Imidacloprid (Admire®) Residue Levels Following In-furrow Application in Potato Fields in Prince Edward Island and New Brunswick

Final Report

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INTRODUCTION

Imidacloprid (ADMIRE® 240F), is a synthetic systemic chloronicotinyl insecticide, produced for the control of Colorado potato beetles, aphids, flea beetles, and leafhoppers on potato crops ([redacted] et al., 1991; [redacted], 1999). Imidacloprid is an agonist at nicotinic acetylcholine receptors that demonstrates selective toxicity for insects over vertebrates, and has the fastest growing sales of any insecticide worldwide. Since its initial registration in France (1991), in January 1995, the Pesticide Management Regulatory Agency (PMRA) received applications requesting the registration of imidacloprid, and in April 1995, the PMRA granted temporary registration under section 17 of the Pest Control Products Regulations of Admire 240 F for the control of Colorado potato beetles in potatoes in Eastern Canada. In April 1999, imidacloprid was approved for use in potatoes across Canada and as a broad spectrum pesticide, it is presently registered in 100 countries for use on over 65 crops.

The high molecular mobility of Admire in the xylem of treated plants is due to its high water solubility (510 mg/L) ([redacted] *et al*. 1998; [redacted] et al., 1991). The molecular ability of imidacloprid makes it an ideal candidate for the use on potatoes and numerous other crops (apples, lettuce, tomatoes, mustard, canola, cucumber, corn, etc.). Due to its long term action, this chloronicotinoid is highly effective and has been used extensively as an in-furrow treatment for Colorado potato beetle. In potato fields the recommended in-furrow rate of application is 850 ml to 1.3 L / ha. Due to its residual activity, imidacloprid has become the most popular control agent for Colorado potato beetle.

Despite worldwide recognition, the use of Admire® has been in question following reports by French bee keepers of "disoriented" honey bees that had been foraging in imidacloprid (Gaucho®) treated sunflower fields. The bee keepers in France also reported that the honey bees had high rates of mortality, and low honey production due to a decrease in colony strength. In Canada, the PMRA's initial review of imidacloprid concluded that although pollinators (honey bees) could be at risk due to its high toxicity, the risk could be mitigated by a label statement contraindicating application of the product to blooming crops when bees are visiting the treatment area. Since that time, the question of whether systemic residues of imidacloprid may occur in nectar and pollen of flowering crops at concentrations harmful to honey bees has been the focus of an extensive research program. A study conducted by [redacted] (2000) examined the effects of imidacloprid (Gaucho®) seed treated sunflowers on honey bees and found no evidence to support the claims made by French beekeepers. In an investigation on the foraging behavior and orientation ability of honey bees by [redacted] changes in behavior were found for imidacloprid concentrations of 20 ppb (parts per billion) to 100 ppb, although no effect was observed at 10 ppb. Although the effects on the behavior of bees were observed to start at imidacloprid concentrations of 20 ppb, no damage to the test populations was observed for the range of concentrations tested up to 100 ppb.

With the release of the information from France, some bee keepers in Prince Edward Island and New Brunswick, complained of similar problems following placement of colonies near clover fields that had been previously treated with ADMIRE®, and requested a moratorium on the use of Admire® on Prince Edward Island. With this concern expressed, it was important to determine whether imidacloprid residue levels following use in potato fields was negatively affecting honey bee health on Prince Edward Island.

The objectives of this study were to determine if residue levels (ppb) of imidacloprid applied in-furrow,plus two metabolites, (hydroxy-imidacloprid and olefin-imidacloprid), were present one and two years following application of Admire in:

1) soil, clover leaves, and clover flowers, and wild flowers
2) pollen, and nectar collected from honey bees foraging in previously treated clover fields
3) uncapped honey collected from the hives placed in previously treated clover fields

The following report is a review of the protocol and results of the project.

MATERIALS AND METHODS

STUDY SITES

The collections were conducted at eighteen sites between Charlottetown and Summerside on Prince Edward Island (Figure 1), and at five sites between Woodstock and Florenceville (Wicklow), New Brunswick (Figure 2, Table 2, Appendix D)).

Figure 1. Eighteen Prince Edward Island Field Locations and treatment types.

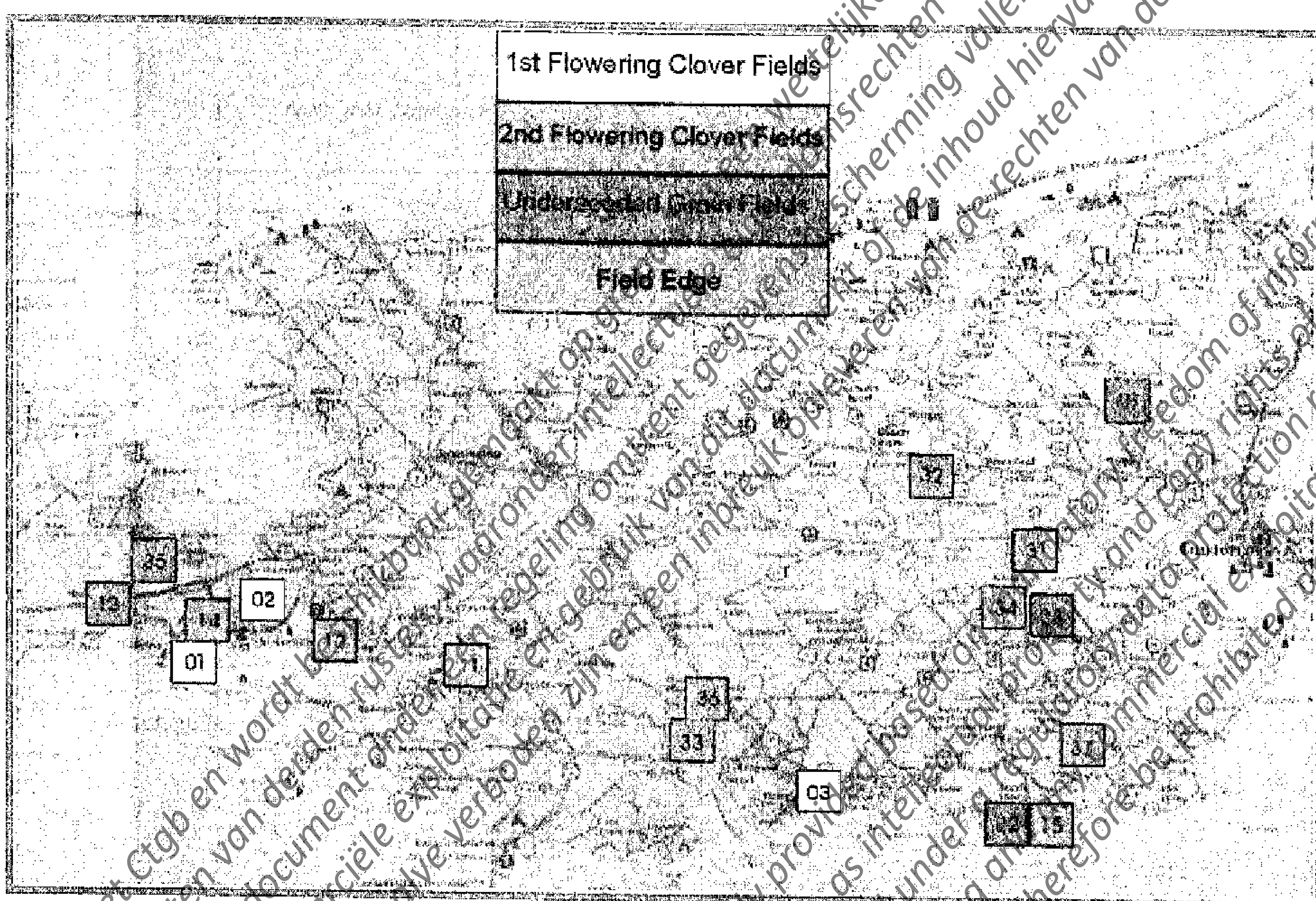
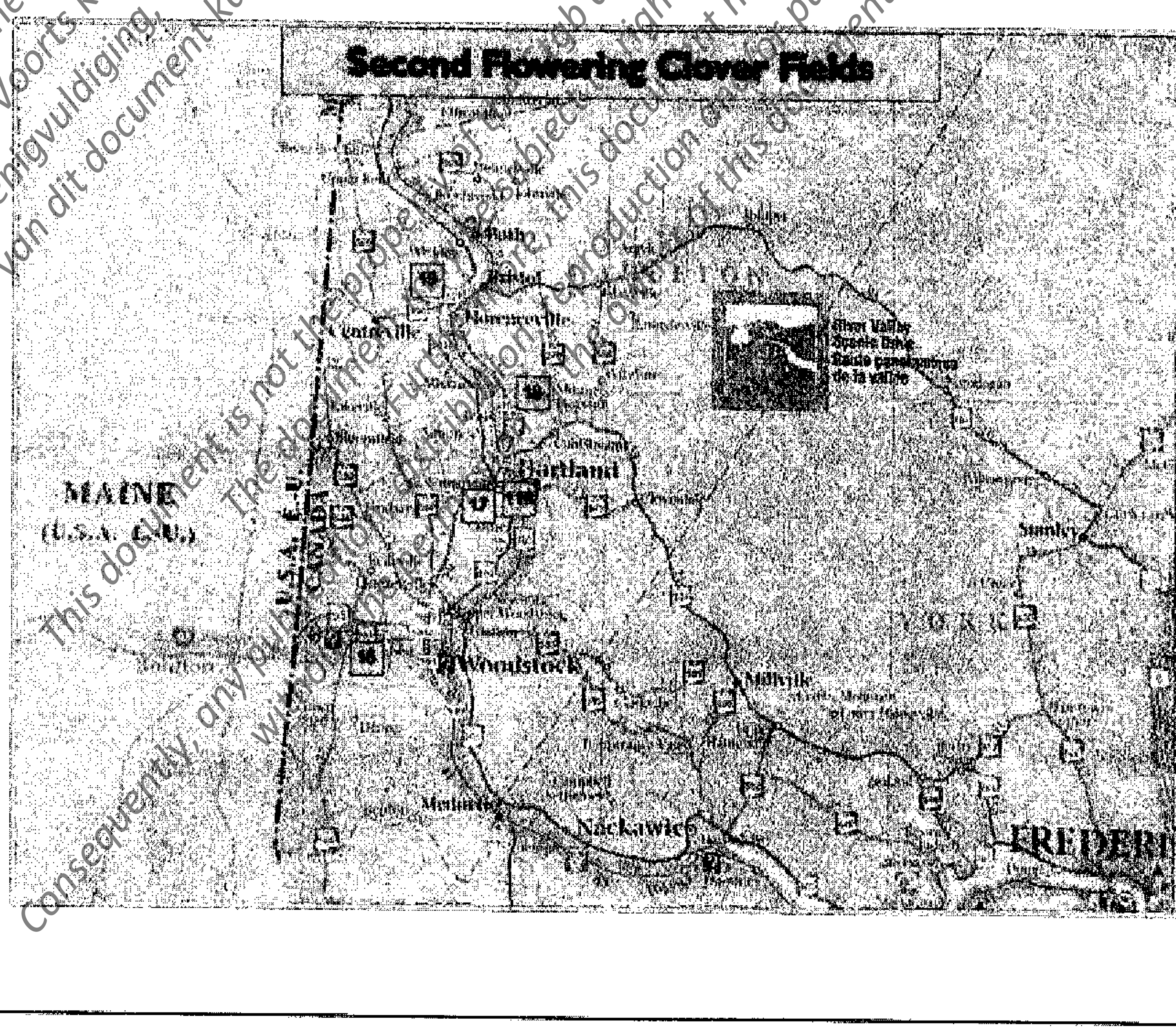


Figure 2. Five New Brunswick Field Locations and treatment type.



Three classifications of fields were used in this study (Table 1): 1) Potato fields (Year 1), 2) Underseeded grain fields (Year 2 field), 3) First and Second flowering clover fields (Year 3 field. Runoff areas of some year 1 and year 2 fields were subcategories for soil and wildflower sampling.

Table 1. Field Year was based upon when field had been treated with Admire.

Year Designation	Admire Application	Crop Planted
Year 1 field	Admire applied in Spring 2001	Potato Field
Year 2 field	Admire applied in Spring 2000	Grain Field
Year 3 field	Admire applied in Spring 1999	Clover Field

The fields used in this study had been planted in potatoes and treated with an in-furrow application of Admire (Bayer Corporation, active ingredient – imidacloprid) at the rate of 850 ml per hectare at the time of planting, except for the following fields: 1) fields 15 and 37 (Control fields, no treatment), 2) field 03 (½ the field treated at the rate of 850 ml/hectare, and ½ the field treated at the rate of 1300 ml/hectare), 3) field 110 (foliar application of imidacloprid). Underseeded grain fields were planted in either oats or barley and underseeded with a mixture of red clover (*Trifolium pretense*), alsike clover (*Trifolium hybridum*), and timothy. First flowering clover fields (first cut hay), and second flowering clover fields (second cut hay) contained a mixture of red clover (*Trifolium pretense*), alsike clover (*Trifolium hybridum*), and timothy.

Table 2. Overview of Field Design and Samples Collected at each Field in Prince Edward Island and New Brunswick

First Flowering Clover Fields Prince Edward Island		
Year 3 Field – Admire applied in Spring 1999		
Field 01	Field 02	Field 03
Floral Soil	Floral Soil	Floral Soil

Second Flowering Clover Fields Prince Edward Island				
Year 3 Field – Admire applied in Spring 1999				Control No Treatment
Field 11	Field 12	Field 13	Field 14	Field 15
Floral Leaf Soil Honey (08/22) Honey (09/14 Nectar Pollen	Floral Leaf Soil Honey (08/22) Honey (09/14 Nectar Pollen	Floral Leaf Soil Honey (08/22) Honey (09/14 Nectar Pollen	Floral Leaf Soil Honey (08/22) Honey (09/14 Nectar Pollen	Floral Leaf Soil Honey (08/22) Honey (09/14 Nectar Pollen

Underseeded Grain Fields Prince Edward Island		
Year 2 Field – Admire applied in Spring 2000		
Field 21	Field 22	Field 23
Leaf Soil	Leaf Soil	Leaf Soil

Runoff Fields – Soil &Floral Collections Prince Edward Island						
Year 1 (Potato Field)			Year 2 (Underseeded Grain Field)			Control (Grain)
Field 31	Field 32	Field 33	Field 34	Field 35	Field 36	Field 37
Goldenrod Fireweed Asters Soil	Goldenrod Soil	Goldenrod Soil	Goldenrod Asters Soil	Goldenrod Soil	Goldenrod Soil	Goldenrod Fireweed Asters Soil

Second Flowering Clover Fields New Brunswick				
Year 3 Field – Admire applied in Spring 1999				Foliar Application
Field 16	Field 17	Field 18	Field 19	Field 110
Floral Leaf	Floral Leaf	Floral Leaf	Floral Leaf	Floral Leaf

