. No other pesticide applications were made to the area of

the test plots during the study dates. Plots were not irrigated between application and sample collection.

Equipment and Application Procedures

The same application equipment was used for all trials. Applications were scheduled for the afternoon so that samples could be collected approximately 18 hours after each treatment.

Study personnel completed the loading and application of granular imidacloprid and also measured the amount of liquid material to add to the mix tank. A licensed applicator added the liquid product to the mix tank as measured along with 10 gallons of water and completed the application.

Liquid application: Liquid imidacloprid was applied using a power take-off driven pump and a 300-gallon Demco sprayer, with a 10-foot boom and Turbo Teejet spray nozzles (size=11005), pulled by a John Deere 1250 tractor. The grower/applicator calibrated the equipment on September 13, 2004 with the tractor ground speed at range I, gear 2 and approximately 2000 RPM, to deliver 109 gal/A. Actual time to traverse 400 ft was recorded for all five applications (approximately three min).

The target application rate for Merit[®] 2 Insecticide was 0.1 lb and 0.4 lb ai/A for the low and high rates, respectively. No other chemicals were tank-mixed with the Merit[®] formulation. Study personnel confirmed imidacloprid applications by observing and documenting the mixing, loading, and application activities.

Granular application: The study director obtained a pesticide research authorization from the Department of Pesticide Regulation (DPR 409019) for the experimental use because Merit[®] 0.5 G Insecticide is not registered for use on sod farms. Study personnel conducted the granular application using a Gandy[®] (Gandy Co., Owatonna, MN) push box drop spreader 36 inches wide. The granular application equipment was calibrated on September 22, 2004 by loading known weights of the granular material into the spreader, making a 100-foot pass, and then weighing the remaining material to calculate the amount applied. A gear setting of 19 was used to deliver 0.1 lb ai/A and a setting of 28 to deliver 0.4 lb ai/A was used during the study. The granular formulation required no dilution.

Environmental conditions: No rainfall occurred between the study applications and the sample collection. Environmental conditions during the applications are presented in Appendix 2.

Quality Control Samples

Formulation: One sample of approximately 50 -100 mL was collected from each of the two Merit[®] 2 lot numbers used in the study and one sample of approximately 2 ounces in weight was collected from the Merit[®] 0.5 G container. All formulation samples were chilled on ice and stored in a separate cooler from all other samples.

Tank Mix: One composite sample of approximately 50 - 100 mL was collected from the nozzles of each tank mix sprayed. The tank mix samples were stored in glass jars or polypropylene containers (Trial 2 only) and chilled on ice in a separate cooler from all other samples.

Pre-Application: One sample from each subplot was collected by each sampling method (CR and MCR) each study day before application. Pre-application samples were handled in the same manner as the TTR samples (see below).

Application Rates: Deposition samples were used to evaluate the liquid and granular application rates. Prior to liquid applications three 10 x 10 cm squares of material identical to that used to construct the TTR samples, backed with aluminum foil and mounted on a paper plate were anchored to each turf subplot. All deposition squares were collected immediately after application. The cloth and foil assembly was carefully folded with the exposed side together and placed in a pre-labeled glass jar. The jar was placed in a cooler containing dry ice.

Prior to granular applications, three deposition pans were placed on each turf subplot. All deposition pans were collected immediately after application. The granules from the aluminum pans (8 x 8 x 15/8 inches) from Trials 1 and 2 were carefully transferred to a glass jar. For Trials 3 through 5, plastic containers (5.5 x 5.5 x 2.0 inches) with tight-fitting lids were used to eliminate transferring the granules to another container. The sealed jars and plastic containers were kept at ambient temperature until weighed.

Field Fortification: The laboratory Principle Analytical Investigator (PAI) prepared field fortification solutions in ampoules containing 20, 200 and 2000 µg imidacloprid in 1 mL methanol. Each vial was labeled with the amount of test substance it contained. Ampoules were stored under refrigeration and delivered to the test site stored chilled on ice in a designated insulated cooler.

Field fortification media consisted of 100% pima cotton cloth approximately 27" x 39". For each trial, field fortification samples consisted of two solvent blanks and two field fortification samples at each of three fortification levels.

On each sample collection day folding tables were set outdoors at least 50 feet away from the treatment area. Field fortification samples and solvent blanks, prepared at the field site, were on separate tables, approximately 10 feet apart. The tables were covered with plastic (6 mil) and clean new package wrapping paper placed on top of the plastic each day. The table dedicated to field fortifications had three separate labeled stations to accommodate the three respective fortification levels from low to high rate and with their own supplies. The cloth media for each field fortification and solvent blank was folded to fit and placed in a disposable aluminum pan. The order of sample preparation was solvent blanks first, followed by low, medium, and high fortification rates, respectively. Application of prepared fortification solutions to cloth matrices was completed by emptying a vial, as uniformly as possible over the exposed surface of the cloth matrix. Each vial was rinsed with methanol two times. The rinsate solutions were also applied to the matrix. The sample was collected immediately by carefully folding the treated side in and placing it in the appropriate pre-labeled glass-jar. The sample jars were sealed and placed on dry ice in an ice chest, separate from all other samples.

Field Method Blank: Field method blanks were collected each study day where no chemical was applied and consisted of two samples collected by each sampling method (CR and MCR). Field method blank samples were handled in the same manner as the TTR samples (see below).

Transferable Turf Residue

TTR was evaluated using the MCR and the CR techniques described below. Each day, from each treated subplot two samples were collected by each sampling method using a random block design with two blocks/subplot. TTR samples were collected approximately 18 hours after application, except Trial 5. For Trial 5, the turf was still very damp at 18 hours post application; sample collection started at 21.5 hours post-application. (A more detailed description of the assembly and use of the methods is presented in Appendix 3).

For the MCR samples, the sampling medium consisted of 100% pima cotton, cloth measuring approximately 27" x 39". This size cloth allowed space to clamp it to a rigid frame and a 24.5" x 36" area that is exposed to the turf (5690.31 cm²). The cloth was covered with 6-mil plastic sheeting, measuring approximately 29" by 41" also secured with clamps to the rigid plastic frame. The frame assembly was carefully placed on the sampling plot with the cloth face down, so that the cotton cloth touched the turf. Once placed on the turf, the frame assembly was not adjusted or moved as this would result in the cloth coming into contact with a greater surface area than 5690 cm². Spikes secured the frame to the plot. A roller of known size and weight (see Appendix 3) was placed on the assembly and rolled over the cotton/plastic sheet assembly five times slowly and evenly. One forward and backward motion is considered one roll. Upon completion of 5 rolls, the roller was taken to a clean area to prevent possible contamination. The roller did not directly contact the turf during or between samples. The frame was then lifted from the turf and taken to the sample-processing table. There the cloth was inspected for visible debris (grass clippings and thatch) that was carefully removed with tweezers. The clamps were unfastened, the frame removed, and the cotton sheet was folded with the exposed side together and placed in a labeled glass jar. The jar was immediately placed in a cooler containing dry ice for storage and transportation to the analytical laboratory facility.

The frames were cleaned between uses and at the end of the day. All frames were thoroughly cleaned with methanol and then washed with water to ensure no contaminating residues remain on the frame. A more detailed description of the assembly and use of the MCR are presented by Fuller *et al.*, (2001).

The CR sampling method did not involve a frame. CR sampling media consisted of white 100% pima cotton cloth, measuring approximately 23.6" x 11.8", the entire area was exposed to the turf (1796.4 cm²). The cloth was centered and attached with pins to 6-mil plastic sheeting, measuring approximately 26" x 14". The plastic sheeting was placed so that it completely covered the cloth. The cloth/plastic sheeting assembly was placed on the sampling plot with the cloth touching the turf and secured with spikes through the corners of the plastic. Study personnel also held the assembly in place to keep movement to a minimum once it was placed on the turf. A CR roller (see Appendix 3 for details) was rolled over the cloth/plastic assembly 20 times slowly and evenly. One forward and backward motion is considered one roll. After rolling, the roller was taken to a clean area to prevent possible contamination. The roller did not directly contact the turf during or between samples. The cloth/plastic assembly was lifted from the turf and transferred to the sample-processing table. There the cloth was inspected for visible debris (grass clippings and thatch) that was carefully removed with clean tweezers. The pins were carefully removed from the assembly. Then the cotton sheet was folded with the exposed

side together and placed in a labeled glass jar. The sample was immediately placed in a cooler containing dry ice for storage and transportation to the analytical laboratory facility. A description of the use of the CR as used in this study is given in Bernard *et al.*, (2001).

All sample-processing tables were covered with clean plastic each day. Package wrapping paper was placed over the plastic and replaced after each sample was processed.

Field Quality Assurance

Quality assurance (QA) inspections were conducted in accordance with SOPs and included protocol, application calculations, application, sample collection, raw data and final report audits.

Sample Storage and Transportation

All samples were placed in the appropriate ice chest, stored frozen or chilled and transported the same day to the California Department of Food and Agriculture, Center for Analytical Chemistry (CDFA/CAC) for analysis. All TTR and field fortification samples were frozen on dry ice; all other samples were chilled on ice after collection and until delivered to the analytical facility. The samples were shipped in accordance with standard operating procedures for sample tracking, shipping and receiving samples.

Sample transportation, storage and receipt by the laboratory was documented with a chain of custody (COC) and used in accordance with standard operating procedures. Once at the laboratory, samples were stored in accordance with the CDFA/CAC Branch Procedures (BP) for sample receiving, login, handling, storage and disposal. A separate COC was maintained for storage and movement of the samples within the laboratory.

Sample Analysis

The CDFA/CAC prepared all analytical and reference substances and performed all chemical analyses for the study. The PAI conducted method evaluation, development, and validation to determine the precision and accuracy of the analytical methodology for extraction and analysis of imidacloprid from 100% cotton fabric. The study director approved the method validation report prior to extraction of any study samples.

Field Fortification and TTR Extraction and Analysis

Field fortification samples were extracted with the TTR samples. The PAI extracted all TTR and fortification samples within 2 to 15 days of their receipt by CDFA/CAC. The 100% cotton sample media were analyzed for imidacloprid by Liquid Chromatography/Mass Spectrometry (LC/MS). Results were reported as micrograms imidacloprid/sample. The entire cloth sample was extracted by adding 400 mL methanol to the sample jar and rolling it on a jar roller for 30 minutes. The extract (45 mL) was rotary evaporated to about 2 mLs and transferred to a test tube using methanol, then concentrated to 5 mL. Analysis was completed on a Finnigan DecaXP LC/MS analytical system with an atmospheric pressure chemical ionization interface. The LC column was a Waters Symmetry Shield, 5 μ m RP18, 3.9 x 150 mm operating at a flow rate of 0.8 mL/min. The gradient was 10% methanol/water to 90% methanol/water at 15 minutes, hold 1 minute, reset to 10% methanol/water, and hold 5 minutes. Acetic acid (0.2%) was added to

each of the mobile phase components to enhance ionization. The sample extract (10 μL) was injected, resulting in an imidacloprid retention time of 8.5 minutes. The limit of detection (LOD) was determined to be 3 μg of imidacloprid per sample for TTR sample media.

Deposition, Tank Mix and Formulation Sample Extraction and Analysis

Deposition Samples: CDFA/CAC Formulations Laboratory conducted deposition sample analysis of the imidacloprid liquid formulation by LC/MS. Extraction times ranged from one to fourteen days after sample receipt. The results were reported in μg per sample. The study director used the laboratory scale to weigh the granular formulation deposition samples and reported the results in μg per sample.

Tank Mix and Formulation Samples: CDFA/CAC Formulations Laboratory conducted the tank mix and formulation sample analysis for imidacloprid by high performance liquid chromatography; results were reported as percent imidacloprid. All tank mix and formulation samples were extracted within fifteen days of receipt.

Sample Retention and Disposal

TTR and fortification samples were not retained after residue extraction. Sample extracts, tank mix samples, and other samples collected for the study were retained and will be disposed of in accordance with DPR WH&S policy (Schneider, 2000) for studies conducted under GLP. Disposal of any samples or extracts will be documented.

Meteorological Data Collection

Daily meteorological measurements were obtained from the California Irrigation Management Information System (CIMIS) weather station near Davis (Station # 6, approximately four miles west of the test site), including air temperature, relative humidity, and wind speed and direction (Appendix 2). CIMIS does not operate in adherence with GLP.

Data Analysis

All raw data is stored in a single Microsoft Access database.

The data, reported in μg per sample, were prepared for statistical analysis by substituting one-half the detection limit (one-half of 3 μg per sample = 1.5 μg) for the value of any sample that was non-detected. Sample values were next converted to $\mu\text{g}/\text{cm}^2$ by dividing by sample area (1800 cm^2 for CR; 5690 cm^2 for MCR). These values were then normalized by the target application rate in pounds ai/A, 0.1 or 0.4 lb ai/A for the low and high rates, respectively. The normalization calculation is as follows: $\mu\text{g}/\text{cm}^2$ was divided by 0.0001 to convert it to $\mu\text{g}/\text{m}^2$ then divided by the lb ai/A in order to obtain $\mu\text{g}/\text{m}^2$ per lb ai applied. Finally, the two samples for each method, rate and formulation for each day were averaged. Since no imidacloprid was found on any pre-application sample it was not necessary to correct averaged data points.

Analysis of variance on restricted dataset with target application rate as an independent variable: Because the liquid formulation was not applied at the target rate in the first two trials, the initial statistical analysis used only the data from Trials 3, 4 and 5. Only the data from Trials

2, 3 and 4 were used initially for the granular formulation, because in Trials 1 and 5, all (or all but one) granular samples were non-detects at both application rates.

A mixed-model analysis of variance (ANOVA) was used to test the effects of formulation (liquid vs. granular), application rate (0.1 vs. 0.4 lb ai/A) and TTR method (CR vs. MCR) on sensitivity of the TTR measurement (expressed as $\mu\text{g}/\text{cm}^2$ of measured TTR per lb of ai applied per acre). The model is called “mixed” because it contains both between- and within-units sources of variability (Myers, 1972, Ch. 8). In this model, the unit is a subplot treated with a given combination of formulation and rate. Formulation and rate are between-units sources of variance because each combination of levels of these factors is applied to a separate subplot. Method is a within-units source of variance because both methods were used on each subplot.

The ANOVA was implemented using SAS PROC GLM (SAS V9.1). All effects were tested at the 0.05 level of significance. Before conducting the ANOVA, the assumptions of normality and equal variances were tested. Normality was tested using SAS PROC UNIVARIATE to calculate tests of normality within each treatment (i.e., each combination of formulation, rate and method). The null hypothesis of normality was not rejected for any treatment. Homogeneity of variance was tested using Levene’s test (Milliken and Johnson, 1984, p.17-19; implemented using SAS PROC GLM), which showed that variances were significantly greater with liquid than with granular formulation. The data were therefore transformed by taking the natural logarithms (the values that were log-transformed were the averages of the two samples for each method, rate and formulation for each day). The tests of normality and homogeneity of variance were repeated and the log-transformed data met the assumptions of normality and homogeneity of variance. The ANOVA was therefore done on the log-transformed data. This initial ANOVA showed that the sensitivity of TTR measurement varied significantly between formulations and between methods, but not significantly different between high and low application rates. Within each application method and within each formulation, the variance in method sensitivity was not significantly greater for the high or low application rates.

Analysis of variance on full dataset without application rate as independent variable: The absence of significant main or interaction effects of rate on method sensitivity in the initial ANOVA indicates that the observed effects of method and formulation are independent of application rate. Therefore, the liquid data from Trials 1 and 2, which were made at different application rates than those intended, were included in the second ANOVA. For this analysis, the data in $\mu\text{g}/\text{cm}^2$ were normalized by the *actual* application rates in pounds ai/A.

In addition, the granular data from Trials 1 and 5, excluded from the initial analysis because almost all of them were nondetects, were included in this ANOVA. This was desirable because these data contain valid information that need to be represented in the analysis. The simple substitution of one-half the detection limit could not be used, however, because the treatment combinations with all nondetects would have zero variance. Instead, each nondetect was replaced with a random value from a uniform distribution on the interval (0,3), where 3 is the detection limit. This was implemented in a Microsoft Excel spreadsheet using the Excel RAND function. In addition, since we did not have the amount of granular imidacloprid applied for Trial 4 (due to a spill), the target application rate was substituted as the application rate.

For this analysis, the data in μg per sample were prepared by substituting a random uniform (0,3) value for any nondetected sample. Next, values were converted to $\mu\text{g}/\text{cm}^2$ by dividing by sample area (1800 cm^2 for CR; 5690 cm^2 for MCR). These values were normalized by the actual application rates in pounds ai/A as described above. Finally, the two samples for each method, rate and formulation for each day were averaged.

A mixed-model ANOVA was used to test the effects of formulation and method on sensitivity of the TTR measurement. The experimental unit is still a subplot treated with a given combination of formulation and rate, but because rate is no longer a factor in the ANOVA, the subplots treated at different rates become replicates within formulations. Formulation is a between-units factor and method is a within-units factor.

The assumptions of normality and equal variances were tested as for the first ANOVA. Normality was tested within each combination of formulation and method. The null hypothesis of normality was not rejected for any treatment. Neither was the null hypothesis of homogeneity of variance across treatments rejected. The log-transformed data also met the assumptions of normality and homogeneity of variance. For consistency with the first analysis, and because TTR data are typically log-transformed for analysis, the ANOVA was done on the log-transformed data.

Protocol Amendments and Protocol, SOP or BP Deviations

The study director issued six protocol amendments. All protocol and SOP deviations were documented and reported to the study director (Appendix 1). Documentation included the date each deviation occurred, the nature of the deviation, and the potential effect(s) on the study. Original signed and dated documentation of deviations to the protocol are archived as raw data. Documentation provided replacement or additional text, reason for amendment, and was signed and dated by the study director. Where no SOP existed, study-specific procedures are documented in the raw data.

Results

Formulated Product

The results of the formulated product samples are reported in Table 1 below. The formulated product analysis appears to fall within acceptable ranges.

Table 1. Formulated imidacloprid analysis

Sample ID No.	Formulation	Imidacloprid Expected (%)	Imidacloprid Found (%)
SS01-1029	Liquid	21.4	20.42
SS04-4029	Liquid	21.4	21.7
SS01-1030	Granular	0.5	0.38

Application Rates

Liquid Formulation: Liquid formulation application rates were verified by tank mix samples and deposition samples. Table 2 contains the liquid tank mix sample results. Results show that Trial 1, high rate and Trial 2, both rates were applied at lower rates than targeted. Liquid imidacloprid

target and actual application rates are shown in (Table 3). The actual rates are those obtained from the measured amounts put in the application tank. Tank mix sample results confirm the non-target liquid application rates of Trial 1 and 2. Furthermore, the actual application rate was 98 – 104% of the target application rate for all other trials.