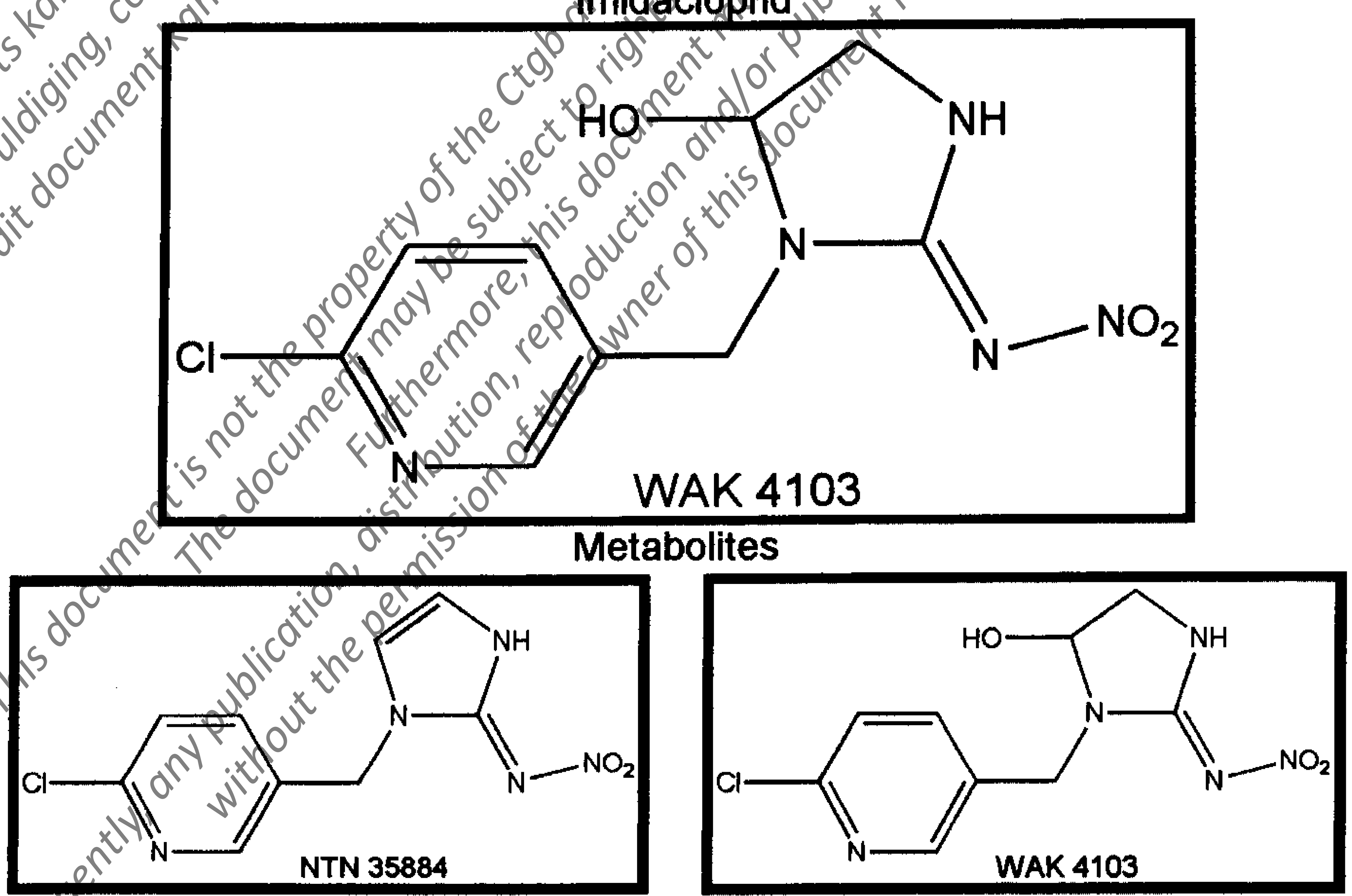
RESIDUE ANALYSIS

Residue analysis was performed by Enviro-Test Laboratories, Edmonton, Alberta. Data was generated in compliance with PMRA DiR 98-01 which outlines the requirements of OECD GPL principals and in compliance with Good Laboratory Practices according to EPA-FIFRA section 4- CFR part 160 (Oct 16, 1989) (Appendix A). The quality assurance unit of Enviro-Test Laboratories inspected and/or audited the analytical phase of the study and the report, and reported its findings to the Study Director, and to ETL Management.

The objectives of this part of the study were: 1) To determine LOD/LOQ (Limit of Detection/Limit of Quantification) and validate the modified analytical methods: Method No. 00554, Method No. 00537, Method 00537/E001 and Method 106428 (Soil Method dated Aug. 24/94), 2) To analyze soil, pollen, nectar, honey, and plant samples for imidacloprid, NTN 35884 (Olefin metabolite), and WAK 4103 (Hydroxy metabolite) (Figure 3). The methods used for reference was Bayer method no. 00537 (report no. MR-551/98), method no 00537/E001 (report no. MR-568/99), and no 00554 (report no. MR-812/98).

Samples that had been stored in a freezer at -20 ± 5°C at the University of Prince Edward Island, were shipped to Enviro-Test Laboratories in coolers containing dry ice. Samples were received in good condition and were immediately stored in a freezer at -25 ± 5°C. The nectar, honey, pollen and most of the flower samples were received processed and did not require further processing. Flower and leaf samples were prepared in a food processor in the presence of dry ice. Soil samples were sieved into a homogenous mixture. The % moisture was determined for all soil samples. The samples were then analyzed by High Performance Liquid Chromatography-Electrospray Ionization Mass Spectrometry (HPLC-MS/MS). Quantification was accomplished by using weighted (1/x) linear regression from an eight to nine point calibration curve.

Figure 3. Imidacloprid, Olefin and Hyroxy metabolites



The method of validation for pollen, nectar, and honey consisted of 6 spiked samples: 1 control, 3 at LOQ (Level of Quantification), and 2 at 5 times LOQ. For Soil, leaf, and flowers, the method of validation consisted of 8 spiked samples: 1 control, 5 at LOQ, and 2 at 5 times LOQ. The average verification recoveries (Table 2) and in-phase recoveries (Table 3)were good for all analytes.

Table 2. Average Verification Recoveries

Sample	Average Verification Recoveries and SD		
	Imidacloprid	Hydroxy Metabolite	Olefin Metabolite
Runoff Soil	99±7.9%	Not tested	Not tested
Field Soil	90±22%	Not tested	Not tested
Clover flowers	96±9.8%	92±10%	96±15%
Clover leaves	110±6.6%	105±14%	92±7.3%
Goldenrod flowers	112±3.6%	112±4.3%	107±10%
Pollen	104±16%	96±18%	89±15%
Honey	101±9.3%	85±19%	91±22%

Table 3. Average In-phase Recoveries

Sample	Average In-Phase Recoveries		
	Imidacloprid	Hydroxy Metabolite	Olefin Metabolite
Runoff Soil	98%	Not tested	Not tested
Field Soil	91%	Not tested	Not tested
Clover flowers	87%	85%	90%
Clover leaves	88%	90%	79%
Goldenrod flowers	106%	96%	105%
Pollen	96%	94%	74%
Honey	101±9.3%	85±19%	91±22%

HONEY BEES

Source

Honey bee colonies were placed on site to supply the foraging bees from which the pollen and nectar were collected. The hives and colonies of honey bees were supplied by the Prince Edward Island division of [redacted]. They also supplied additional supers when needed for colony management. The bees were New Zealand stock imported in the spring of 2001 and the equipment was previously used (imported from western Canada). Eight hives of honey bees, on two pallets of four, were moved to the edge of each of 4 second bloom fields and 1 second bloom control field on July 18 (n=40 hives). The hives were positioned in such a way as to maximize foraging activity on the study fields.

Management & Assessments

On July 25-27, all colonies at each site were equalized (i.e. "adjusted for strength – similar quantities of food stores (pollen and nectar), brood in all stages of development and adults covering at least 10 frames"), qualitatively assessed for general colony health, and managed for swarm prevention. Colonies were again assessed on September 14-15. Apistan strips and sticky boards were installed at the time of assessment to survey for varroa mite. The strips and boards were removed and inspected for varroa on July 17. Jasper Wyman & Son removed the colonies from the study fields on September 18.

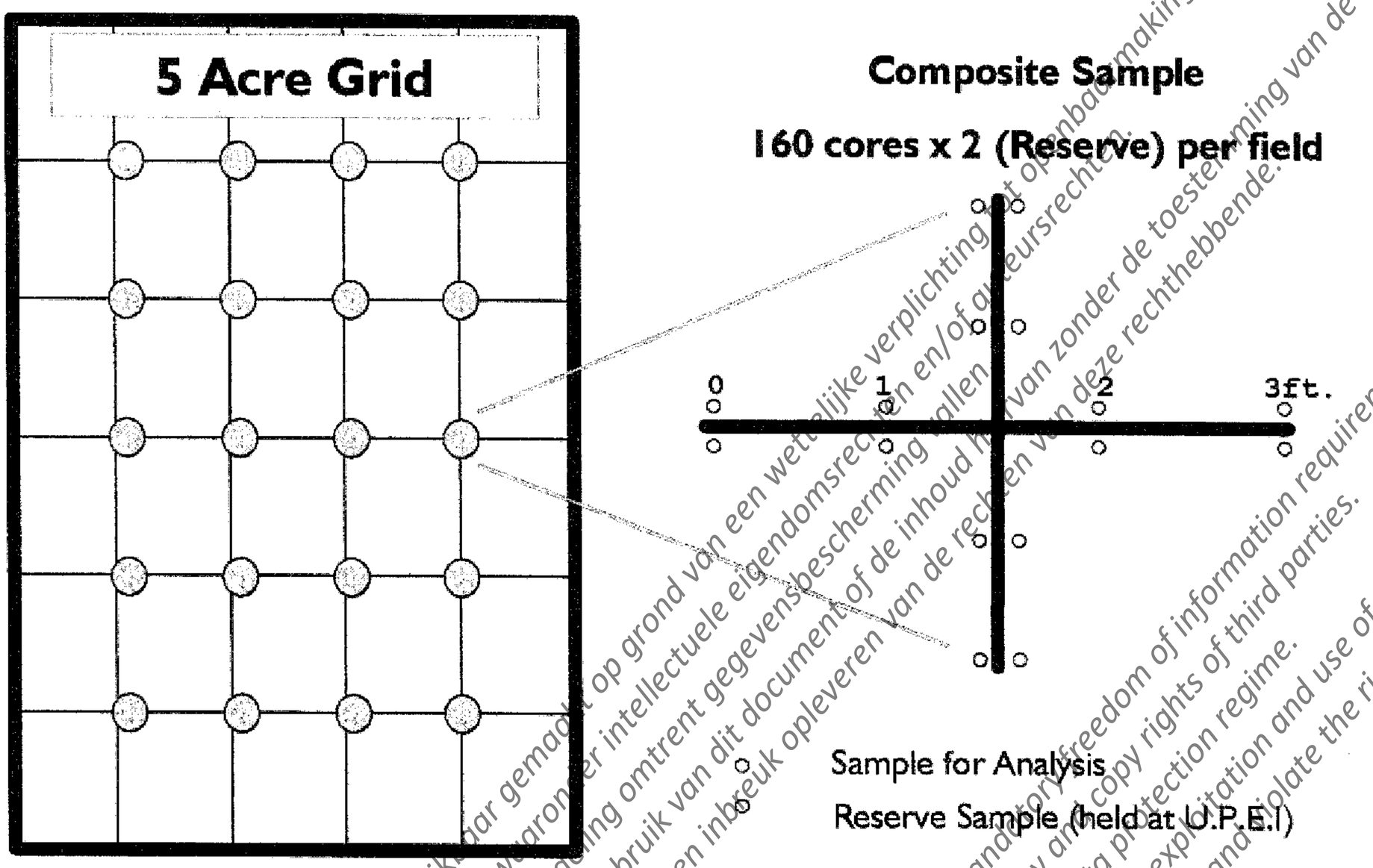
COLLECTION METHODS

Soil

Field Plots

A composite sample of one hundred sixty soil cores (18cm x 13mm diameter) per field were collected from eleven fields. A five acre plot was measured and staked out on each field, and divided into twenty collection points. Eight soil cores at one foot intervals were collected at each point (Figure 4) to ensure that a treated furrow would be sampled.

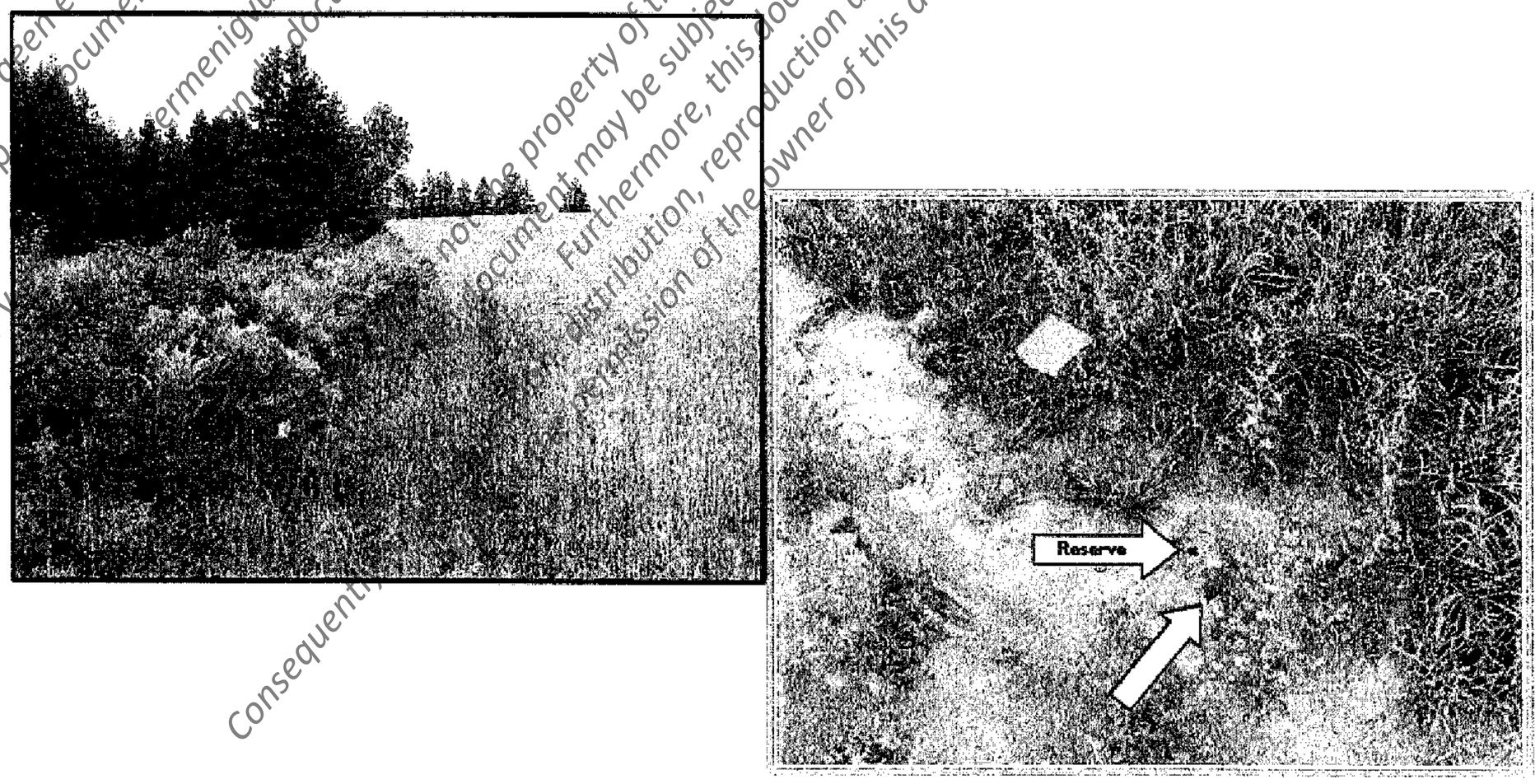
Figure 4. Field Soil Core Collections



Field edge runoff areas

A composite sample of twenty soil cores (18cm x 13mm diameter) per field were collected from seven runoff fields. At runoff locations where wildflowers were present, the field edge bottom containing wildflowers was divided into 20 sampling locations. At each location one soil core was collected near the base of the wildflower plants (Figure 5).