Table 2. Liquid imidacloprid tank mix sample results

Target lb ai ^a /acre	Theoretical % ai in tank mix	Percent				
		Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
0.1	0.0112	0.009	0.0017 ^b	0.009	0.010	0.0089
0.4	0.0449	0.016 ^b	0.0045 ^b	0.031	0.034	0.030

^a ai – active ingredient

^b Not the target rate

Table 3. Liquid imidacloprid target and actual application rates

Target Rate (lb active ingredient/acre)	Trial	Amount Mixed (mL/10gal)	Actual Application Rate ^a (lb active ingredient/acre)	Target Application Rate (%)
0.1	1	18	0.104	103.67
	2	2.25 ^b	0.013 ^b	12.95
	3	17	0.098	97.91
	4	17	0.098	97.91
	5	17	0.098	97.91
0.4	1	30 ^b	0.173 ^b	43.20
	2	8.5 ^b	0.049 ^b	12.24
	3	68	0.392	97.91
	4	68	0.392	97.91
	5	68	0.392	97.91

^a Actual application rate is determined by the measured amount of active ingredient added to the application tank

^b Not the target rate

Deposition samples (Table 4) were also used to evaluate the liquid application rate. The arithmetic means of deposited residues was 0.88 µg/cm² (± 0.22) and 3.57 µg/cm² (± 0.87) for the low and high rate, respectively (without Trial 1, high rate and Trial 2, high and low rate). This is equivalent to approximately 78.3% (±19.48) and 79.8% (± 28.54) of the theoretical deposition at an application rate of 0.1 and 0.4 lb ai/A, respectively. Appendix 4 contains the raw data for the liquid deposition samples.

Table 4. Liquid imidacloprid deposition on 100% cotton cloth

Trial	Application Date	Imidacloprid Rate	Apparent Imidacloprid		Arithmetic Mean ($\mu\text{g}/\text{cm}^2$)	Theoretical deposition (%)
			($\mu\text{g}/\text{sample}$)	($\mu\text{g}/\text{cm}^2$) ^a		
1	9/23/2004	Low	50.9 85.9 73.1	0.51 0.86 0.73	0.70	62.38
		High	150.1 129.6 155.7	1.50 1.30 1.56		
2	9/24/2004	Low	16.6 18.1 16.4	0.17 0.18 0.16	0.17	15.20
		High	56.6 49.0 48.4	0.57 0.49 0.48		
3	10/6/2004	Low	67.9 76.7 60.5	0.68 0.77 0.61	0.68	60.94
		High	254.1 237.9 363.2	2.54 2.38 3.63		
4	10/7/2004	Low	132.1 97.6 102.1	1.32 0.98 1.02	1.11	98.60
		High	576.9 457.5 329.0	5.77 4.57 3.29		
5	10/14/2004	Low	102.3 100.3 104.9	1.02 1.00 1.05	1.03	91.39
		High	231.5 367.2 398.5	2.32 3.67 3.98		
Grand Mean^b		Low			0.88	78.3
		High			3.57	79.8

^a Deposition area = 100 cm²

^b Grand mean = without Trail 1 High rate and Trial 2 Low and High rates

Granular Formulation: In Trial 4 low rate application, the material left over in the spreader was spilled while collecting it for weighing, thus we could not measure the amount applied. Table 5 shows the actual application rate of granular imidacloprid as measured by weighing the granular imidacloprid before and after application. (Appendix 5 contains raw data from the granular deposition samples.) Deposition during the application was also measured (Table 6) and ranged from 60.9% (Trial 1, high rate) to 153.6% (Trial 5, high rate) of the target application rate. The arithmetic mean of deposited residues of granular imidacloprid was 1.29 $\mu\text{g}/\text{cm}^2$ (± 0.36) and 4.6 $\mu\text{g}/\text{cm}^2$ (± 1.55) for the low and high rate, respectively. These residues are approximately equivalent 114.9% (± 32.08) and 102.5% (± 34.59) of the theoretical deposition at an application rate of 0.1 and 0.4 lb ai/A, respectively.

Table 5. Granular imidacloprid target and actual application rate

Target rate (lb ai ^a /acre)	Target (oz product/acre)	Trial	Amount Applied (oz product/acre)	Actual Application Rate ^b (lb ai/acre)
0.1	313.6	1	479.2	0.150
		2	290.4	0.091
		3	290.4	0.091
		4	435.6	0.136
		5	290.4	0.091
0.4	1254.5	1	885.7	0.277
		2	1161.6	0.363
		3	1452.0	0.454
		4	NA ^c	NA ^c
		5	1742.4	0.545

^a Active ingredient

^b Actual application rate is determined by the measured amount of active ingredient delivered by the spreader box.

^c Imidacloprid granular material spilled, weight of amount remaining after application not obtained.
NA=Not available

Table 6. Granular imidacloprid collected in deposition pans

Trial	Application		Imidacloprid ($\mu\text{g}/\text{cm}^2$)	Mean ($\mu\text{g}/\text{cm}^2$)	Theoretical deposition (%)
	Date	Rate			
1	9/23/2004	Low	1.53		
			0.90		
			1.07	1.17	103.9
		High	NS ^a		
			3.56		
			1.91	2.73	60.9
2	9/24/2004	Low	1.81		
			0.64		
			2.50	1.65	147.2
		High	3.48		
			4.30		
			6.20	4.66	103.8
3	10/6/2004	Low	0.76		
			0.61		
			0.85	0.74	65.9
		High	4.22		
			2.01		
			4.99	3.74	83.3
4	10/7/2004	Low	2.29		
			1.16		
			0.57	1.34	119.6
		High	5.26		
			4.59		
			5.11	4.99	111.1
5	10/14/2004	Low	2.93		
			0.48		
			1.23	1.55	137.9
		High	4.86		
			6.55		
			9.27	6.89	153.6
Grand Mean		Low		1.29	114.9
		High		4.60	102.5

^a No sample

Laboratory Fortification

Laboratory_fortification results are all within acceptable levels; results range from 85 to 112% (Table 7).

Table 7. Percent analytical recoveries of imidacloprid found in laboratory fortification samples

Trial	Level of Fortification								
	20 µg/sample			200 µg/sample			1000 µg/sample		
1	89.9	91.0	NS ^a	84.9	91.1	94.9	100.7	103.8	102.6
2	107.6	103.3	101.6	101.0	89.8	88.4	99.9	91.3	99.2
3	86.1	103.9	98.8	99.1	91.3	96.6	93.4	105.1	95.7
4	93.3	95.6	91.0	92.2	93.6	93.3	102.7	98.0	99.9
5	101.4	112.0	88.9	102.0	98.0	95.5	105.1	94.2	104.3

^a No sample

Field Fortification

With the exception of two samples at the low fortification level, all results were within 70-120% of the theoretical applied rate (Table 8). (The analysis of the reference substance in the field fortification ampoules is found in Appendix 6. Field fortification raw data is in Appendix 7.) The range of recoveries from the field fortification was similar to the recoveries reported for the laboratory fortification. There did not appear to be significant losses in the field or during shipment or storage. The solvent blank samples were all non-detects.

Table 8. Percent analytical recoveries of imidacloprid found in field fortification samples

Trial	Fortification (% of theoretical)					
	20µg		200 µg		2000 µg	
1	70	67	92	96	95	94
2	100	91	103	89	109	93
3	98	104	102	94	98	106
4	92	92	98	98	98	95
5	91	65	107	194	103	95

TTR Samples

Table 9 summarizes the mean results of TTR sampling each day; individual sample data are given in Appendix 8. With the exception of one method blank, all pre-application and method blank TTR samples were below the detection limit. (Pre-application and method blank sample results are in Appendices 9 and 10, respectively.) The overall mean TTR results for the liquid applied at the low rate (0.1 lb ai/A) were 0.0091 µg/cm² and 0.0045 µg/cm² for CR and MCR, respectively. The overall mean TTR results for the liquid applied at the high rate (0.4 lb ai/A) were 0.0298 µg/cm² and 0.0143 µg/cm² for CR and MCR, respectively. The overall mean TTR results for the granular applied at the low rate (0.1 lb ai/A) were 0.0012 µg/cm² and 0.0015 µg/cm² for CR and MCR, respectively. Finally, the overall mean TTR results for the granular applied at the high rate (0.4 lb ai/A) were 0.0062 µg/cm² and 0.0016 µg/cm² for CR and MCR, respectively. Examination of Table 9 shows that residues were below the LOD for the majority of samples collected after application of the granular formulation at the low rate.

Table 9. Mean transferable turf residue results

Trial	Application		California Roller ^a		Modified California Roller ^a	
	Date	Rate	Liquid ($\mu\text{g}/\text{cm}^2$)	Granular ($\mu\text{g}/\text{cm}^2$)	Liquid ($\mu\text{g}/\text{cm}^2$)	Granular ($\mu\text{g}/\text{cm}^2$)
1	9/23/2004	Low	0.0111	0.0008 ^b	0.0068	0.0003 ^b
		High ^c	0.0185	0.0008 ^b	0.0092	0.0003 ^b
2	9/24/2004	Low ^c	0.0008 ^b	0.0024 ^d	0.0010	0.0008 ^d
		High ^c	0.0066	0.0032	0.0033	0.0009
3	10/6/2004	Low	0.0089	0.0014 ^d	0.0040	0.0004 ^d
		High	0.0398	0.0177	0.0199	0.0053
4	10/7/2004	Low	0.0140	0.0008 ^b	0.0037	0.0011 ^d
		High	0.0362	0.0071	0.0124	0.0014
5	10/14/2004	Low	0.0109	0.0008 ^b	0.0069	0.0048 ^d
		High	0.0478	0.0022 ^d	0.0267	0.0003 ^b
Grand Mean		Low	0.0091	0.0012	0.0045	0.0015
		High	0.0298	0.0062	0.0143	0.0016

^a Each value is the mean of duplicate samples.

^b Both duplicate samples were below the 3.0 $\mu\text{g}/\text{sample}$ limit of detection (LOD). One-half of the LOD (1.5 $\mu\text{g}/\text{sample}$) was substituted for both samples in calculating the mean.

^c Application rate differed from target rate (see Table 3 for liquid, Table 5 for granular).

^d One of the duplicate samples was below the 3.0 $\mu\text{g}/\text{sample}$ LOD. One-half of the LOD (1.5 $\mu\text{g}/\text{sample}$) was substituted for that sample in calculating the mean.

The ANOVA on the restricted dataset (omitting liquid Trials 1 and 2 and granular Trials 1 and 5) showed significant effects of both formulation and method on the sensitivity of TTR measurement (Table 10). There were no significant interactions, nor was there a significant effect of rate.

The ANOVA results mean that the CR method is significantly more sensitive than the MCR method. In other words, for a given amount of active ingredient applied to turf, the CR method finds a greater amount of TTR. The absence of significant interactions means that the difference between methods is the same for both formulations and both application rates. In addition, sensitivity is greater with the liquid formulation than the granular. The absence of significant interactions means that the difference between formulations is the same for both methods and both application rates. Figure 1 shows the significant effects graphically.

The ANOVA on the full dataset also showed significant effects of both formulation and method on the sensitivity of TTR measurement (Table 11). The formulation by method interaction was not significant.

These results support conclusions drawn from the first ANOVA, first showing that sensitivity (detection of residues at a particular application rate) is greater with the CR than with the MCR method. The absence of significant interaction suggests that within each application method, the variance in method sensitivity was not significantly greater for either formulation nor for the high or low application rates. The difference in sensitivity between the two methods suggests

that it is not appropriate to pool data from samples collected with MCR and the CR method tested; furthermore, other CR methods should be compared with MCR before pooling data.

In addition, sensitivity is greater with the liquid formulation than the granular, and the absence of significant interaction suggests that within each formulation, the variance in method sensitivity was not significantly greater for either TTR method. This difference in sensitivity suggests that it may not be appropriate to pool data from liquid and granular formulations. Figure 2 shows these significant effects graphically.

Table 10. Analysis of variance table for dependent variable $\ln(\mu\text{g}/\text{cm}^2 \text{ per lb ai/A})$ normalized to target application rate^a

Source of Variance ^b	df	Mean Square	Error Term	F Value	p
<i>Between</i>					
Formulation	1	23.747	Subplots (Formulation x Rate)	49.4	< 0.001
Rate	1	0.0185	Subplots (Formulation x Rate)	0.04	> 0.10
Formulation x Rate	1	0.00094	Subplots (Formulation x Rate)	0.00	> 0.10
Subplots (Formulation x Rate)	8	0.48072			
<i>Within</i>					
Method	1	5.0007	Subplots x Method (Formulation x Rate)	42.6	< 0.001
Formulation x Method	1	0.05294	Subplots x Method (Formulation x Rate)	0.45	> 0.10
Rate x Method	1	0.13394	Subplots x Method (Formulation x Rate)	1.14	> 0.10
Formulation x Rate x Method	1	0.21037	Subplots x Method (Formulation x Rate)	1.79	> 0.10
Subplots x Method (Formulation x Rate)	8	0.11727			
<i>Total</i>	23				
<p>a This ANOVA used only the data from liquid formulation Trials 3, 4 and 5 for and granular Trials 2, 3 and 4. $\mu\text{g}/\text{cm}^2$ = micrograms/square centimeter; ai/A = active ingredient per acre</p> <p>b Sources of variance and their error terms for the mixed design with two between-, one within-units variables are given in Myers (1972, p. 206).</p>					

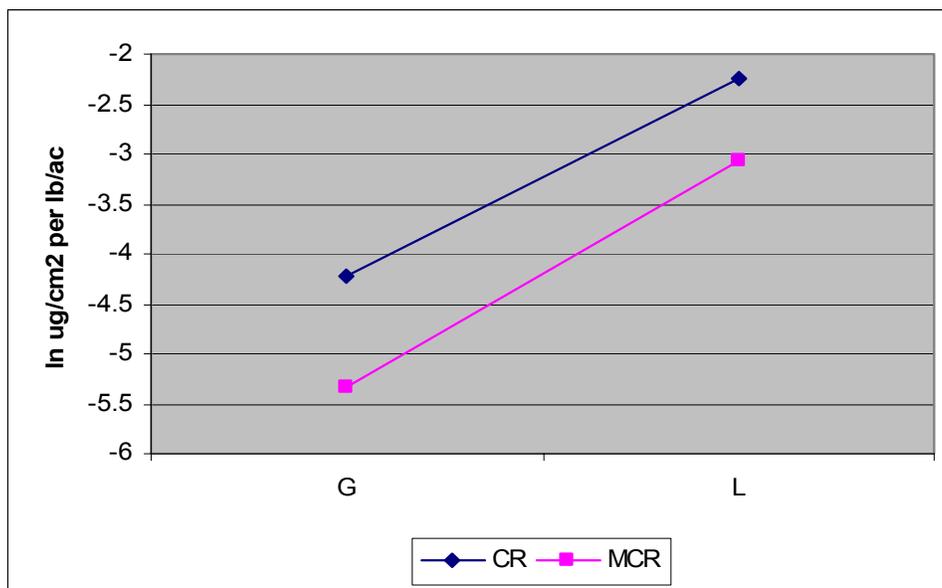
Table 11. Analysis of variance table for dependent variable $\ln(\mu\text{g}/\text{cm}^2 \text{ per lb ai/A})$ normalized to measured application^a

Source of Variance ^b	df	Mean Square	Error Term	F Value	p
<i>Between</i>					
Formulation	1	66.051	Subplots (Formulation)	93.6	< 0.001
Subplots (Formulation)	18	0.7059			
<i>Within</i>					
Method	1	6.3486	Subplots x Method (Formulation)	22.7	< 0.001
Formulation x Method	1	0.07284	Subplots x Method (Formulation)	0.26	> 0.10
Subplots x Method (Formulation)	18	0.280			
<i>Total</i>	39				

a This ANOVA used the data from all applications.

b Sources of variance and their error terms for the mixed design with one between-, one within-units variables are given in Myers (1972, p. 195).

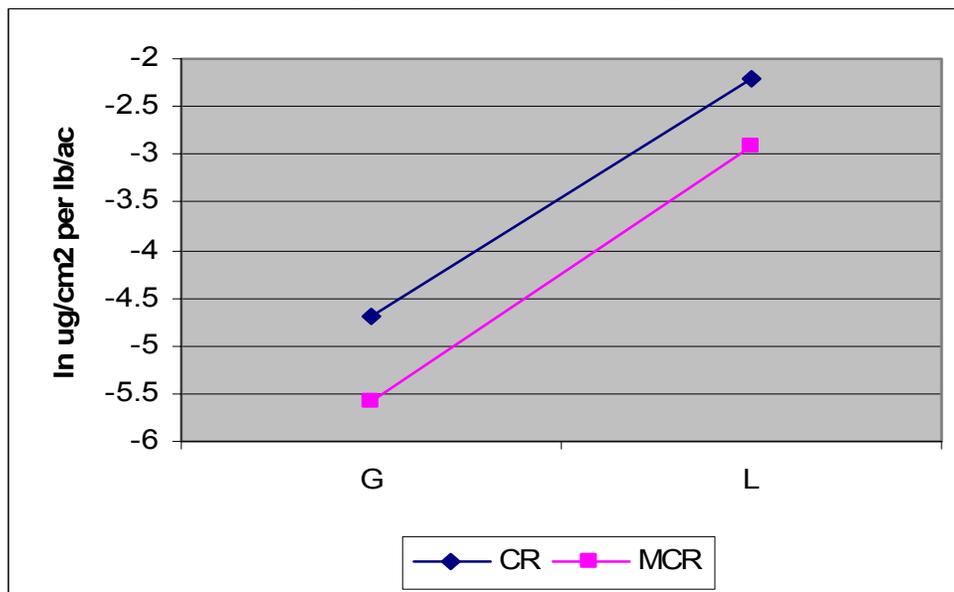
Figure 1. Significant effects of formulation (granular vs. liquid) on transferable turf residue (TTR) method (California Roller vs. Modified California Roller) on sensitivity of TTR measurement analysis of variance on limited dataset^a



^a TTR is normalized to target application rate; plotted points are means of all applications at both application rates. First ANOVA is based on a limited dataset (without liquid Trial 1 and 2 and granular Trial 1 and 5)

G – Granular; L – Liquid; CR – California Roller; MCR – Modified

Figure 2. Significant effects of formulation (granular vs. liquid) on transferable turf residue (TTR) method (California Roller vs. Modified California Roller) on sensitivity of TTR measurement from analysis of variance on complete^a dataset



^a TTR is normalized to actual application rate; plotted points are means of all applications at all application rates.