Figure 5. Field edge runoff soil core collections



Plants

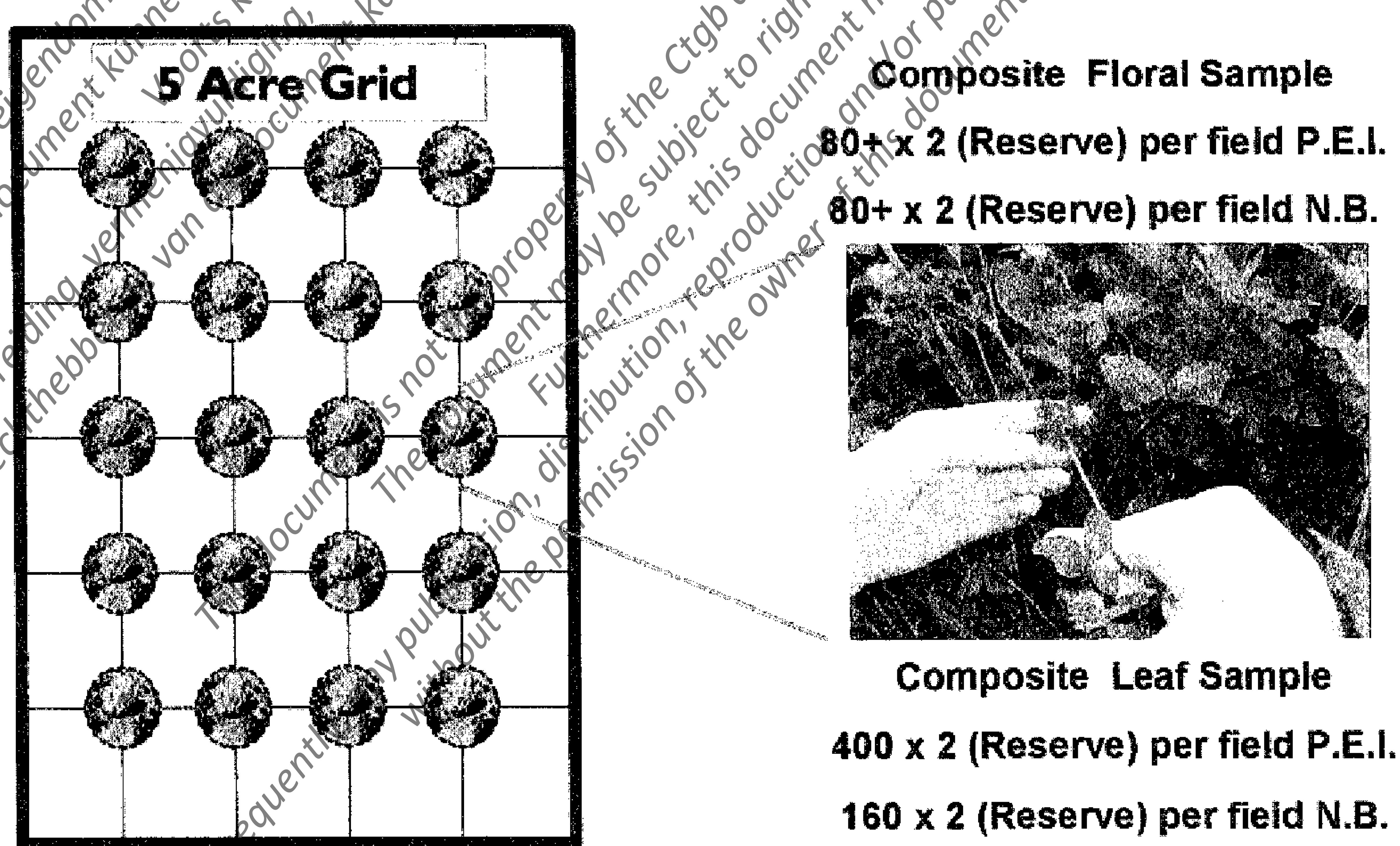
Clover Flowers

A composite sample of 80+ clover flowers per field were collected from eight fields in Prince Edward Island (Figure 6). In New Brunswick, five acre plots were measured and staked out on each field, and divided into twenty collection points, similar to those on Prince Edward Island. The same five acre plot , and twenty collection points used to collect soil in Prince Edward Island were used to collect the clover flowers. In both locations a minimum of four flowers (freshly opened inflorescences) were collected per field point. Depending upon the type of plant present, for every one red clover flower collected, two white clover flowers were collected as the white clover flowers (inflorescences)were much smaller. In both locations, the flowers were removed from the plant and immediately stored in a bag, and placed in a cooler containing dry ice.

Clover Leaves

A composite sample of four hundred clover leaves per field were collected from eight fields in Prince Edward Island (Figure 6). In New Brunswick, a composite sample of one hundred and sixty clover leaves per field were collected from five fields. The same five acre plot , and twenty collection points used to collect soil in Prince Edward Island were used to collect the leaves. In New Brunswick, five acre plots were measured and staked out on each field, and divided into twenty collection points, similar to those on Prince Edward Island. In Prince Edward Island, 20 leaves were collected per point, and in New Brunswick, 8 leaves were collected per point. In both locations, the upper-most fully expanded leaf was removed from the plant, and immediately stored in a bag and placed in a cooler containing dry ice.

Figure 6. Field Soil Core Collections



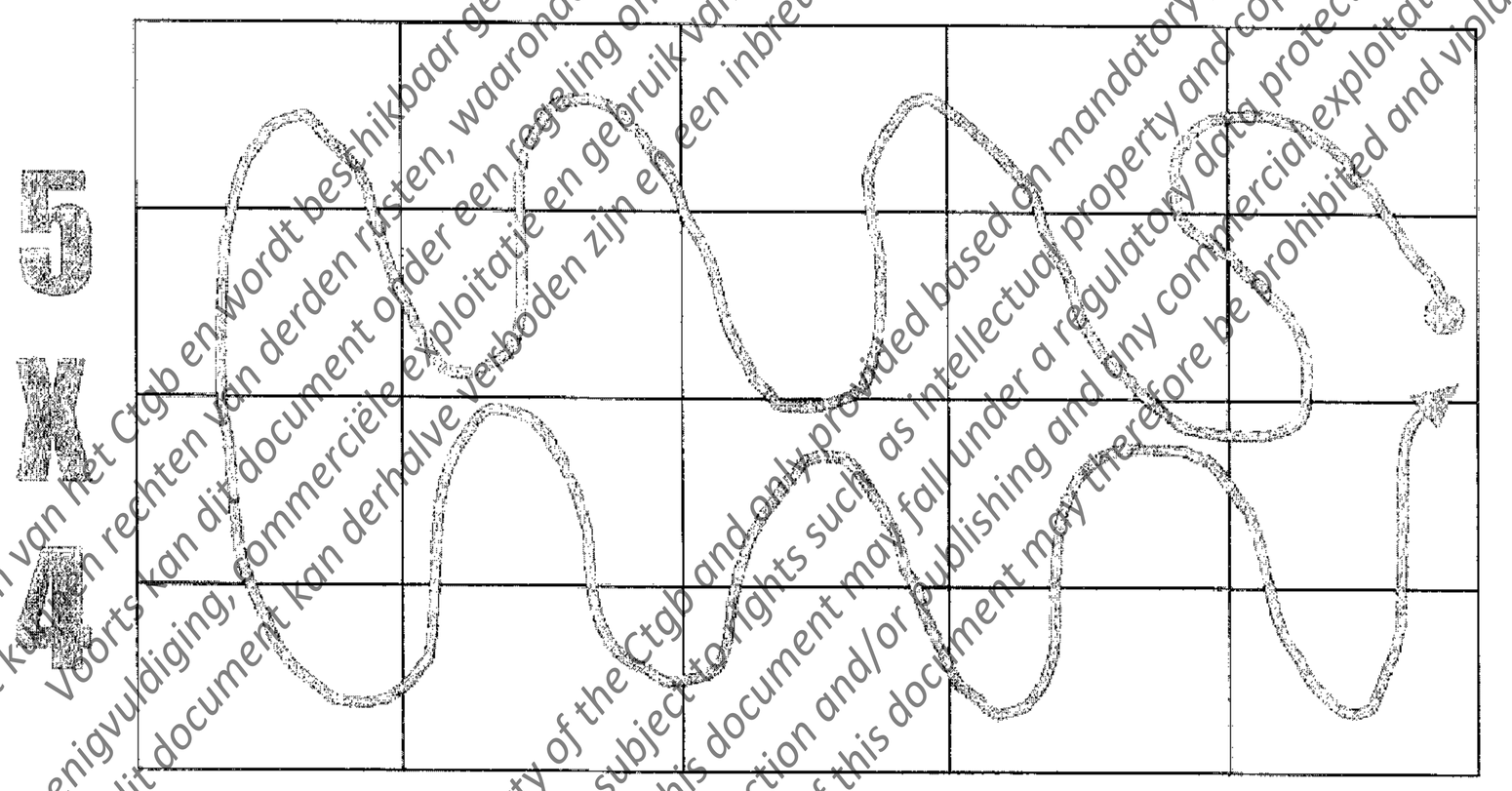
Wildflowers

A composite sample of forty grams per species of Goldenrod (*Solidago canadensis*) inflorescences, Fireweed (*Epilobium angustifolium*) flowers, and Aster (*Aster novi-belgii*) flowers were collected if present from seven runoff fields. The twenty collection points used to collect runoff soil was used to collect the wildflowers (Figure 5). Once the flowers were removed from the plant they were immediately stored in a bag, and placed in a cooler containing dry ice.

Honey Bees

Using a portable bug vac and a serpentine collecting pattern (Figure 7), pollen and nectar collecting honey bees were collected from each second bloom treated, and second bloom control, fields during the period late July to early September. The collected bees were immediately placed on dry ice and at the end of each day transported to the lab at UPEI for long-term storage in a freezer at –20°C ± 5°C.

Figure 7. Sampling method for honey bee collection



NECTAR & POLLEN EXTRACTION

In October, the collected honey bees were transported to an AAFC lab in Kentville, NS on dry ice and then stored in an upright freezer. At the Kentville lab, [redacted] with the assistance of three students, sorted the bees into pollen and nectar carrying bees in a coldroom, and then extracted the pollen and nectar loads in the lab, under a microscope at room temperature. After extraction, samples were refrozen and on October 30 were transported back to UPEI on dry ice. On October 31, the samples were shipped to EnviroTest for analysis. The protocol followed for the extraction process is outlined below.

General

1. Work on only one sample from one site on one date at a time.
2. In the cold room (5° C), work as quickly as possible to sort the sample into bees with pollen loads, nectar loads, and those with neither. The cold room helps minimize condensation on the bees.
3. Process bees promptly. DO NOT leave them out while doing some other task.
4. DO NOT leave samples unattended for any length of time.
5. Return portions of large samples to the freezer until such time you will need them.
6. Maintain strict sanitation by working in clean workspaces. DO NOT contaminate samples.
7. Clean instruments before working on samples and between samples. Do this by cleansing in 70+% EtOH and them rinsing with distilled water.
8. Wear new vinyl or latex gloves at all times while working with the bees. Change gloves before handling bees from a new sample site.
9. Bumble bees can be used for pollen loads if available.

Nectar

1. Sort out the nectar bees in the cold room.
2. Place an even single layer of bees in each sterile petrie dish.
3. Work on groups of 50 or fewer bees at one time. Keep the remaining bees in the sample frozen.
4. Process at least 100 bees for the main sample, and another 100+ bees for the reserve sample.
5. Do the nectar removal by inserting a 12.7 mm ultra-fine hypodermic needle, attached to a 0.3 cc syringe, into the honey stomach of the subject honey bee. Draw off the nectar and then deposit it in a sterile vial.
6. Label the vial by writing in permanent ink on the supplied label and affix to the outside of the vial.
7. Weigh each sample to ensure that the minimum sample weigh was achieved. Minimum required nectar sample weight is 1 g. Target for sample is 2-4 g.
8. Place the vial in a ziploc plastic bag and freeze immediately.
9. NOTE: Maximum nectar loads, for estimation purposes, are about 1/2 the weight of a bee (90/2 = 45 mg).

Pollen

1. Sort out the pollen carrying bees in the cold room.
2. Place an even single layer of bees in each sterile petrie dish.
3. Cover with paper towel , put petrie dish lid on, and then secure with elastic band.
4. Place petrie dishes with bees in drying oven at 75° F for 45 minutes.
5. Return nonpollen carrying bees to the freezer promptly after sorting.
6. Process at least 200 bees for the main sample, and another 200+ bees for the reserve sample.
7. Do the pollen removal in a sterile petrie dish using sterile forceps and probes.
8. Place all of the removed pollen loads from one sample site into a sterile vial.

9. Label the vial by writing in permanent ink on the supplied label and affix to the outside of the vial.
10. Weigh each sample to ensure that the minimum sample weight is achieved. Minimum required pollen sample weight is 2 g. Target for sample is 4-5 g.
11. Place the vial in a ziploc plastic bag and freeze immediately.
12. NOTE: Maximum pollen loads, for estimation purposes, are about 1/6 the weight of a bee (90/6 = 15 mg).

Uncapped Honey Collection

Unripe honey was collected from the hives on August 22 and September 14 (Figure 8). The equivalent of 1-2 frames of uncapped honey were collected, either on drawn comb, or comb freshly drawn in an empty frame space. The uncapped honey was extracted by cutting the comb into chunks and placing them into a strainer over a plastic bowl. After crushing the comb, the honey was allowed to drip through the strainer for several hours. A 45 g sample of honey was decanted into a sterile sample vial, labeled, and frozen. A duplicate sample was also be collected. All samples were shipped on dry ice to Enviro Test on October 30.

Figure 8. Honey Collection

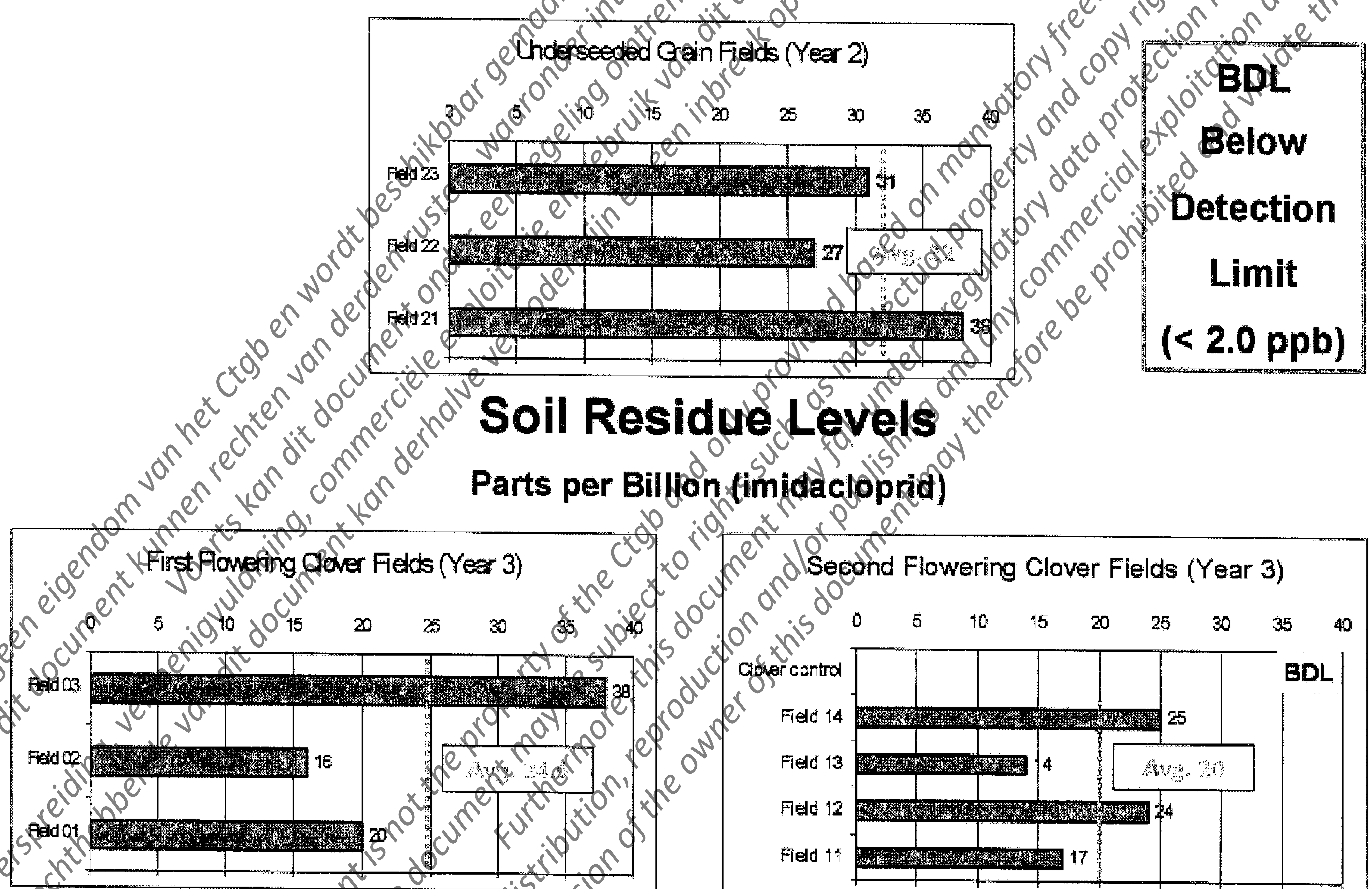
RESULTS (Appendix B)

Soil

Field Plots

Residue levels of imidacloprid in Prince Edward Island field soil samples ranged from <2.0 ppb (parts per billion) to 38 ppb. Levels of hydroxy and olefin metabolites were not tested. Samples from underseeded grain fields (Year 2 fields) ranged from 27 ppb to 38 ppb (average = 32 ppb). Samples from first flowering clover fields (Year 3 fields) ranged from 16 ppb to 38 ppb (average = 24.6 ppb). Samples from second flowering clover fields ranged from 14 ppb to 25 ppb (average = 20 ppb). The control field had no quantifiable residues of imidacloprid (< 2.0 ppb (below detection limit - LOQ) (Figure 9). Soil samples were also sent to the P.E.I. Department of Agriculture and Forestry for soil analysis (Appendix C).