G – Granular; L – Liquid; CR – California Roller; MCR – Modified California Roller

Discussion

The study found no significant main or interaction effects of application rate. If the sensitivity of a TTR method varied depending on how much ai were applied (main effect), and especially if it varied more with one measurement method or one formulation than the other (interaction effects), it would signal a fundamental problem with the methodology.

The study did find that sensitivity is higher for liquid than for granular formulations (significant main effect of formulation). This was seen previously in the ORETF Moses Lake study and the Rosenheck *et al.*, (2001a) methods study (Table 12). (One previous study (Klonne *et al.*, 2001b) found higher sensitivity for granular formulation.) This finding suggests that it *may* not be appropriate to pool data from liquid and granular formulations. Further research is needed to determine whether the observed difference is real, i.e., whether there actually *is* less transferable residue with granular formulation. The ultimate question is whether the relationship of dermal exposure to TTR is the same for liquid and granular formulation. Answering that question will require collecting dermal exposure data along with TTR measurements to determine whether dermal exposure is proportionately lower with granular formulations. The ORETF Moses Lake study suggested that may be the case; both measured TTR and dermal exposure were lower with granular product. However, the range of TTR values represented in that study was insufficient to characterize the quantitative relationship of dermal exposure to TTR for granular formulation, or to support a conclusion about whether that relationship is the same as for liquid formulation.

Table 12. Sensitivity of California Roller (CR) and Modified California Roller (MCR) in transferable turf methods comparison and Moses Lake studies

Study	Method	$\mu\text{g} / \text{m}^2$ per lb ai /acre			
		Day 0		Day 1	
		Liquid	Granular	Liquid	Granular
Moses Lake CHAPS ^{a,b}	MCR	120	3.9	47	3.2
Moses Lake Jazz ^{a,b}	MCR	212	6.4	57	4.1
Rosenheck <i>et al.</i> , 2001 ^b	MCR	496	44	95	50
DPR Study 0404 ^c	MCR	--	--	565	50
DPR Study 0404 ^c	CR	--	--	1117	141
Klonne <i>et al.</i> , 2001 ^b	CR	389	439	5.0	14

a For Moses Lake, Day 0 = Session 1, Day 1 = Session 2 (both sessions were on Day 0).

b Applications of dithiopyr at 0.5 lb/ac.

c Applications of imidacloprid at 0.01 to 0.4 lb/ac.

No previous study has directly compared the CR and MCR methods. The current study found a significant main effect of method, with the CR method giving TTR values averaging 2-3 times higher than the MCR method in side-by-side samples (Table 12). Within each application method, the variance in method sensitivity was not significantly greater for either formulation nor for the high or low application rates. This difference in sensitivity suggests that it is *not* appropriate to pool data from CR and MCR measurements. Moreover, given the differences we noticed between previous studies in implementation of the CR method, it may not be appropriate to pool the data from those studies. The ultimate question, as before, is the relationship of dermal exposure to TTR. Although dermal exposure was not measured in this study, if it had been measured, the result would have been one set of dermal exposures from each treated plot and two sets of differing TTR values. The only possible conclusion is that TTR measurements by the CR method we tested and the MCR method are incommensurable.

Further Research

There may exist sufficient data to characterize the relationship of dermal exposure (DE) to TTR for liquid formulations. Data are certainly needed to characterize the relationship of DE to TTR for granular formulations. TTR should be measured using the CR method, both in order that it be commensurable with existing data on liquid formulations and in order to ensure positive detections on most samples. A wide range of application rates (wider than in the current study) should be used in order to allow quantifying the relationship of DE to TTR. Exposure may be measured using Jazzercise, since the Moses Lake study established that Jazzercise and CHAPS give similar results on a per-unit-time basis. Exposure should be monitored for the T-shirt and short pants clothing scenario, since that is the scenario of concern in outdoor residential exposure.

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Disclaimer

Product or company names are for providing specific information only. Their mention does not imply an endorsement or recommendation over others not mentioned.

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Appendix 1. Protocol Amendments and Protocol, SOP and GLP Deviations

Protocol Amendments

Amendment No.	Amendment Date ^a	Original Protocol Requirement	Amendment	Effect on Study
1	Sept. 13	“In order to provide a uniform distribution of pesticide, the liquid formulation will be applied with ground-rig boom equipment and the granular formulation with a drop-type or rotary-type spreader.”	The study director will use a walk-behind broadcast spreader to deliver the granular material.	Probably no effect. It may be more difficult to accurately calibrate the broadcast spreader.
2	Sept. 13	The CR as used in Bernard, <i>et al.</i> , 2001 will be provided by R.I. Krieger.	The CR was fabricated by WH&S staff using the design specifications in Bernard, <i>et al.</i> , 2001.	None.
3	Sept. 15	Carefully place the cloth on the sampling plot. Do not adjust or move the sampling medium once it hits the turf as this will result in the cotton cloth coming into contact with a greater surface area than 1800 cm ² . Place the plastic sheeting over the cloth so that it completely covers the cloth.” “The plastic sheeting will be lifted from the turf and discarded. Next the cloth is lifted from the plot and inspected for visible debris (grass clippings and thatch) that is carefully removed.	The CR sampling media is preassembled using pins to keep the cloth and plastic together.	Positive effect. During the prestudy practice run, the cloth moved around under the plastic as the roller ran over the top of the plastic.
4	Sept. 13	The protocol states sample collection will be approximately 18 hours post application on page 18, but not on page 6, 21.	The sample collection will commence approximately 18 hours after application. The exact timing will be documented.	No effect, the study director added approximately 18 hours in one location and neglected to correct the other two locations in the protocol.
5	Sept. 15	The preassembled MCR sample media and frames will be stored in plastic boxes.	Plastic boxes of the necessary size could not be found. We will use the cardboard boxes that the frames were shipped in.	No effect.

Appendix 1 (continued)

Amendment No.	Amendment Date	Original Protocol Requirement	Amendment	Effect on Study
6	Sept. 15	Changes are needed in preassembling the CR sample media and thus the sample collection procedures. The field conditions suggested other changes were needed to ensure sound science and eliminate contamination.	Changes are needed in preassembling the CR sample media and thus the sample collection procedures. The field conditions suggested other changes were needed.	Positive effect. During the prestudy practice run, the cloth moved around under the plastic as the roller ran over the top of the plastic

^a All dates refer to 2004.

SOP and GLP Deviations

Deviation No.	Trial No.	Date Occurred ^a	Requirement	Deviation	Effect on Study ^b
1	2	Sept. 23	Protocol. Tank mix samples will be stored on wet ice in glass bottles.	For Trial #2, the tank mix samples were collected in polypropylene bottles.	Probably no effect. Merit [®] 2 Insecticide, is sold in a plastic container.
2	4	Oct. 6	Protocol. The following information will be recorded for each application: The volume of tank mix or weight of granules remaining after each application and total amount of product and active ingredient used for each treatment.	For Trial #4, a known amount was put into the granular spreader. After application, the left over material was poured from the spreader onto a plastic sheet. While attempting to transfer from the plastic sheet to a container for weighing, the granules spilled on the ground.	We will not know the actual amount of imidacloprid used for the granular high application rate. However, we will be able to make estimates based on the timing of the application and on the material found in the deposition samples.
3	2 - 5	Sept. 24 Oct. 6, 7 Oct. 14	Protocol. For the MCR, the sample media cloth will be cut to 27" x 39" (24.5 x 36" exposed area) and the plastic will be cut 29" x 41".	The cloth and plastic for the MCR was cut slightly larger to keep them from pulling out of the frames. The exposed area of the cloth remained the same.	This deviation should have little effect on the study. The exposed area of the cloth remains the same, however, there may be some minor effect on the laboratory quantification limits as the cloth itself is slightly larger.

Appendix 1 (continued)

Deviation No.	Trial No.	Date Occurred ^a	Requirement	Deviation	Effect on Study ^b
4	1 - 5	Sept. 13	Protocol. Study staff (Pest Control Advisor & Pest Control Operator) will calibrate the equipment before the first application.	The manager of the sod farm and applicator of the liquid formulation calibrated the equipment for applying the liquid formulation.	The deviation should have no effect on the study. The equipment was calibrated by an experienced applicator.
5	2	Sept. 23	Protocol. “The application rates are the highest allowed label rate (full-rate) of 0.4 lb of active ingredient per acre and a low rate 0.1 lb active ingredient per acre.	For Trial #2, the liquid formulation application rates were applied at lower rates than in the protocol. Both the high and low rates were miscalculated by 8 to 10-fold	It was originally thought that the study results could not be used. However, the findings of the statistical analysis on a limited dataset (without this data) determined that data from this trial could be used.
6	3 - 5	Oct. 5, 6, 13	Protocol. Prior to granular (G) application 3 deposition pans (aluminum) will be place on each turf subplot. ...The granules from the pans will be carefully transferred to a 1-pint glass jar. The jar will be placed in the deposition cooler containing dry ice.	For Trials 3 – 5, plastic containers were used for collection of granular deposition samples. The granules were left in the plastic containers before weighing and not transferred to a glass jar. The plastic containers were not placed on dry ice	The deviation probably had a positive effect on the study. The granules may have bounced out of the deposition pans in Trial 1. For Trial 2, scientists lined the pans with aluminum pans with foil. For Trials 3 - 5 plastic containers were used and no transfer was necessary. Granular deposition pans were not placed on dry ice. This will have no effect on the study, as formulated product was not frozen prior to use. Frozen storage is not required to maintain product stability.