Figure 9. Field soil residue results for imidacloprid

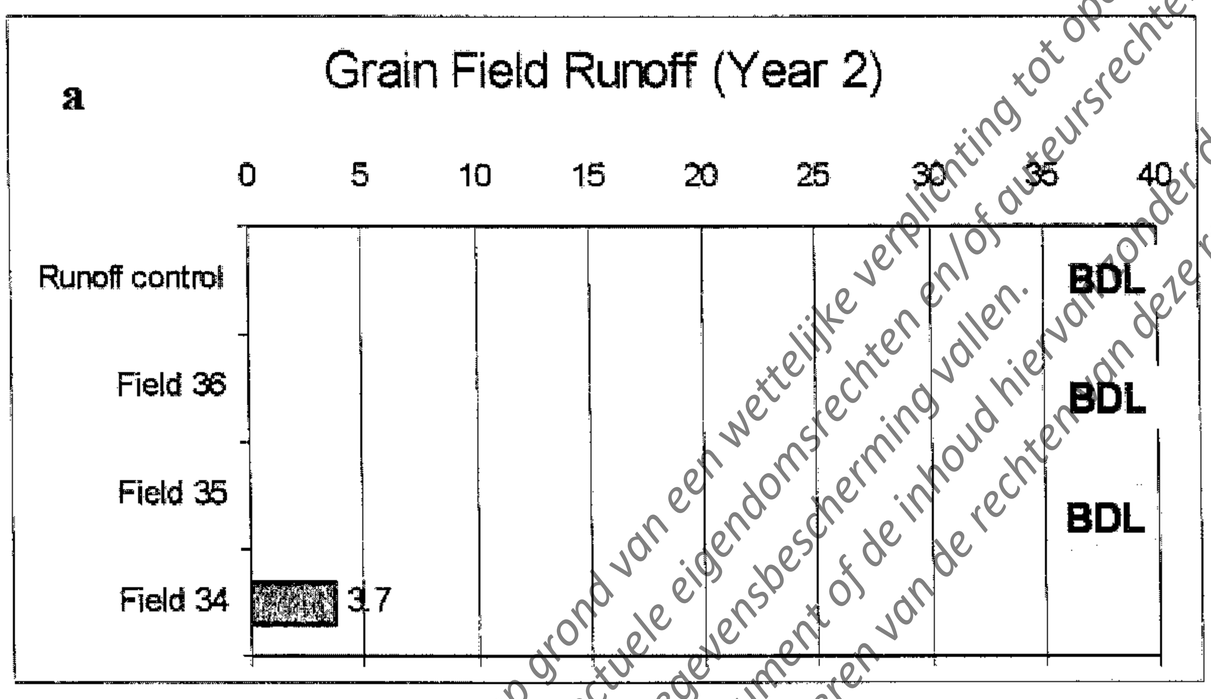


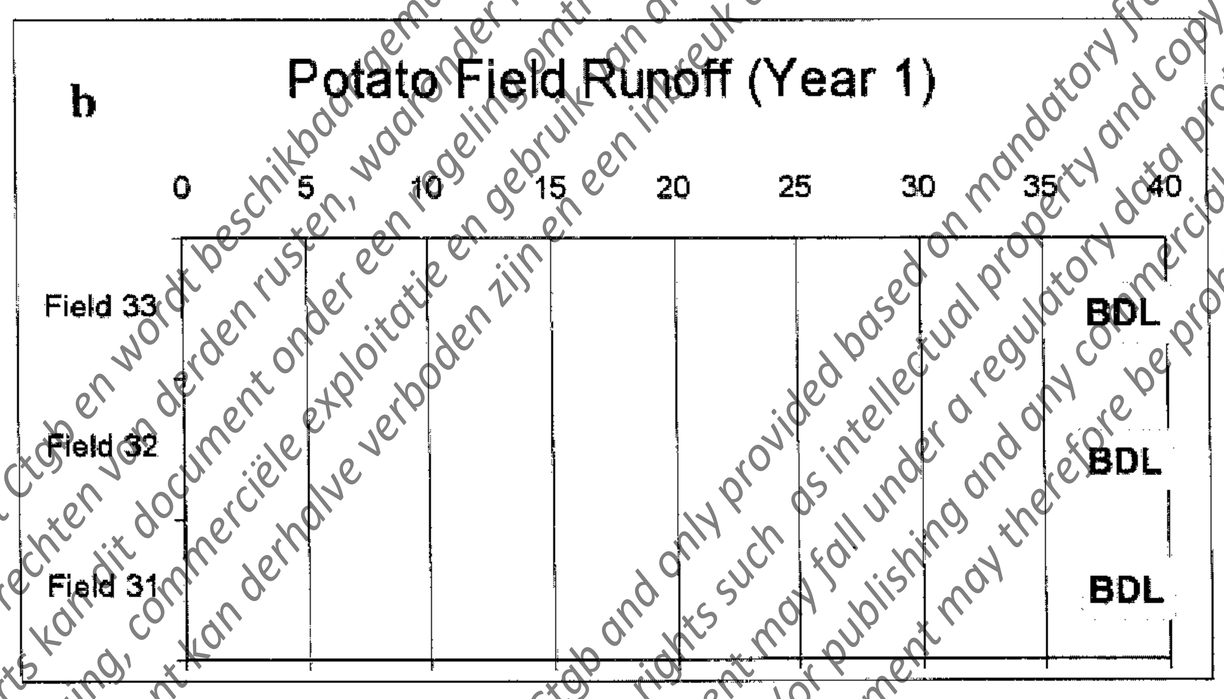
Field edge runoff areas

Residue levels of imidacloprid in Prince Edward Island field runoff soil samples was detected in only one sample (Figure 10a & b). Levels of hydroxy and olefin metabolites were not tested. Samples from potato field runoffs (Year 1 fields) were all below detection limit (< 2.0 LOQ). One sample from underseeded grain fields had a level of 3.7

ppb, all other samples were below detection limit (<2.0 ppb). The control field had no quantifiable residues of imidacloprid (< 2.0 ppb (below detection limit - LOQ).

Figure 10 a & b. Grain field runoff residue results for imidacloprid. BDL = below detectable limit of 2 ppb (synonymous with LOQ).



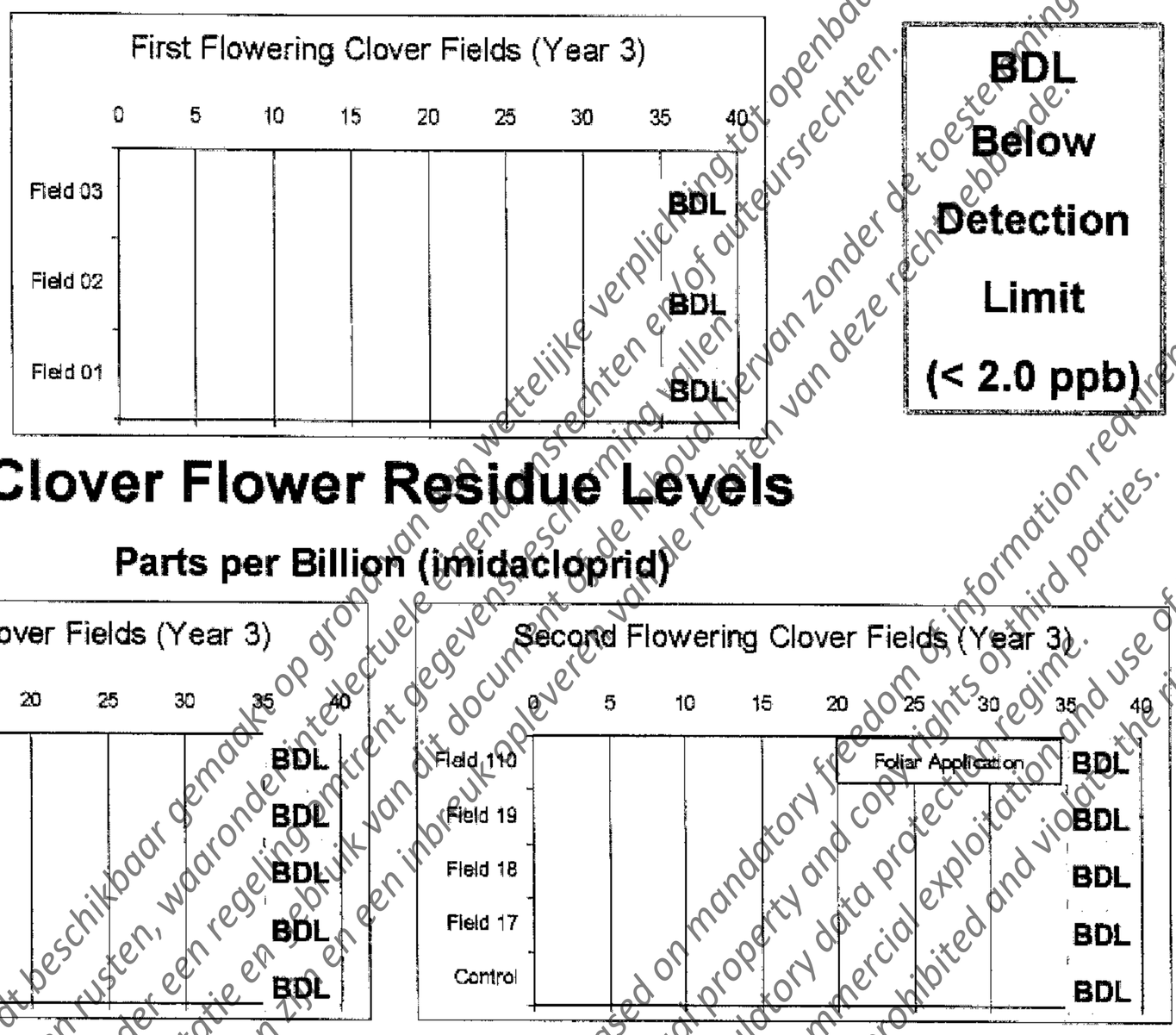


Plants

Clover Flowers

Residue levels of imidacloprid and the hydroxyl and olefin metabolites in Prince Edward Island and New Brunswick clover flower samples from first and second flowering clover fields (year 3 fields) were all below level of detection (<2.0 ppb) (Figure 11). The control field had no quantifiable residues of imidacloprid or metabolites (< 2.0 ppb).

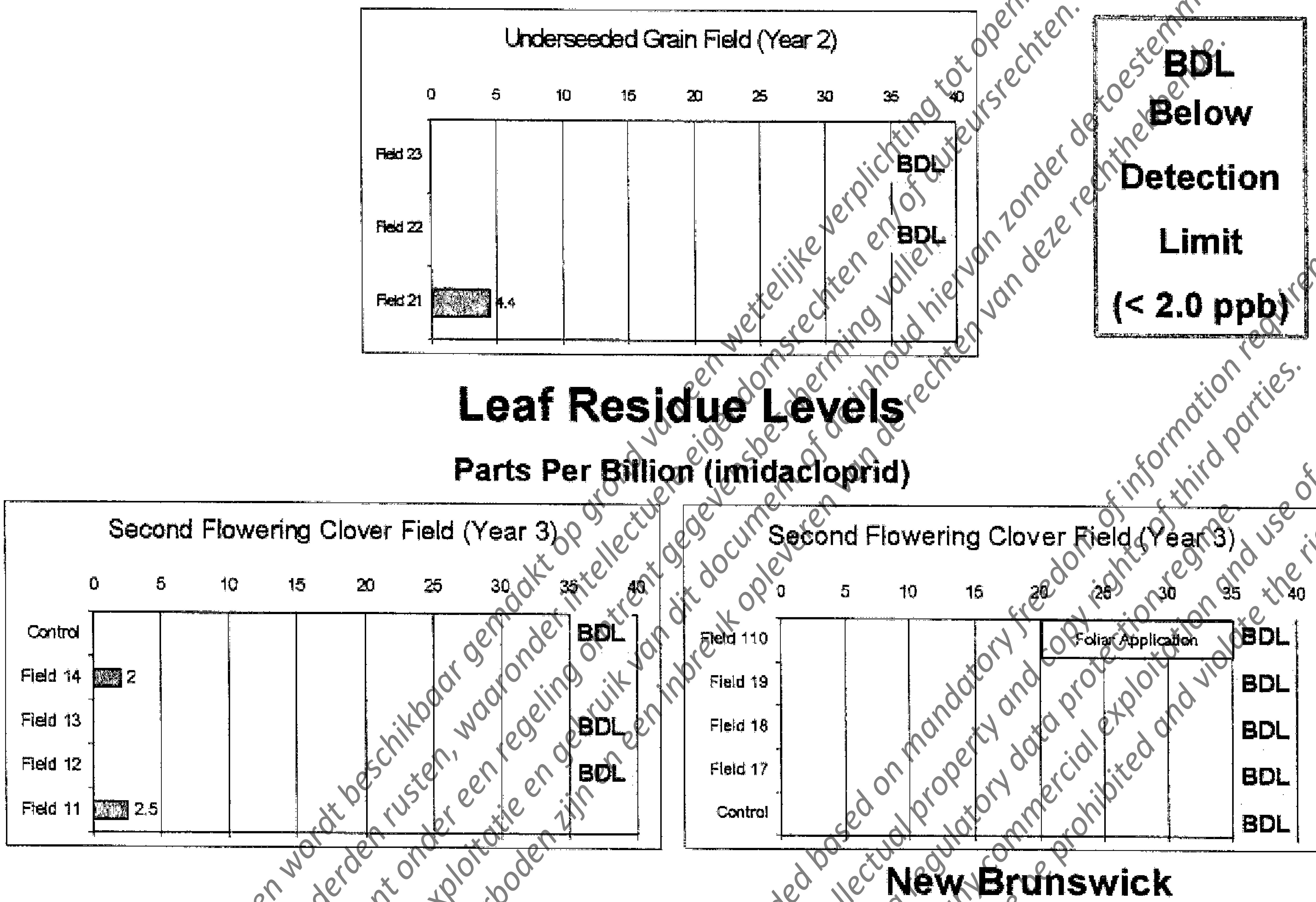
Figure 11. Clover flower residue results for imidacloprid



Clover Leaves

Residue levels of imidacloprid in Prince Edward Island and New Brunswick clover leaf samples ranged from <2.0 ppb (parts per billion) to 4.4 ppb (Figure 12). Levels of hydroxy and olefin metabolites were below detection limit (<2.0 ppb) for all fields tested. Samples from underseeded grain fields (Year 2 fields) ranged from <2.0 ppb to 4.4 ppb. Samples from second flowering clover fields (Year 3 fields) in Prince Edward Island ranged from <2.0 ppb to 2.5 ppb. Samples from second flowering clover fields in New Brunswick were all below detection limit (<2.0 ppb). The second flowering control field in Prince Edward Island had no quantifiable residues of imidacloprid or metabolites (< 2.0 ppb (below detection limit - LOQ).

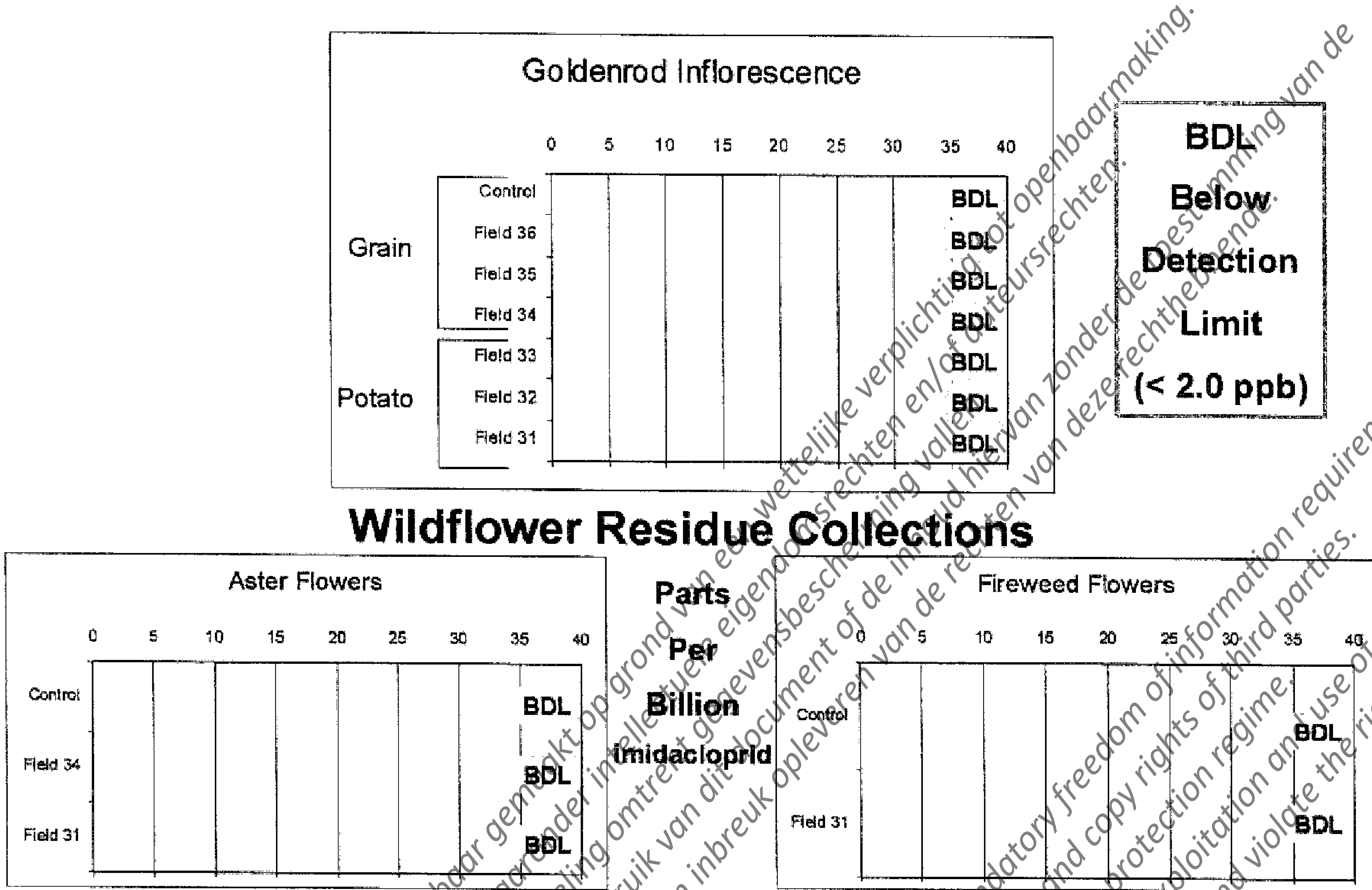
Figure 12. Clover leaf residue results for imidacloprid



Wildflowers

Residue levels of imidacloprid and the hydroxyl and olefin metabolites in Prince Edward Island wildflower samples (goldenrod, fireweed, and asters) from runoff areas of potato fields (year 1 fields) and underseeded grain fields (year 2 fields) were all below level of detection (<2.0 ppb) (Figure 13). The control field had no quantifiable residues of imidacloprid or metabolites (< 2.0 ppb).

Figure 13. Wildflower residue results for imidacloprid

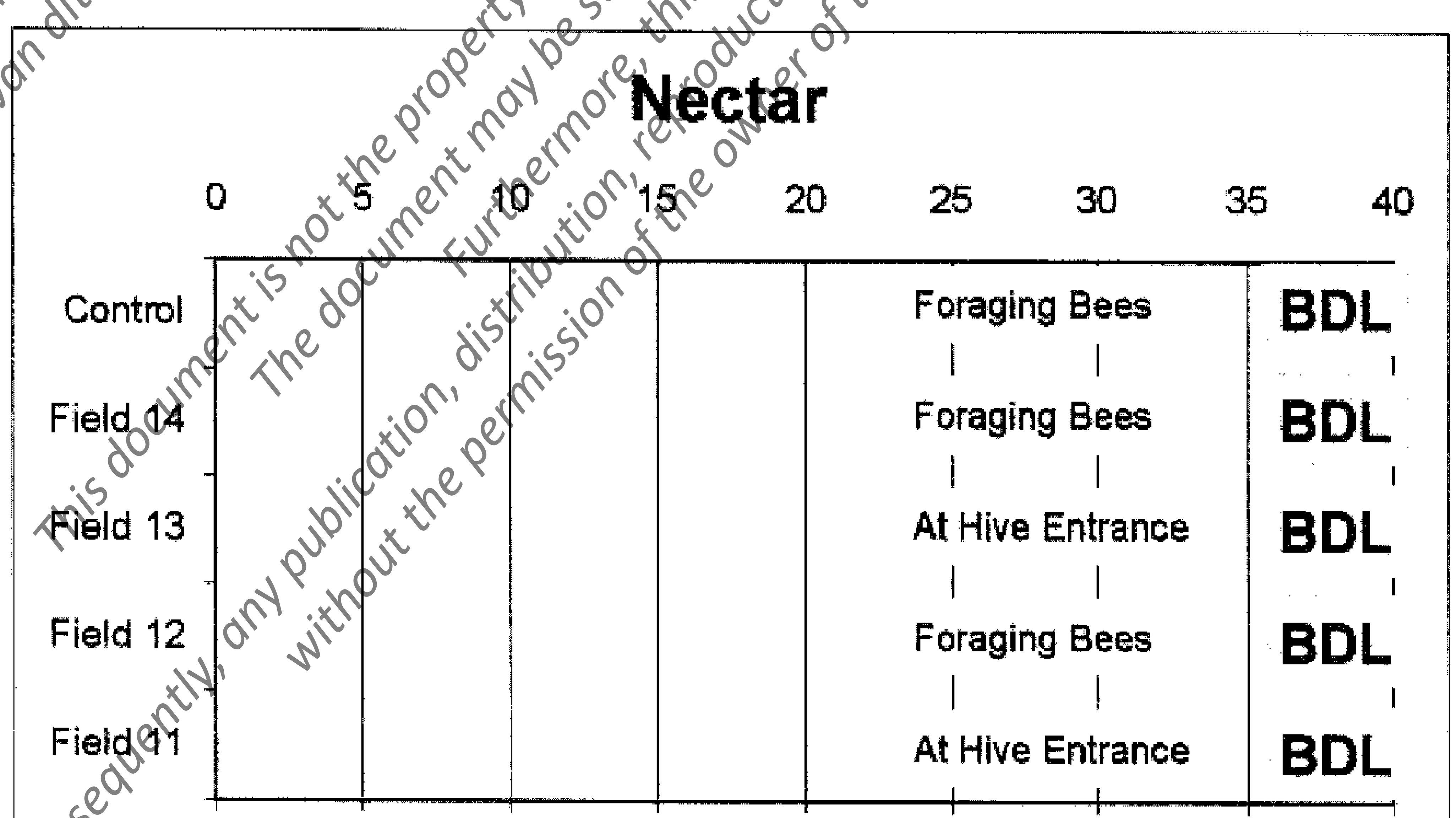


Honey Bees

Nectar

Residue levels of imidacloprid and the hydroxy and olefin metabolites in Prince Edward Island nectar samples collected from honey bees in second flowering clover fields (year 3 fields) were all below level of detection (<2.0 ppb) (Figure 14). The control field had no quantifiable residues of imidacloprid or metabolites(< 2.0 ppb).

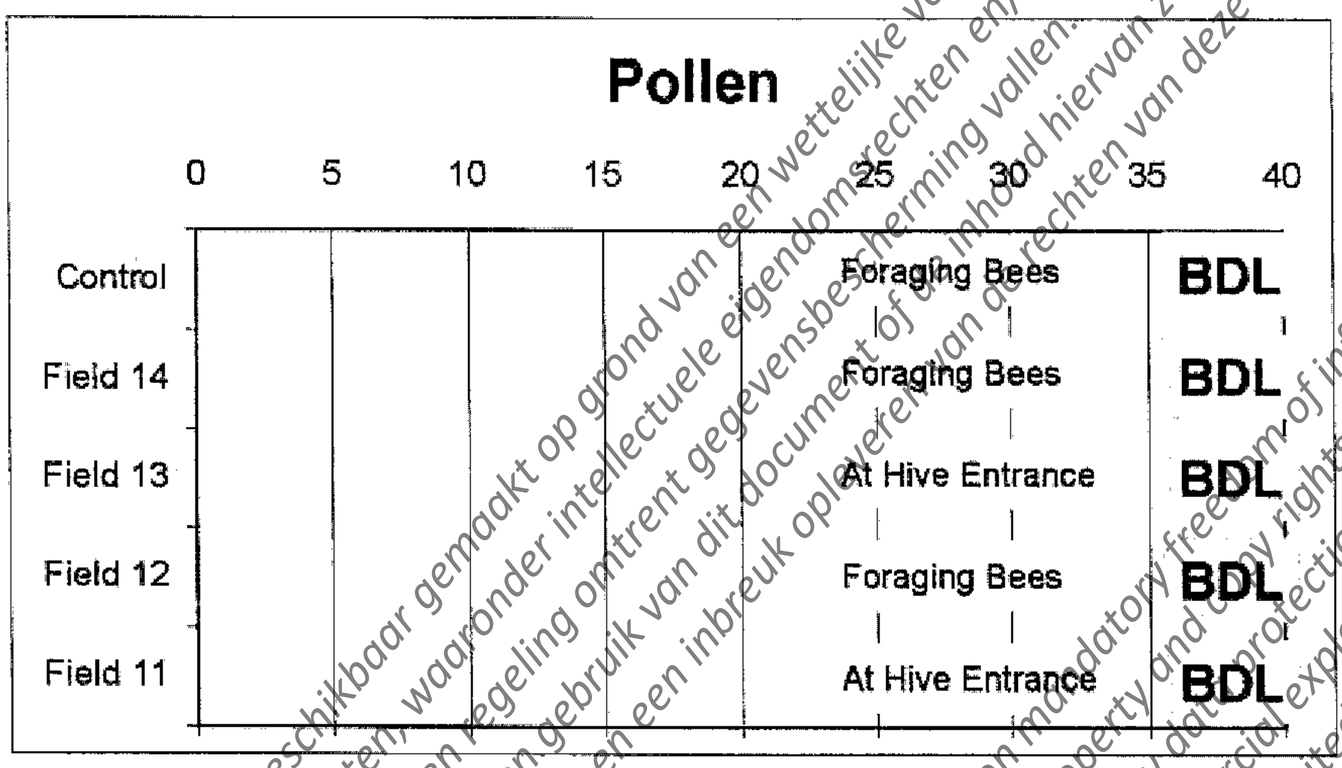
Figure 14. Nectar residue results for imidacloprid



Pollen

Residue levels of imidacloprid and the hydroxy and olefin metabolites in Prince Edward Island pollen samples collected from honey bees in second flowering clover fields (year 3 fields) were all below level of detection (<2.0 ppb) (Figure 15). The control field had no quantifiable residues of imidacloprid or metabolites(< 2.0 ppb).

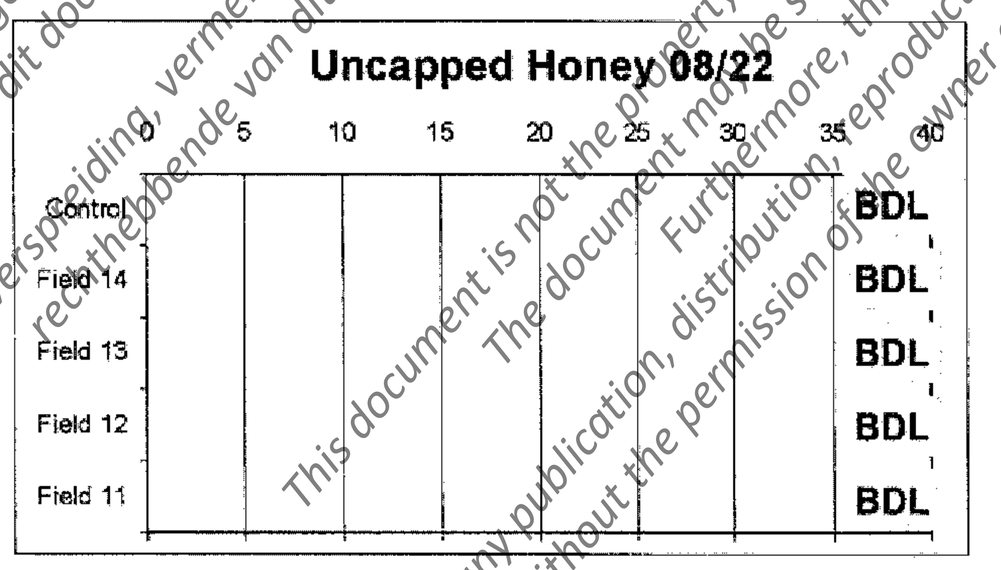
Figure 15. Nectar residue results for imidacloprid

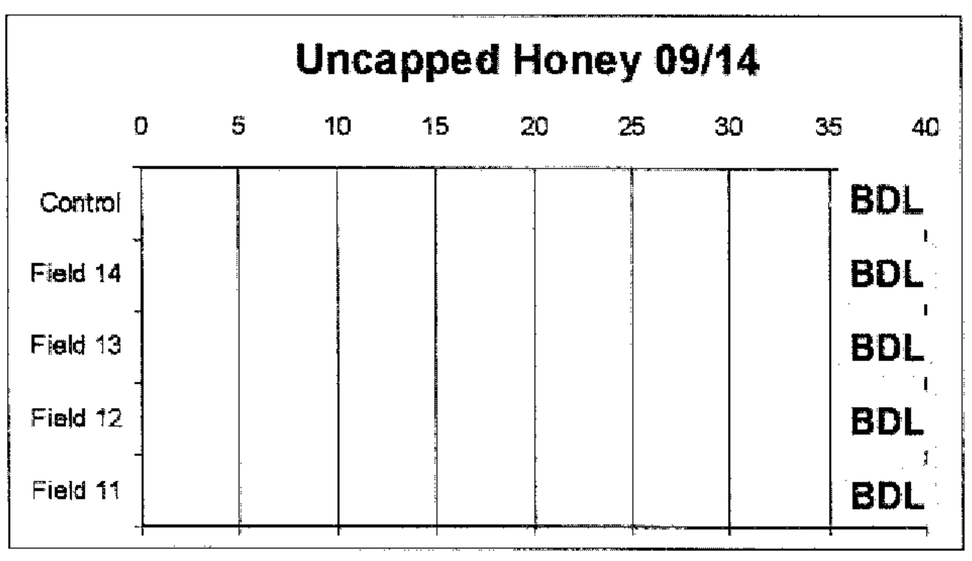


Uncapped Honey

Residue levels of imidacloprid and the hydroxy and olefin metabolites in Prince Edward Island uncapped honey samples collected from hives placed in second flowering clover fields (year 3 fields) were all below level of detection (<2.0 ppb) (Figure 16). The control field had no quantifiable residues of imidacloprid or metabolites(< 2.0 ppb).

Figure 16. Uncapped honey residue results for imidacloprid





Colony Assessments

At the first inspection (Table 4), colonies were found to be of normal strength and condition for spring packages that were used for lowbush blueberry pollination. There were sufficient bees, brood, pollen, and honey stores to warrant the addition of supers to provide space for colony expansion. The second inspection (Table 5) found some colonies and apiaries doing better than others. Except for apiary #15, all apiaries, on average, had minimal levels of disease, increased bee numbers, and good honey crops. Apiary #12 and #15 were positive for varroa. It is interesting to note that these same two apiaries also had noticeable smaller honey crops. Apiary #11 had consistently aggressive colonies and the aggressiveness seemed to be related to queen stock. Most colonies had New Zealand stock, however, where Australian queens were used, the bees were very aggressive.

Table 4. Average ratings and scores for colony strength and health. Assessments done July 26-27, 2001.

Field Number	11	12	13	14	15
n	8	8	8	8	8
# Supers Start[1]	2	2	2	2	2
Frames of Bees[2]	16.8	18.1	17.9	16.8	15.3
Brood (0-2)[3]	1.9	1.8	2.0	1.9	1.6
Honey (0-5)[4]	2.4	2.7	3.0	2.9	1.6
# Laying[5]	8	7	8	7	7
# EFB[6]	0	3	0	0	0
# AFB[7]	0	0	0	0	0
# SAC[8]	0	0	0	0	0
# CB[9]	3	4	6	0	5
# VM[10]	n/a	n/a	n/a	n/a	n/a
Aggressive[11]	Yes	No	No	No	No

Note: All colonies supered as needed

[1] # Supers Start = the number of supers and brood boxes that make up the hive at the start of each inspection.

[2] Frames of Bees = the estimated number of frame equivalents that are 100% covered with a single layer of bees.

[3] Brood = a qualitative assessment of the amount of brood present scored on a scale of 0-2; 0 = no brood, 1 = some brood, 2 = lots of brood.

[4] Honey = a qualitative assessment of the amount of honey (both capped and uncapped) present based on a scale of 0-5; 0 = none, 1 = very little, 2 = little, 3 = moderate, 4 = lots, 5 = full

[5] # Laying = number of colonies with laying queens

[6] # EFB = the number of colonies with some level of European foulbrood

[7] # AFB = the number of colonies with some level of American foulbrood

[8] # SAC = the number of colonies with some level of sacbrood

[9] # CB = the number of colonies with some level of chalkbrood

[10] # VM = an apiary is positive if varroa mites are detected in a survey using Apistan and sticky boards

[11] Aggressive = an apiary was considered aggressive if full protective gear was required and bees were persistent in pursuit

Table 5. Average ratings and scores for colony strength and health. Assessments done September 14-15, 2001.