Appendix 1 (continued)

Deviation No.	Trial No.	Date Occurred ^a	Requirement	Deviation	Effect on Study ^b
7	1 - 5	Sept. 23, 24 Oct. 6, 7, 14	Protocol says “Duplicate samples per day at 3 levels plus a solvent blank will be prepared, labeled, stored & shipped in same manner as TTR samples” – “A solution of certified primary reference standard of known concentration will be poured onto a cotton sheet (folded in half) from a glass ampoule”	Scientist prepared 2 solvent blanks each sample collection day. The cotton sheets were folded in half twice <u>prior</u> to spiking with reference standards or the solvent blank	Deviation probably has no effect on the study.
8	1 - 5	Sept. 23, 24 Oct. 6, 7, 14	Protocol. “Study personnel will record minimum and maximum daily temperatures on site for the study period.	Site-specific maximum and minimum temperatures were not recorded.	Deviation has no effect on the study. Hourly weather data was collected from a nearby weather station.
9	1 - 5	Sept. 23, 24 Oct. 6, 7, 14	SOP WHS-FO08. Use a single line to cross out wrong entries so as not to obscure the original entry. Date and initial each error at the time of the change; include the appropriate error code in the notation.	Corrections were made on the spot to correct wrong entries. Many of the scratch out marks were initialed and dated at the time they occurred; however, some were not and some changes were made illegible.	No effect on study. Results of event timing were not used in the study results.

Appendix 1 (continued)

Deviation No.	Trial No.	Date Occurred ^a	Requirement	Deviation	Effect on Study ^b
10	1 - 5	Sept. 23, 24 Oct. 6, 7, 14	SOP WHS-PS02. "Each individual involved in a GLP study, including management, supervisors, study directors, field personnel, QAU staff, and support staff must have adequate training, education, experience or an appropriate combination thereof, to properly carry out their assigned responsibilities." In addition the SOP requires "All individuals conducting GLP studies must have on file a current record of training, education and experience."	Some staff involved did not have GLP training or GLP training records on file.	No effect on study. All staff had years of field research experience, knew how to do their jobs in this study and met the requirements of their duty statements. Staff not trained in GLP made corrections to the sample collection timing data and completely obliterated the original mark. In a few locations, this was not initialed and dated.
11	5	Oct. 13	Protocol. "The following information will be recorded for each application: ..." "g. Time required to traverse each plot."	In Trial SS05, the granular applications were not timed.	Deviation probably has no effect on the study. We have the actual weight of the amount of granular material applied. The timing is a check on the amount applied and it is not essential to the study when we know the amount applied.
12	5	Oct. 13	Protocol. "Applications will be timed so that intervals between treatments will match the time required for sampling (approximately 18 hours)."	In Trial 5, the grass was too wet at 18 hours. So sample collection was initiated at approximately 21 hours post application, when the turf was dry.	Deviation may affect the study results. The difference in the timing of sample collection will be accounted for during the data analysis. Sample collection while grass was wet may have resulted in biased sample collection.

Appendix 1 (continued)

Deviation No.	Trial No.	Date Occurred ^a	Requirement	Deviation	Effect on Study ^b
13	1	Sept. 22	Protocol. “The application rates are the highest allowed label rate (full-rate) of 0.4 lb of active ingredient per acre and a low rate 0.1 lb active ingredient per acre.	For Trial 1, the high, liquid formulation application rate was applied at lower rates than described in the protocol.	It was originally thought that the study results could not be used. However, the findings of the statistical analysis on a limited dataset (without this data) determined that data from this trial could be used.
14	1-5	Through out study	Protocol. The study director was to provide the CDFFA/CAC QAU with a copy of the protocol. The QAU was to conduct several audits during various portions of the sample analyses. Lab QAU was to sign a quality assurance statement for the laboratory portion of the study.	The PAI provided the lab QAU with the protocol. In-process audits were not completed. No audits performed, no compliance statement written.	Unknown effect on the study. Previous audits of the CDFFA/CAC QAU have been very positive. However, for this study, compliance with the protocol, BP, SOPs, etc. is unknown, with the exception of the method validation report (had QAU review and approval).
15	1	Sept. 22	Protocol. “The application rates are the highest allowed label rate (full-rate) of 0.4 lb of active ingredient per acre and a low rate 0.1 lb active ingredient per acre.	For Trial #1, the high liquid formulation application rate was applied at lower rates than in the protocol.	It was originally thought that the study results could not be used. However, the findings of the statistical analysis on a limited dataset (without this data) determined that data from this trial could be used.

^a All dates are in 2004.

^b Since no human subjects are involved in this study, the deviation does not affect study subjects.

Appendix 2. Daily Meteorological Summary (Sacramento Valley, CIMIS Station 6, Davis)

Date ^a	Air Temperature (°F)		Average Wind Speed (MPH)	Precipitation (inches)	Relative Humidity (%)	
	Maximum	Minimum			Maximum	Minimum
9/22/2004	84.5	47.3	2.8	0	70	18
9/23/2004	88.5	--	2.6	0	59	16
9/24/2004	92.8	51.3	3.1	0	57	15
10/5/2004	84.2	46.7	3.3	0	89	34
10/6/2004	86.9	50	3.4	0	84	29
10/7/2004	84.9	50.6	3.7	0	85	34
10/13/2004	94.2	54.4	4.6	0	48	15
10/14/2004	77.5	51.1	2.1	0	70	35

^a Trial 1: application – 9/22; sample collection – 9/23
 Trial 2: application – 9/23; sample collection – 9/24
 Trial 3: application – 10/5; sample collection – 10/6
 Trial 4: application – 10/6; sample collection – 10/7
 Trial 5: application – 10/13; sample collection – 10/14

Appendix 3. Sampling Method/Technique Specifications

Modified California Roller Specifications

Roller Construction

Weight - 32 lbs (±1 lb)
 Length – 24”
 Diameter 4” (without PUF covering)
 PUF covering outside
 ~48” handle

Dosimeters

27” x 39” (24.5” x 36” exposed area)
 100% cotton cloth
 Plastic sheeting to cover cloth during sampling (29” x 41”)

Frame

¼” thick flat plastic frame with an open area of 24.5” x 36”
 Clamps and angle iron on the top and bottom
 Small single point clamp about ½ way on each side of the frame

California Roller Specifications

Roller Construction

Weight - 30 lbs
 Length – 12” (sampling distance)
 Diameter – 4” (without PUF covering)
 PUF covering outside
 ~24” handle

Dosimeters

23.6" x 11.8" (entire area exposed)

100% cotton cloth

Plastic sheeting to cover cloth during sampling (26" x 14")

Frame - None

Sampling Order

- The order of the application of blocks will be randomized.
- Sampling will commence 18 hours after the application.
- Each block will be sampled in the order in which it was treated.

Sample Collection

- Each sampling team will consist of 3 people, each responsible for one of the following: Sample rolling (Person A), sample assembly placement/pickup (Person B) and sample processing (Person C).
- At each sample interval one CR and one MCR sampling will be conducted simultaneously from a single block within a subplot. Two of each sample type will be collected from each subplot. Each team will be responsible for one sample type.
- MCR Sample Collection:
 1. Person B, wearing clean latex or vinyl gloves, carefully places the frame on the sampling area face down, so that the cotton cloth touches the turf. The frame assembly should not be adjusted or moved once it is placed on the turf.
 2. Use spikes to secure the frame on the plot. Change to clean gloves for step 5.
 3. Person A places the MCR roller on the MCR frame assembly. Gently and evenly (do not add downward pressure) move the roller over the frame five times to capture transferable residues. One forward and backward motion is considered one roll.
 4. After rolling, the roller is picked up by Person A and taken to a clean area to prevent possible contamination. The roller should not directly contact the turf between samples.
 5. Person B (with clean gloves on) will gently lift the frame from the turf. Set the frame, cloth side up on a piece of cardboard (covered with clean butcher paper) and carry it to the sample-processing table. The table will also be covered with clean butcher paper.
 6. At the table, two different staff (Person C from both sampling teams) wearing clean gloves will inspect the cloth for visible debris (grass clippings and thatch) that is carefully removed with clean tweezers. Granules that stayed attached to the sheet cloth, when frame was lifted and turned to place it on the cardboard, will not be removed. Do not shake the sheet and frame to remove debris. Tweezers will be cleaned with alcohol each time, at the end of this step.
 7. Unfasten the top end clamps allowing the cotton sheet to fold in the middle so that the side in contact with the turf is folded together.
 8. Release the center and bottom end clamps and remove the cotton sheet from the frame. Fold the cotton sheet in half two more times and place the sheet in the appropriately labeled 1-quart glass jar.
 9. Place the sample jar in a cooler containing dry ice.
 10. The MCR frame is set-aside on a box. (It will be cleaned at the end of the day.)