Field Number	11	12	13	14	15
n	8	8	8	8	8
# Supers Start	3.1	3.8	3.8	3.8	3.4
Frames of Bees	24.4	32.0	34.4	33.0	16.8
Brood (0-2)	0.9	1.8	2.0	2.0	1.8
Honey (0-5)	3.7	2.7	4.6	4.4	1.8
# Laying	6	7	8	8	7
# EFB	0	1	0	0	0
# AFB	0	0	0	0	0
# SAC	0	0	0	0	0
# CB	2	0	1	1	8
# VM	-	+	-	-	+
Aggressive	Yes	No	No	No	No

[1] # Supers Start = the number of supers and brood boxes that make up the hive at the start of each inspection.
[2] Frames of Bees = the estimated number of frame equivalents that are 100% covered with a single layer of bees.
[3] Brood = a qualitative assessment of the amount of brood present scored on a scale of 0-2; 0 = no brood, 1 = some brood, 2 = lots of brood.
[4] Honey = a qualitative assessment of the amount of honey (both capped and uncapped) present based on a scale of 0-5; 0 = none, 1 = very little, 2 = little, 3 = moderate, 4 = lots, 5 = full
[5] # Laying = number of colonies with laying queens
[6] # EFB = the number of colonies with some level of European foulbrood
[7] # AFB = the number of colonies with some level of American foulbrood
[8] # SAC = the number of colonies with some level of sacbrood
[9] # CB = the number of colonies with some level of chalkbrood
[10] # VM = an apiary is positive if varroa mites are detected in a survey using Apistan and sticky boards
[11] Aggressive = an apiary was considered aggressive if full protective gear was required and bees were persistent in pursuit

ADDITIONAL INFORMATION

Meteorological Data

Meteorological data was monitored for each collection period. Meteorological data was also provided by Agriculture Canada (Charlottetown), for the Kensington area of Prince Edward Island. The mean temperature for the period from June 1, 2001 to September 30, 2001 was slightly above normal (Figure 15). The mean rainfall for the same period was lower than normal, resulting in near drought-like conditions (Figure 16). Clover plants in fields in Prince Edward Island appeared smaller than usual, but the clover plants in New Brunswick were in good condition due to greater rainfall in the region.

Figure 15. Daily Temperature for Prince Edward Island (June 1 – September 30, 2001)

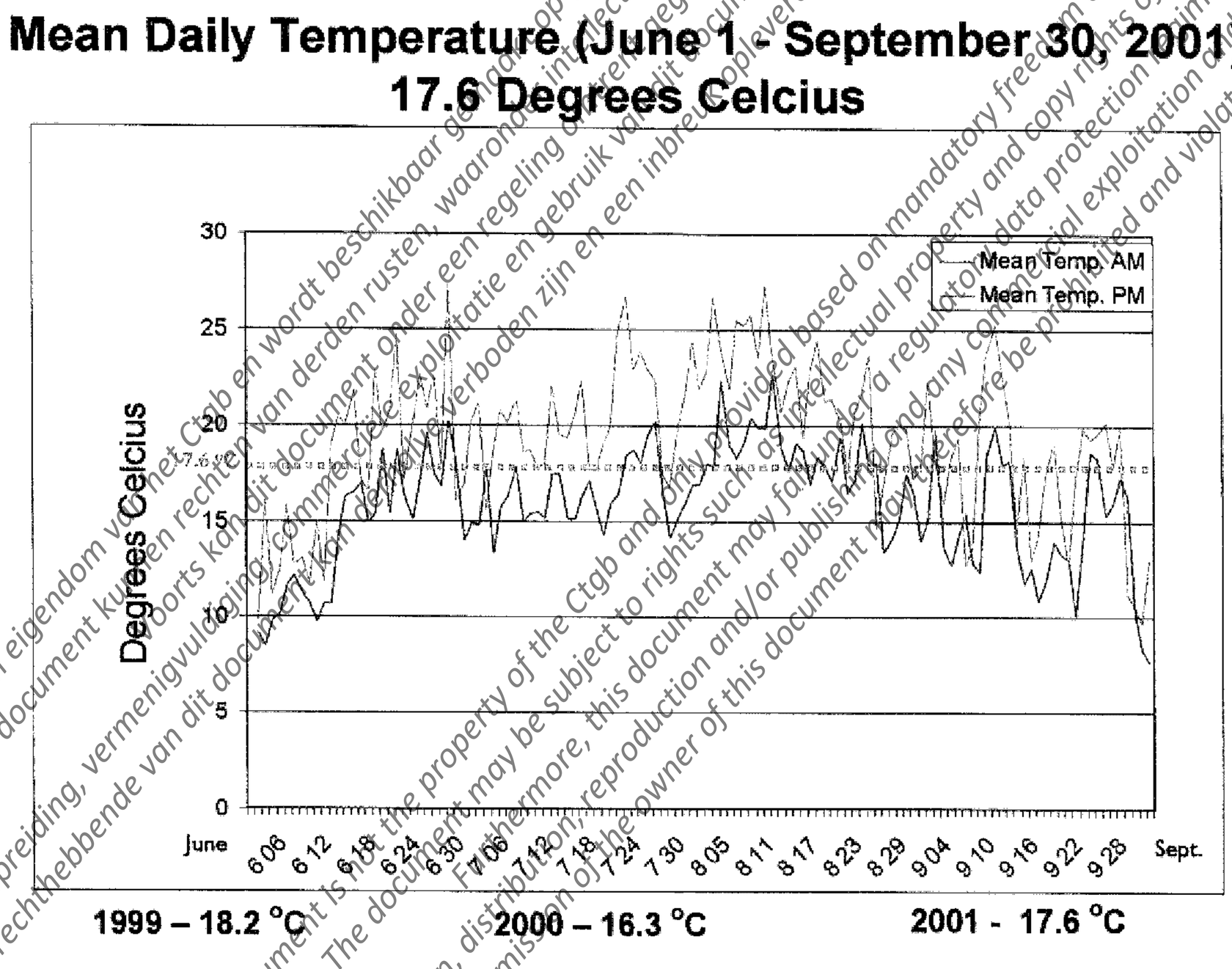
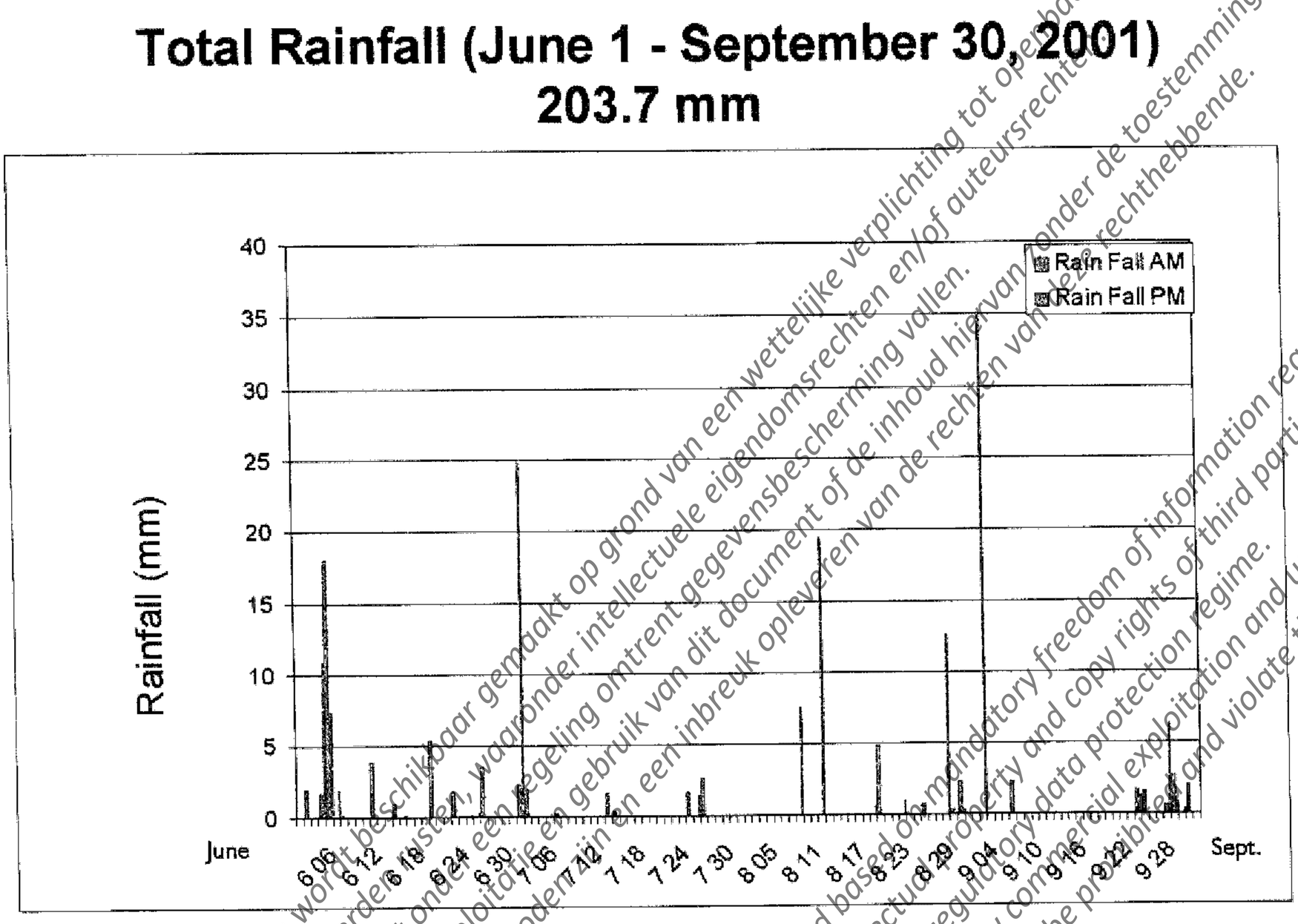


Figure 16. Rainfall for Prince Edward Island (June 1 – September 30, 2001)



1999 – 483.6 mm **2000 – 268.6 mm** **2001 – 203.7 mm**

SUMMARY

Residue levels of imidacloprid were detected in soil in all treated fields. The edges of sloped fields in first year rotation (i.e. potato fields) exhibited only one case of residue in soil. Metabolites were not included in the soil analysis because honey bees are not exposed to them in the soil. Three fields had residue levels of imidacloprid in clover leaves at just above detectable levels. Otherwise, all clover flowers, wildflowers, pollen, nectar, and uncapped honey did not have detectable levels of imidacloprid or its hydroxy and olefin metabolites (Figure 17). Data collected on bee colonies placed in clover fields that were previously treated with Admire®, did not indicate adverse effects during the time frame of this study.

Figure 17. Summary of Imidacloprid and imidacloprid metabolites residue levels in Prince Edward Island and New Brunswick. (Olefin and Hydroxy)

	Imidacloprid	Metabolites
Soil	Yes: Field 10 of 10 Runoff 1 of 6	Not analyzed
Clover Leaves	Very Low: 3 of 11	No
Clover Flowers	No	No
Wildflowers	No	No
Pollen	No	No
Nectar	No	No
Honey	No	No

The information contained in this report were presented at an imidacloprid conference in Calgary on January 30, 2002, at a research symposium jointly sponsored by the Canadian Association of Professional Apiculturists and Canadian Honey Council in Banff on February 1, 2002, and to the PEI beekeepers.

Some beekeepers are still concerned about the health of their bees and are asking the question: If imidacloprid is not detectable in pollen and nectar, then 1) Why are my bees still dying ? and 2) How can I prevent further losses ? The following recommendations address these concerns.

1) Examine hives with problems to see if disease or management practices are creating part of the problem.
2) Examine the movement of hives to see if certain locations and agricultural practices in the vicinity of the hives are creating problems.
3) Examine honey bees for the presence of viruses and spiroplasmas.

A result of the Calgary/Banff meetings was that a Research Review Committee was formed to come up with recommendations and statements that would reflect the agreement amongst the CAPA and CHC memberships. The results are as follows.

1) Based on a substantial body of research to date there is no consistent evidence that imidacloprid when used as a systemic insecticide following label directions, poses a serious threat to honey bees and bumble bees.

2) Beekeepers in Canada are experiencing an increasing incidence of unexplained and substantial colony mortality. There is a need to conduct multi-year studies to investigate the potential factors responsible for colony losses including pesticides, diseases, pests, climate, nutrition, genetics, management and the interactions between these and other factors. Research should focus on regions that are experiencing high colony mortality and should include, but not be limited to studies of low level chronic exposure to contaminants.

3) It is recommended that the Pest Management Regulatory Agency (Health Canada) consider the affect of all new insecticide registrations, including systemic insecticides and genetically modified crops on pollinators.

4) It is recommended that the Canadian beekeeping industry propose to meet with pesticide manufacturers (Crop Life Canada) to establish relationships leading to expanded testing of pesticide impacts on pollinators. Particular emphasis should be directed to pesticides with new modes of action.

ACKNOWLEDGEMENTS

[redacted]

The authors [redacted] would be pleased to answer any further questions with regard to this study.

REFERENCES

[redacted] 1991. Imidacloprid – a new systemic Insecticide. Pflanzenschutz Nachrichten Bayer 44: 113-136.

[redacted] 1998. In: I. Ishaaya and D. Degheele (Eds.) Insecticides with Novel Modes of Action. Springer Publishing. Pp 50 – 73.

[redacted] 1999. No causal relationship between Gaucho® seed dressing in Sunflowers and the French bee syndrome. Pflanzenschutz Nachrichten Bayer 52(3): 257 – 299

[redacted] 2000. Factors involved in the French bee malady. Hivelights (Canadian Honey Council) 13(3): 22-24.

Appendix A

Enviro-Test Laboratories

Study Protocol

STUDY PROTOCOL

VALIDATION AND ANALYSIS OF RESIDUES OF IMIDACLOPRID, HYDROXY METABOLITE, AND OLEFIN METABOLITE IN NECTAR, SOIL, BIOTA, AND POLLEN

ENVIRO-TEST LABS PROJECT NO.: ETL01BAY03

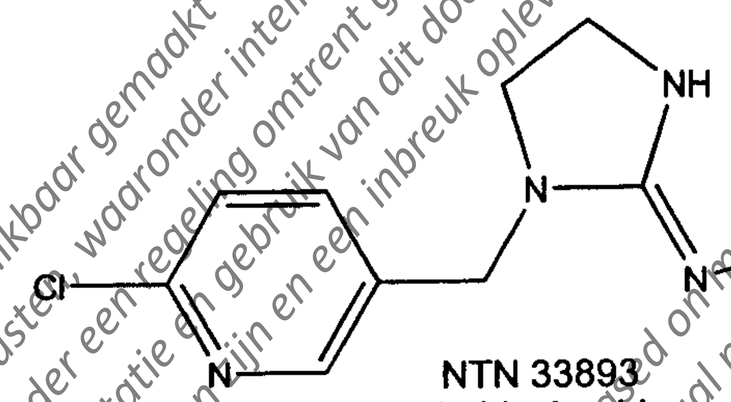
TEST SUBSTANCE: ADMIRE

REFERENCE SUBSTANCES: Imidacloprid, Hydroxy and Olefin Metabolites

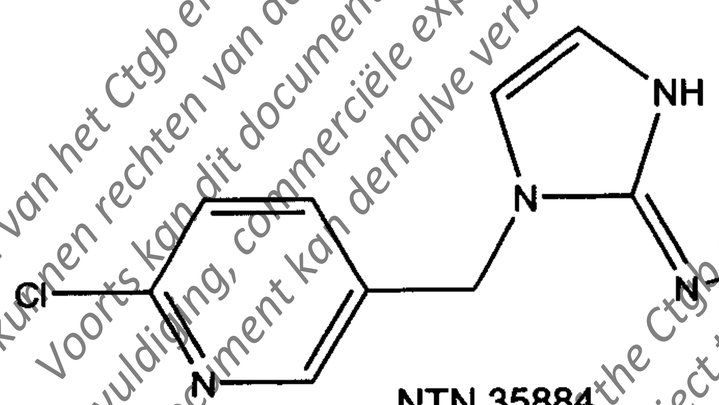
TEST SYSTEM: Soil, Nectar (Honey), Pollen and Plants

REGULATORY GUIDELINE: PMRA DIR 98-01, which outlines the requirements OECD GLP principles

STRUCTURAL FORMULAS:



NTN 33893
Imidacloprid



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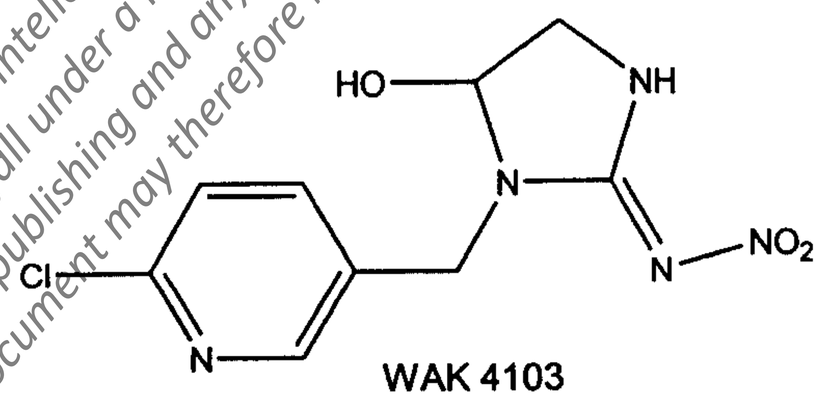


WAK 4103

PROPOSED INITIATION DATE: November 2001

PROPOSED COMPLETION DATE: January 2002

TEST FACILITY:

Enviro-Test Laboratories
9936 - 67 Avenue
Edmonton, Alberta T6E 0P5
Ph: (780) 413-5227 Fx: (780) 434-9178

STUDY SPONSOR:

BAYER Corp.
17745 South Metcalf
Stilwell, KS 66085-9104
Ph: (913) 433-5312 Fx: (913) 433-5389

APPROVALS:

Study Monitor: [redacted] Jan. 02, 2002
Date

Study Director: [redacted] Dec 7/01
Date

ETL Management: [redacted] Dec. 10/01
Date

Field Principal Investigator: [redacted] Jan 4, 2002
Date

Verified by:

Quality Assurance Unit: [redacted] Dec. 7, 2001
Date

STUDY OBJECTIVE:

The objectives of this demonstration study are to:

1) To determine LOD/LOQ and validate the modified analytical methods: Method No. 00554, Method No. 00537, Method 00537/E001 and Method 106428 (Soil Method dated Aug. 24/94).

2) To analyze soil, pollen, nectar (honey) and plant samples for Imidacloprid, NTN 35884 (Olefin metabolite) and WAK 4103 (Hydroxy metabolite).

The methods used for reference will be Bayer method no. 00537 (report no. MR-551/98), method no. 00537/E001 (report no. MR-568/99) and method no. 00554 (report no. MR-812/98).

TEST SUBSTANCES:

The Imidacloprid, Hydroxy-Metabolite, and Olefin Metabolite will be supplied by the Bayer Corporation. Characterization and archive of these test substances is the responsibility of the Sponsor. If available, a purity statement will accompany each shipment of material. Upon receipt and when not in use, the test substances will be stored in a freezer set at approximately -20°C, unless otherwise specified by the Sponsor.

TEST SYSTEM IDENTIFICATION:

Samples will be shipped to Enviro-Test Laboratories. All sample arrival conditions will be documented. Each sample will be logged in under LIMS and have a unique laboratory sample number for identification. All samples are to be stored in a freezer set at approximately -20°C.

Control nectar (liquid honey) will be obtained from hives operated by [redacted]. All controls will be analyzed for Imidacloprid and metabolites.

VALIDATION PROCEDURES:

Phase 1. Range Finding

Method trials will be performed for range finding purposes. Controls and fortified samples will be analyzed and the LOD and LOQ will be determined for soil, nectar, pollen and plants for each analyte. The estimated LOQ will be in the 0.001 to 0.005 ppm range.

Bayer will provide the reference analytical methods: Method No. 00554, Method No. 00537, and Method 00537/E001.

The Analytical Principal Investigator and Study Director will review the methods and ensure that all analytical requirements can be met. Any changes to the methods will be discussed with the Study Monitor, validated and documented in the final report.

Phase 2. Validation

Prior to analyzing replicate fortified control samples, the lab will optimize instrument parameters for each analyte. A validation set will be done for soil, pollen, nectar and biota.

A method validation set for each matrix (soil, pollen, nectar (honey), and biota) will be prepared and analyzed. Each validation set will include:

1. Two control (unfortified) samples.
2. Three replicate fortifications at LOQ
3. Two replicate fortifications at 10 x LOQ

Following completion of the method validation trials, the Analytical Principal Investigator will contact the Study Director and Study Monitor with a summary of the results. If the individual recoveries at each spiking level fall between 70 and 120% and the relative standard deviation (RSD) of replicate measurements is less than 20%, then the validation trial is deemed acceptable and the field samples may be analyzed.

Phase 3. Analysis of Samples

All samples will be analyzed for Imidacloprid and metabolites. Each set of samples will have 2 spikes, one at the LOQ and the other at a higher level.

COMMUNICATIONS:

All communications between the Analytical Principal Investigator and the Study Director regarding the conduct of this study will be documented and retained as part of the study file.

STATISTICAL PROCEDURES:

Statistical calculations will be limited to average recoveries and RSD.

RECORDS TO BE MAINTAINED:

[redacted]

FINAL REPORT:

The DRAFT report of the study will be prepared and be submitted to the Field Principal Investigator and Study Monitor for review.

After addressing any comments/suggestions regarding the DRAFT report, ETL will submit a FINAL report, to the Field Principal Investigator.

RESPONSIBILITIES:

[redacted] will act as the Study Director and [redacted] will act as Study Monitor. These individuals will review analytical data and reports for method and protocol compliance.

AMENDMENTS/DEVIATIONS:

Any alterations to the project must be communicated in a timely manner and approved by the Study Director. The alteration will be documented describing the alteration, the reason for the alteration, and the effect on the study, if any.

SOP deviations and method revisions are to be documented in the raw data and approved by the Study Director.

QUALITY ASSURANCE/GLP COMPLIANCE:

[redacted]

Appendix B

Enviro-Test Laboratories

Residue Report

ENVIRO-TEST LABORATORIES (ETL)
9936 - 67 AVENUE
EDMONTON, ALBERTA T6E 0P5
CANADA

STUDY TITLE: Validation and Analysis of Residues of Imidacloprid, Hydroxy Metabolite, and Olefin Metabolite in Nectar, Soil, Biota and Pollen

AUTHORS: [redacted]

STUDY MONITOR: [redacted]

SPONSOR:
Bayer Corporation
Agriculture Division
17745 South Metcalf
Stilwell, KS 66085-9104
Ph: (913) 433-5312 Fx: (913) 433-5389

STUDY DIRECTOR: [redacted]

PERFORMING LABORATORY:
Enviro-Test Laboratories
9936 - 67 Avenue
Edmonton, Alberta T6E 0P5 Canada
Ph: (780) 413-5227 Fx: (780) 434-9178

STUDY DATES:
Study Initiation Date: Dec. 7, 2001
Analytical Initiation Date: January 2, 2002
Analytical Termination Date: January 24, 2002

PROTOCOL NO: ETL01BAY03

ETL REFERENCE NO.: 01BAY12.REP

REPORT DATE: January 25, 2002

TOTAL PAGES: 66

STATEMENT OF COMPLIANCE WITH GOOD LABORATORY PRACTICE STANDARDS

[redacted]

[redacted]

Date 1 juli 23, 2002

STATEMENT OF THE QUALITY ASSURANCE UNIT

[redacted]

[redacted]

[redacted]

Jan. 25, 2002
Date

STUDY TEAM APPROVAL PAGE

[redacted]

JAN. 25/2002.
Date

Date Jan 25/02

TABLE OF CONTENTS

APPENDICES:

1. SUMMARY

The objectives of this study was:

1) To determine LOD/LOQ and validate the modified analytical methods: Method No. 00554, Method No. 00537, Method 00537/E001 and Method 106428 (Soil Method dated Aug. 24/94).

2) To analyze soil, pollen, nectar (honey) and plant samples for Imidacloprid, NTN 35884 (Olefin metabolite) and WAK 4103 (Hydroxy metabolite).

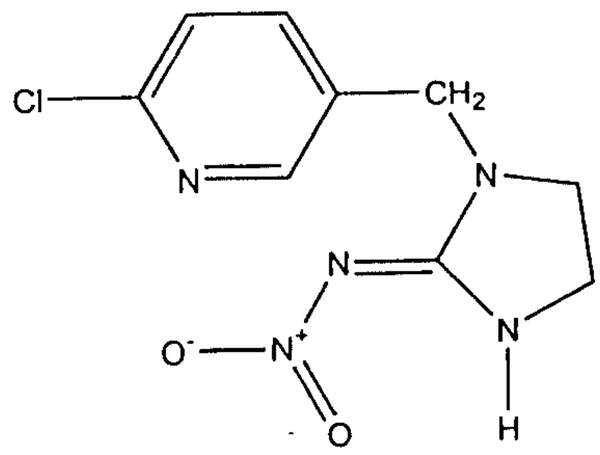
The methods used for reference was Bayer method no. 00537 (report no. MR-551/98), method no. 00537/E001 (report no. MR-568/99) and method no. 00554 (report no. MR-812/98).

2. MATERIALS

2.1 Reference Substances and Chemical Structures:

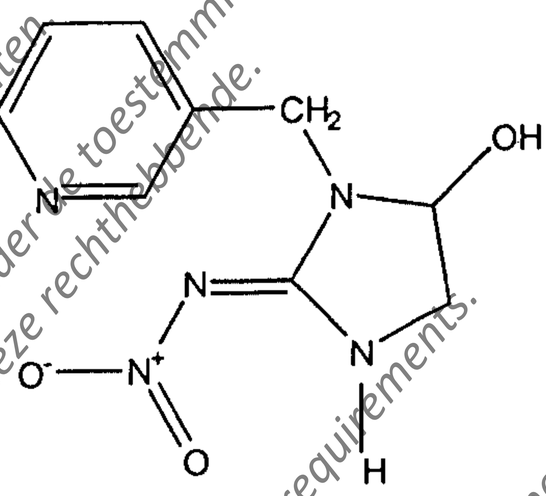
2.1.1 Characterization Information

Standard Name: Imidacloprid (NTN 33893)
ID#: K-664
Chemical Name: 1-[(6-chloro-3-pyridinyl)methyl]-N-nitro-2-imidazolidinimine
Date of Receipt: September 12, 2000
Reference No.: 93R-008-140
CAS No.: 138261-41-3
Expiration Date: June 19, 2003



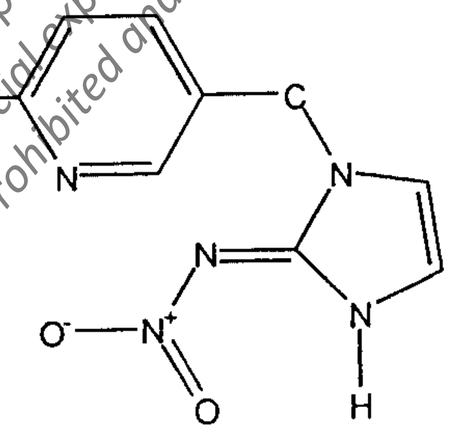
Imidacloprid (NTN 33893)

Standard Name: Hydroxy NTN 33893 (WAK 4103)
ID#: K-527
Chemical Name: 3-[(6-chloro-3-pyridinyl)methyl]-
2-nitroimino)-5-imidazolidnol
Date of Receipt: Sep. 12, 2000
Reference No.: 98r83-144
Expiration Date: July 19, 2004



Hydroxy NTN 33893

Standard Name: Olefin NTN 33893 (wak 3745)
ID#: K-954
Chemical Name: 1-[(6-chloro-3-pyridinyl)methyl]
-N-nitro-1H-imidazol-2-amine
Date of Receipt: November 21, 2001
Reference No.: M11453
Expiration Date: July 31, 2002



Olefin NTN

2.1.2 Standard Solution Preparation:

All standard solutions were stored at -15°C to -20°C with teflon lined screw caps.

2.1.2.1 Analyte Stock Solutions

Imidacloprid and Hydroxy neat standard (Lot No.: 93R-008-140, purity 98.4%, Lot No.: 98r 83-144, purity 99.3%) respectively, were received from Bayer on September 12, 2000. The Olefin neat standard (Lot No.: M11453, purity 98.6%), was received from Bayer on November 21, 2000. A nominal 1000 ppm of each stock solution was prepared in acetonitrile.

2.1.2.2 Analyte Fortification Solutions

Fortification standards for Imidacloprid and metabolites were prepared in concentrations of 100 ppm, 10.0 ppm, and 1.00 ppm in 20% acetonitrile/0.1% acetic acid.

2.1.2.3. Analyte Calibration Solutions

Calibration standards for Imidacloprid and metabolites were prepared in 20% acetonitrile/0.1% acetic acid. Calibration standards were in the range of 0.200 ppm to 0.00100 ppm.

2.1.2.5 Calibration Curve Standard Solutions for Imidacloprid, Hydroxy Met. And Olefin Met.

Conc./Vol. of Imidicloprid spiking solution (ppm)/(mL)	Conc./Vol. of Hydroxy Met. (ppm)/(mL)	Conc./Vol. of Olefin Met. standard (ppm)/(mL)	Imidacloprid Standard Conc. (ppm)	Hydroxy Met. Standard Conc. (ppm)	Olefin Met. Standard Conc. (ppm)
1801/0.555	745/1.34	1302/0.768	100	100	100
100/1.0	100/1.0	100/1.0	10.0	10.0	10.0
100/0.100	100/0.100	100/0.100	1.00	1.00	1.00
10.0/0.200	10.0/0.200	10.0/0.200	0.200	0.200	0.200
10.0/0.150	10.0/0.150	10.0/0.150	0.150	0.150	0.150
10.0/0.100	10.0/0.100	10.0/0.100	0.100	0.100	0.100
10.0/0.075	10.0/0.075	10.0/0.075	0.0750	0.0750	0.0750
10.0/0.050	10.0/0.050	10.0/0.050	0.0500	0.0500	0.0500
10.0/0.040	10.0/0.040	10.0/0.040	0.0400	0.0400	0.0400
10.0/0.020	10.0/0.020	10.0/0.020	0.0200	0.0200	0.0200
1.00/0.100	1.00/0.100	1.00/0.100	0.0100	0.0100	0.0100
1.00/0.050	1.00/0.050	1.00/0.050	0.00500	0.00500	0.00500
1.00/0.020	1.00/0.020	1.00/0.020	0.00200	0.00200	0.00200
1.00/0.010	1.00/0.010	1.00/0.010	0.00100	0.00100	0.00100

All solutions were brought to 10 mL final volume with 20% acetonitrile/0.1% acetic acid.

2.2 Reagents

Acetic acid - EM Science

Acetonitrile - OmniSolv® EM Science

Calcium chloride - Fischer Scientific

Dichloromethane - OmniSolv® EM Science

Methanol - OmniSolv® EM Science

Sodium chloride - EM Science

Sodium Sulfate - EM Science

Water - OmniSolv® EM Science

2.3 Equipment

Balance - (Mettler)

Controlled Environment Incubator Shaker - New Brunswick Scientific

Centrifuge - Sorvall Superspeed (RC2-B)

- International Equipment Company (Model HN-S)

Culture Tubes, 50 mL

Nitrogen Evaporator - (Meyer)

Rotary Evaporator

Standard Laboratory Glassware

Ultrasonic Bath - Fischer Scientific

Wrist Action Shaker - Burrel

2.4 INSTRUMENTS

2.4.1 LC/MS/MS:

PE SCIEX API III

Apple Power Macintosh 7600/132

Varian Solvent Delivery System

Waters Chromatography Syringe Pump

Rainin Dynamax® Automatic Sample Injector

Sciex Turbo-ionspray source

2.4.2 Operating Conditions

Column: Phenomenex C8, 15 cm x 4.6 mm, 5 um particle size

Column temperature: room temperature

Mobile phase A – 0.1% Acetic acid in water

Mobile phase B – 0.1% Acetic acid in acetonitrile

Flow rate – 1.0 mL/min

Injection volume – 20 uL

Gradient:

Time (min.)	%A	%B
0	50	50
3	20	80
5	20	80
6	50	50

Acquisition ions and retention times:

Analyte	Mass Transition	Approximate Retention time (min)
Imidacloprid	256.5 → 175.0	2:3
Hydroxy	272.5 → 190.7	2:0
Olefin	254.5 → 205.0	1:6

2.5 Sample Management:

Ninety-two samples were received at Enviro-Test Laboratories on July 24, October 25, October 31, 2001 and January 2, 2002. Samples were received in good condition and were immediately stored in the freezer at -25 ± 5 °C. Samples were logged into our system under file No. L1100.