- CR Sample Collection

1. Person B, wearing clean latex or vinyl gloves, will carefully place the cotton sheet on the sampling plot; do not adjust the location once it touches the turf. To get the cotton sheet evenly placed on the turf, this may take two people (Person C may need to help out here). Next place the plastic sheet over the cotton sheet so that the cotton sheet is centered under the plastic.
2. Person B uses spikes to secure the plastic to the plot; one at each corner. Change to clean gloves in order to be ready for step 5.
3. Person A gently places the CR roller on the CR sampling assembly. Gently and evenly (do not add downward pressure) move the roller over the assembly 20 times to capture transferable residues. One forward and backward motion is considered one roll.
4. After rolling, the roller is picked up by Person A and taken to a clean area to prevent possible contamination. The roller should not directly contact the turf between samples.
5. Person B (with clean gloves on) will gently lift the plastic from the turf and discard it. Then lift the cloth from the turf, turn it over and place it exposed side up on a piece of cardboard that has been covered with clean butcher paper. Carry it to the sample-processing table. You may need to hold the corners of the sheet if the wind is blowing. Touch as little of the cloth as possible. The table will also be covered with clean butcher paper.
6. Two different staff (Person C from both sampling teams) wearing clean gloves will inspect the cloth for visible debris (grass clippings and thatch) that is carefully removed with clean tweezers. Granules that stayed attached to the sheet cloth, when it was lifted and turned to place it on the cardboard, will not be removed. The sheet should not be shaken in order to remove debris. Used tweezers will be placed aside and cleaned at the end of the day.
7. Gently fold the cotton sheet in the middle so that the side in contact with the turf is together. Fold the sheet in half two more times so that it will fit into a quart glass jar.
8. Place the sheet in the appropriately labeled glass quart jar.
9. Place the sample jar in a cooler containing dry ice.

- Sampling of Next Block

1. Person A from each sampling team (MCR and CR) will switch duties.
2. All other personnel will conduct the same activities through out the sample collection period.

Equipment Cleaning and Preparation

- All used frames will be thoroughly cleaned with a solvent (alcohol) wash to ensure no contaminating residues remain on the frame for the next day sample.
- Clean all tweezers with a solvent to ensure no residues remain on the frame.
- Rollers should not need to be cleaned. However, if one accidentally touches the treated turf, wash the entire roller assembly thoroughly with alcohol.

Appendix 4. Liquid Formulation Deposition Indexed by Trial

Trial	Application Rate	Rep	Lab No.	WH&S Sample No.	Date	Imidacloprid $\mu\text{g}/\text{sample}$
1	Low	1	04-0318	SS01-1017	9/23/2004	50.9
		2	04-0319	SS01-1018	9/23/2004	85.9
		3	04-0320	SS01-1019	9/23/2004	73.1
	High	1	04-0321	SS01-1020	9/23/2004	150
		2	04-0322	SS01-1021	9/23/2004	130
		3	04-0323	SS01-1022	9/23/2004	156
2	Low	1	04-0364	SS02-2017	9/24/2004	16.6
		2	04-0365	SS02-2018	9/24/2004	18.1
		3	04-0366	SS02-2019	9/24/2004	16.4
	High	1	04-0367	SS02-2020	9/24/2004	56.6
		2	04-0368	SS02-2021	9/24/2004	49.0
		3	04-0369	SS02-2022	9/24/2004	48.4
3	Low	1	04-0438	SS03-3017	10/6/2004	67.9
		2	04-0439	SS03-3018	10/6/2004	76.7
		3	04-0440	SS03-3019	10/6/2004	60.5
	High	1	04-0441	SS03-3020	10/6/2004	254
		2	04-0442	SS03-3021	10/6/2004	238
		3	04-0443	SS03-3022	10/6/2004	363
4	Low	1	04-0482	SS04-4017	10/7/2004	132
		2	04-0483	SS04-4018	10/7/2004	97.6
		3	04-0484	SS04-4019	10/7/2004	102
	High	1	04-0485	SS04-4020	10/7/2004	577
		2	04-0486	SS04-4021	10/7/2004	457
		3	04-0487	SS04-4022	10/7/2004	329
5	Low	1	04-0533	SS05-5017	10/14/2004	102
		2	04-0534	SS05-5018	10/14/2004	100
		3	04-0535	SS05-5019	10/14/2004	105
	High	1	04-0536	SS05-5020	10/14/2004	232
		2	04-0537	SS05-5021	10/14/2004	367
		3	04-0538	SS05-5022	10/14/2004	398

Appendix 5. Granular Deposition Raw Data Indexed by Trial

Trial No.	Application Rate	Sample ID	Container Size (cm ²)	Granular Net Weight (mg)
1	Low	SS01-1023	412.8	125.94
		SS01-1024	412.8	74.46
		SS01-1025	412.8	88.16
	High	SS01-1026	412.8	no sample
		SS01-1027	412.8	293.54
		SS01-1028	412.8	157.54
2	Low	SS02-2023	412.8	149.65
		SS02-2024	412.8	52.82
		SS02-2025	412.8	206.50
	High	SS02-2026	412.8	287.08
		SS02-2027	412.8	354.74
		SS02-2028	412.8	511.53
3	Low	SS03-3023	195.1	29.67
		SS03-3024	195.1	23.78
		SS03-3025	195.1	33.11
	High	SS03-3026	195.1	164.54
		SS03-3027	195.1	78.39
		SS03-3028	195.1	194.69
4	Low	SS04-4023	195.1	89.48
		SS04-4024	195.1	45.33
		SS04-4025	195.1	22.21
	High	SS04-4026	195.1	205.34
		SS04-4027	195.1	179.00
		SS04-4028	195.1	199.39
5	Low	SS05-5023	195.1	114.44
		SS05-5024	195.1	18.81
		SS05-5025	195.1	47.90
	High	SS05-5026	195.1	189.69
		SS05-5027	195.1	255.48
		SS05-5028	195.1	361.59

Appendix 6. Ampoule Fortification Analysis

Ampoule Fortification µg/sample	Lab ID	Sample ID	Date	Imidacloprid µg/sample
20	04-0562	SS05-5054	10/14/2004	18.5
200	04-0561	SS05-5053	10/14/2004	190
2000	04-0563	SS05-5055	10/14/2004	1828

Appendix 7. Field Fortification Raw Data Indexed by Application Number

Trial	Fortification µg/sample	Rep	Lab No.	WH&S Sample No.	Date	Imidacloprid µg/sample
1	20	1	04-0312	SS01-1011	9/23/2004	13.9
		2	04-0313	SS01-1012	9/23/2004	13.3
	200	1	04-0314	SS01-1013	9/23/2004	183
		2	04-0315	SS01-1014	9/23/2004	192
	2000	1	04-0316	SS01-1015	9/23/2004	1890
		2	04-0317	SS01-1016	9/23/2004	1874
2	20	1	04-0358	SS02-2011	9/24/2004	20.0
		2	04-0359	SS02-2012	9/24/2004	18.2
	200	1	04-0360	SS02-2013	9/24/2004	205
		2	04-0361	SS02-2014	9/24/2004	178
	2000	1	04-0362	SS02-2015	9/24/2004	2187
		2	04-0363	SS02-2016	9/24/2004	1869
3	20	1	04-0432	SS03-3011	10/6/2004	19.5
		2	04-0433	SS03-3012	10/6/2004	20.7
	200	1	04-0434	SS03-3013	10/6/2004	203
		2	04-0435	SS03-3014	10/6/2004	187
	2000	1	04-0436	SS03-3015	10/6/2004	1961
		2	04-0437	SS03-3016	10/6/2004	2117
4	20	1	04-0476	SS04-4011	10/7/2004	18.4
		2	04-0477	SS04-4012	10/7/2004	18.4
	200	1	04-0478	SS04-4013	10/7/2004	195
		2	04-0479	SS04-4014	10/7/2004	195
	2000	1	04-0480	SS04-4015	10/7/2004	1958
		2	04-0481	SS04-4016	10/7/2004	1895
5	20	1	04-0527	SS05-5011	10/14/2004	18.2
		2	04-0528	SS05-5012	10/14/2004	18.9
	200	1	04-0529	SS05-5013	10/14/2004	214
		2	04-0530	SS05-5014	10/14/2004	188
	2000	1	04-0531	SS05-5015	10/14/2004	2054
		2	04-0532	SS05-5016	10/14/2004	1901

Appendix 8. Transferable Turf Residue Raw Data Indexed By Trial

Trial	Application		Roller Method ^b	Rep	Sample No.	Sample Collection Date	Imidacloprid ^c (µg/sample)
	Rate	Formulation ^a					
1	Low	L	CR	1	SS01-1033	9/23/2004	20.7
				2	SS01-1034	9/23/2004	19.2
			MCR	1	SS01-1035	9/23/2004	32.4
				2	SS01-1036	9/23/2004	45.4
	High	L	CR	1	SS01-1037	9/23/2004	28.7
				2	SS01-1038	9/23/2004	38
			MCR	1	SS01-1039	9/23/2004	39.2
				2	SS01-1040	9/23/2004	65.1
	Low	G	CR	1	SS01-1041	9/23/2004	ND
				2	SS01-1042	9/23/2004	ND
			MCR	1	SS01-1043	9/23/2004	ND
				2	SS01-1044	9/23/2004	ND
	High	G	CR	1	SS01-1045	9/23/2004	ND
				2	SS01-1046	9/23/2004	ND
			MCR	1	SS01-1047	9/23/2004	ND
				2	SS01-1048	9/23/2004	ND
2	Low	L	CR	1	SS02-2033	9/24/2004	ND
				2	SS02-2034	9/24/2004	ND
			MCR	1	SS02-2035	9/24/2004	4.86
				2	SS02-2036	9/24/2004	6.66
	High	L	CR	1	SS02-2037	9/24/2004	10.5
				2	SS02-2038	9/24/2004	13.4
			MCR	1	SS02-2039	9/24/2004	17
				2	SS02-2040	9/24/2004	20.5
	Low	G	CR	1	SS02-2041	9/24/2004	7.3
				2	SS02-2042	9/24/2004	ND
			MCR	1	SS02-2043	9/24/2004	ND
				2	SS02-2044	9/24/2004	7.47
	High	G	CR	1	SS02-2045	9/24/2004	3.36
				2	SS02-2046	9/24/2004	8.02
			MCR	1	SS02-2047	9/24/2004	7.56
				2	SS02-2048	9/24/2004	3.11
3	Low	L	CR	1	SS03-3033	10/6/2004	14.1
				2	SS03-3034	10/6/2004	18.1
			MCR	1	SS03-3035	10/6/2004	20.3
				2	SS03-3036	10/6/2004	25.7
	High	L	CR	1	SS03-3037	10/6/2004	58.4
				2	SS03-3038	10/6/2004	84.7
			MCR	1	SS03-3039	10/6/2004	118
				2	SS03-3040	10/6/2004	109
	Low	G	CR	1	SS03-3041	10/6/2004	ND
				2	SS03-3042	10/6/2004	3.36
			MCR	1	SS03-3043	10/6/2004	3.5
				2	SS03-3044	10/6/2004	ND
	High	G	CR	1	SS03-3045	10/6/2004	31
				2	SS03-3046	10/6/2004	32.6
			MCR	1	SS03-3047	10/6/2004	20.6
				2	SS03-3048	10/6/2004	39.9

Appendix 8, continued.

Trial	Application		Roller Method ^b	Rep	Sample No.	Sample Collection Date	Imidacloprid ^c µg/sample
	Rate	Formulation ^a					
4	Low	L	CR	1	SS04-4033	10/7/2004	27
				2	SS04-4034	10/7/2004	23.5
			MCR	1	SS04-4035	10/7/2004	20.7
				2	SS04-4036	10/7/2004	21.9
	High	L	CR	1	SS04-4037	10/7/2004	60.3
				2	SS04-4038	10/7/2004	70
			MCR	1	SS04-4039	10/7/2004	41
				2	SS04-4040	10/7/2004	100
	Low	G	CR	1	SS04-4041	10/7/2004	ND
				2	SS04-4042	10/7/2004	ND
			MCR	1	SS04-4043	10/7/2004	ND
				2	SS04-4044	10/7/2004	10.6
	High	G	CR	1	SS04-4045	10/7/2004	14.7
				2	SS04-4046	10/7/2004	10.7
			MCR	1	SS04-4047	10/7/2004	12.6
				2	SS04-4048	10/7/2004	3.38
5	Low	L	CR	1	SS05-5033	10/14/2004	17.3
				2	SS05-5034	10/14/2004	22
			MCR	1	SS05-5035	10/14/2004	33.3
				2	SS05-5036	10/14/2004	45.5
	High	L	CR	1	SS05-5037	10/14/2004	82.6
				2	SS05-5038	10/14/2004	89.5
			MCR	1	SS05-5039	10/14/2004	140
				2	SS05-5040	10/14/2004	164
	Low	G	CR	1	SS05-5041	10/14/2004	ND
				2	SS05-5042	10/14/2004	ND
			MCR	1	SS05-5043	10/14/2004	3.9
				2	SS05-5044	10/14/2004	ND
	High	G	CR	1	SS05-5045	10/14/2004	6.5
				2	SS05-5046	10/14/2004	ND
			MCR	1	SS05-5047	10/14/2004	ND
				2	SS05-5048	10/14/2004	ND

^a L = Liquid imidacloprid application; G = Granular imidacloprid application

^b CR = California Roller method; MCR = Modified California Roller method

^c ND = Not detected (limit of detection = 3 µg/sample)

Appendix 9. Pre-Application Sample Raw Data Indexed by Trial

Trial	Application		Roller Method ^b	Lab No.	Sample No.	Date Sampled	Imidacloprid ^c µg/sample
	Formulation ^a	Rate					
1	L	Low	CR	04-0302	SS01-1001	9/23/04	ND
			MCR	04-0303	SS01-1002	9/23/04	ND
		High	CR	04-0304	SS01-1003	9/23/04	ND
			MCR	04-0305	SS01-1004	9/23/04	ND
	G	Low	CR	04-0306	SS01-1005	9/23/04	ND
			MCR	04-0307	SS01-1006	9/23/04	ND
		High	CR	04-0308	SS01-1007	9/23/04	ND
			MCR	04-0309	SS01-1008	9/23/04	ND
2	L	Low	CR	04-0348	SS02-2001	9/23/2004	ND
			MCR	04-0349	SS02-2002	9/23/2004	ND
		High	CR	04-0350	SS02-2003	9/23/2004	ND
			MCR	04-0351	SS02-2004	9/23/2004	ND
	G	Low	CR	04-0352	SS02-2005	9/23/2004	ND
			MCR	04-0353	SS02-2006	9/23/2004	ND
		High	CR	04-0354	SS02-2007	9/23/2004	ND
			MCR	04-0355	SS02-2008	9/23/2004	ND
3	L	Low	CR	04-0422	SS03-3001	10/6/2004	ND
			MCR	04-0423	SS03-3002	10/6/2004	ND
		High	CR	04-0424	SS03-3003	10/6/2004	ND
			MCR	04-0425	SS03-3004	10/6/2004	ND
	G	Low	CR	04-0426	SS03-3005	10/6/2004	ND
			MCR	04-0427	SS03-3006	10/6/2004	ND
		High	CR	04-0428	SS03-3007	10/6/2004	ND
			MCR	04-0429	SS03-3008	10/6/2004	ND
4	L	Low	CR	04-0466	SS04-4001	10/7/2004	ND
			MCR	04-0467	SS04-4002	10/7/2004	ND
		High	CR	04-0468	SS04-4003	10/7/2004	ND
			MCR	04-0469	SS04-4004	10/7/2004	ND
	G	Low	CR	04-0470	SS04-4005	10/7/2004	ND
			MCR	04-0471	SS04-4006	10/7/2004	ND
		High	CR	04-0472	SS04-4007	10/7/2004	ND
			MCR	04-0473	SS04-4008	10/7/2004	ND
5	L	Low	CR	04-0517	SS05-5001	10/14/2004	ND
			MCR	04-0518	SS05-5002	10/14/2004	ND
		High	CR	04-0519	SS05-5003	10/14/2004	ND
			MCR	04-0520	SS05-5004	10/14/2004	ND
	G	Low	CR	04-0521	SS05-5005	10/14/2004	ND
			MCR	04-0522	SS05-5006	10/14/2004	ND
		High	CR	04-0523	SS05-5007	10/14/2004	ND
			MCR	04-0524	SS05-5008	10/14/2004	ND

^a L = Liquid imidacloprid application; G = Granular imidacloprid application

^b CR = California Roller method; MCR = Modified California Roller method

^c ND = Not detected (limit of detection = 3 µg/sample)

Appendix 10. Field Method Blank Raw Data Indexed by Trial

Trial	Roller Method ^a	Rep	Lab No.	WH&S Sample No.	Date	Imidacloprid ^b µg/sample
1	CR	1	04-0344	SS01-1049	9/23/04	ND
		2	04-0345	SS01-1050	9/23/04	ND
	MCR	1	04-0346	SS01-1051	9/23/04	ND
		2	04-0347	SS01-1052	9/23/04	ND
2	CR	1	04-0388	SS02-2049	9/24/2004	3.35
		2	04-0389	SS02-2050	9/24/2004	ND
	MCR	1	04-0390	SS02-2051	9/24/2004	ND
		2	04-0391	SS02-2052	9/24/2004	ND
3	CR	1	04-0462	SS03-3049	10/6/2004	ND
		2	04-0463	SS03-3050	10/6/2004	ND
	MCR	1	04-0464	SS03-3051	10/6/2004	ND
		2	04-0465	SS03-3052	10/6/2004	ND
4	CR	1	04-0507	SS04-4049	10/7/2004	ND
		2	04-0508	SS04-4050	10/7/2004	ND
	MCR	1	04-0509	SS04-4051	10/7/2004	ND
		2	04-0510	SS04-4052	10/7/2004	ND
5	CR	1	04-0557	SS05-5049	10/14/2004	ND
		2	04-0558	SS05-5050	10/14/2004	ND
	MCR	1	04-0559	SS05-5051	10/14/2004	ND
		2	04-0560	SS05-5052	10/14/2004	ND

^a CR = California Roller method; MCR = Modified California Roller method

^b ND = Not detected (limit of detection = 3 µg/sample)