2.5.1 Sample Preparation:

The nectar, honey, pollen and most of the flower samples were received processed and did not require further processing. Flower and leaf samples that were not received prepared, were prepared in a food processor in the presence of dry ice. Soil samples were sieved to a homogenous mixture. The % moisture was determined for all soil samples.

3.0 ANALYTICAL PROCEDURES

3.1 For Imidacloprid And Metabolites In Nectar And Honey

1. Weigh 1.0 g of nectar or honey sample into a 50 mL culture tube.
2. Fortify recovery sample prior to the extraction and allow sample to equilibrate.
3. Add 10 mL of water and place in ultrasonic bath for 2 min.
4. Add 20 mL methanol (MeOH) and shake on wrist shaker for 2 hours.
5. Centrifuge, decant and transfer to a 250 mL separatory funnel.
6. Add 20 mL of 3:1 MeOH:H_2O to the centrifuge bottle. Shake by hand and centrifuge. Combine the rinse to the original 250 mL sep. funnel.
7. Add 90 mL of water, and ~10g of NaCl, and partition 2 x with 40 mL of dichloromethane (DCM).
8. Combine the extracts into a 250 mL round bottom flask through regular baked sodium sulphate (Na_2SO_4).
9. Rinse Na_2SO_4 with DCM.
10. Evaporate the sample on a rotary evaporator (~40°C).
11. Transfer extract with DCM, and take to dryness on the Nitrogen evaporator.
12. Dissolve residue in 20% acetonitrile(ACN)/0.1% Acetic acid solution and bring to 1 mL final volume.
13. Store extracts in the freezer until ready for LC/MS/MS analysis.

3.2 For Imidacloprid And Metabolites In Pollen, Flower And Leaves

1. Weigh 2.0 g of sample into a 250 mL centrifuge bottle.
2. Fortify recovery sample prior to the extraction and allow sample to equilibrate.
3. Add 30 mL of 3:1 MeOH:H_2O, and allow the sample to soak for 30 minutes.
4. Put on wrist shaker, and shake for 2 hours.
5. Centrifuge, decant and transfer to a 250 mL separatory funnel.
6. Add 30 mL of 3:1 MeOH:H_2O to the centrifuge bottle. Shake by hand and centrifuge. Combine the rinse to the original 250 mL sep. funnel.
7. Add 90 mL of water, and ~10g of NaCl, and partition 2 x with 40 mL of dichloromethane (DCM).
8. Combine the extracts into a 250 mL round bottom flask through regular baked Na_2SO_4.
9. Rinse Na_2SO_4 with DCM.
10. Evaporate the sample on a rotary evaporator (~40°C).
11. Transfer extract with DCM, and take to dryness on the Nitrogen evaporator.
12. Dissolve residue in 20% ACN/0.1% acetic acid solution and bring to 1 mL final volume.
13. Store extracts in the freezer until ready for LC/MS/MS analysis.

3.3 Procedure For Imidacloprid And Metabolites In Soil

1. Weigh 50 g of sample into a 250 mL centrifuge bottle.
2. Fortify recovery sample prior to the extraction and allow sample to equilibrate.
3. Add 20 mL of 0.01M calcium chloride solution, and place in ultrasonic bath for 2 min.
4. Add 180 mL of ACN and shake it on a platform shaker for 2 hours.
5. Centifuge, decant and transfer to a 2L separatory funnel.
6. Re-extract soil with 200 mL of 9:1 ACN/water, and shake for 1 hour.
7. Centrifuge and decant into the same sep. funnel.
8. Add ~1L of water, and ~50 g of NaCl, and partition 2 x with 150 mL of dichloromethane (DCM).
9. Combine the extracts into a 500 mL round bottom flask through regular baked Na_2SO_4.
10. Rinse Na_2SO_4 with DCM.
11. Evaporate the sample on a rotary evaporator (~40°C)
12. Transfer extract with DCM, and take to dryness on the Nitrogen evaporator.
13. Dissolve residue in 20% ACN/0.1% acetic acid solution and bring to 1 mL final volume.
14. Store extracts in the freezer until ready for LC/MS/MS analysis.

4.0 FIGURES

Figure 1: Flow Diagram of the Analysis of Imidacloprid and Metabolites in Nectar/Honey

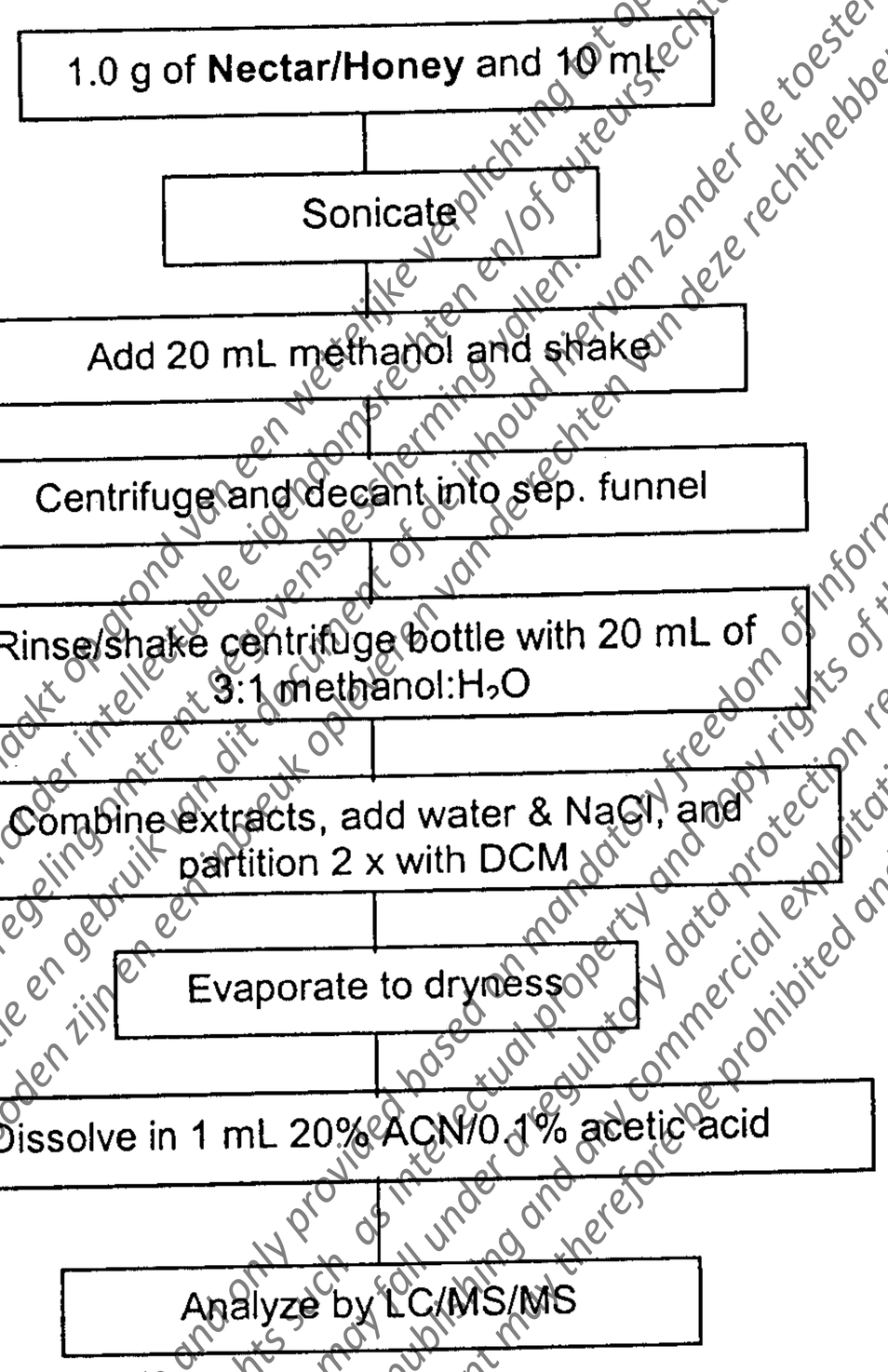


Figure 2: Flow Diagram of the Analysis of Imidacloprid in Flowers, Pollen, and Leaves

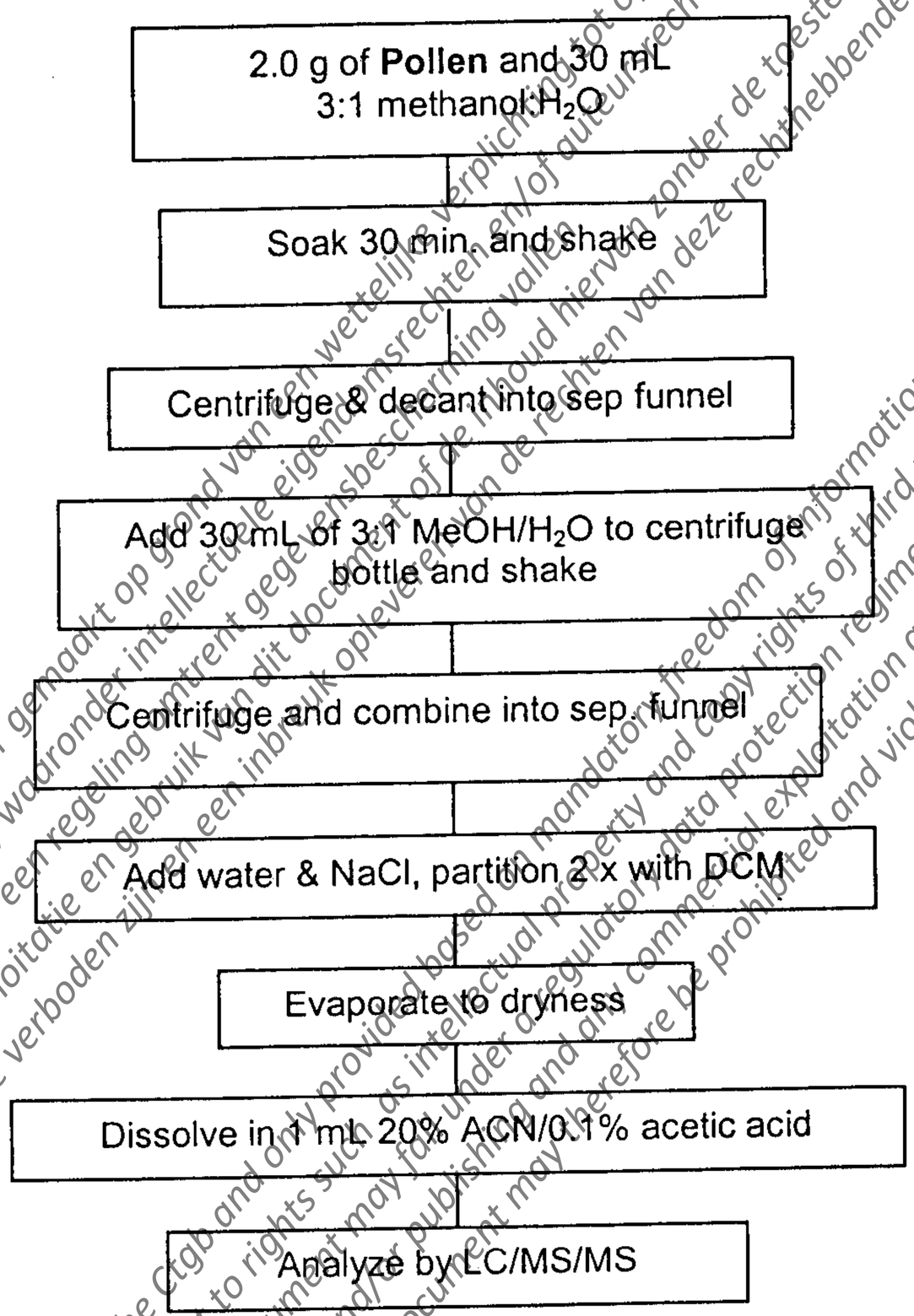
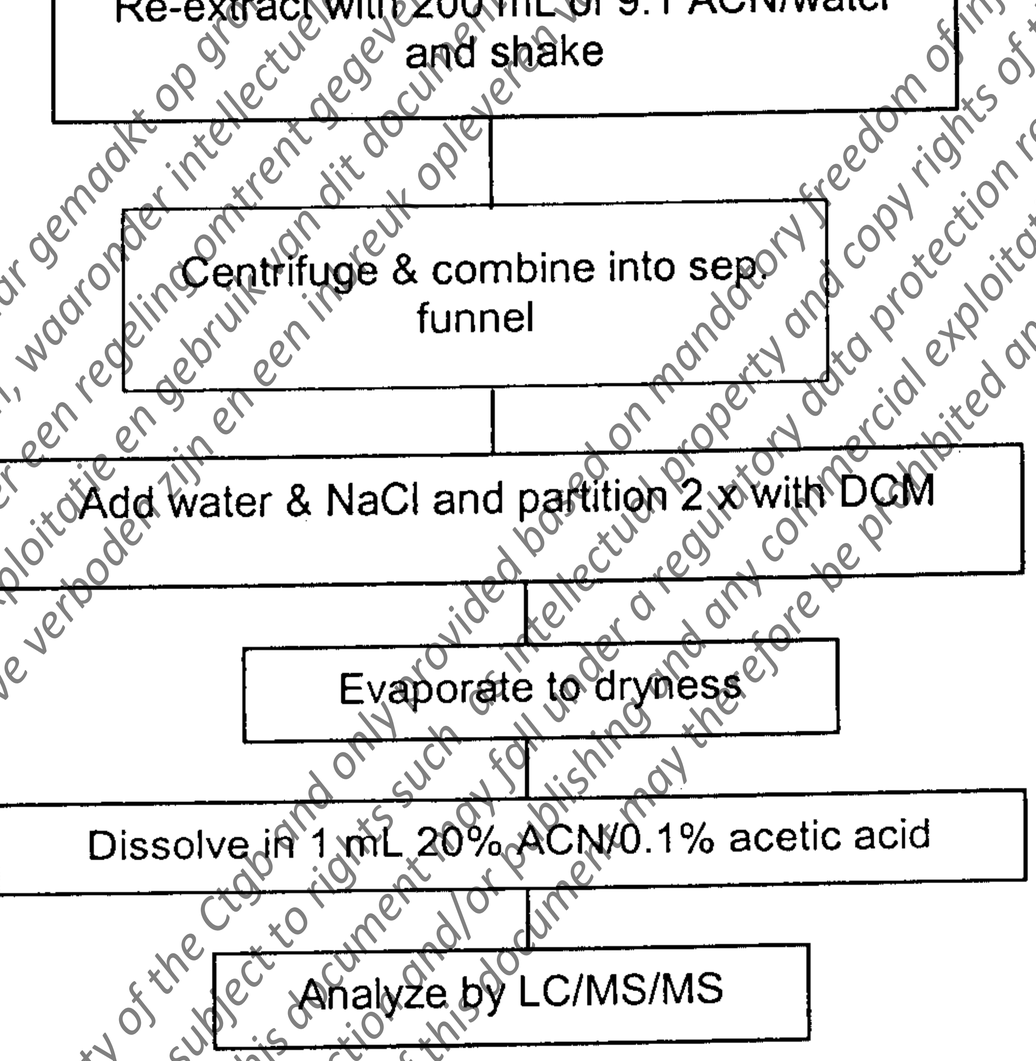


Figure 3: Flow Diagram of Imidacloprid in Soil



5.0 Calculation Procedures

Quantitation was accomplished by using linear thru zero regression from a eight to nine point calibration curve.

Quantitation by LC/MS/MS was performed using a linear regression plot of calibration standards injected intermittenly between samples of an analytical sample

5.1 Concentration of Analyte

$$\text{Conc.(ug/g)} = \frac{\left[\frac{\text{Peak Area} - \text{Intercept}}{\text{Slope}}\right] \times \text{Dilution Factor (D.F.)}}{*1000}$$

Where:

$$\text{D.F.} = \frac{\text{Final Volume (mL)}}{\text{Sample Mass (g)}}$$

* ppb/1000 = ppm (µg/g)

Intercept = 0

5.2 Percent Recovery

$$\%\,\text{Recovery} = \frac{\text{Amount Recovered (ppm)}}{\text{Amount Fortified (ppm)}} \times 100$$

5.3 Example of a Field Sample Calculation

Compound: Imidacloprid
Sample I.D.: LT100-50
Matrix: Field Soil
Analysis Date: January 11, 2002
File Name: NB-0111C01 (See Appendix 1)

$$\text{D.F.} = \frac{10\ \text{mL}}{50\ \text{g}} = 0.2$$

$$\frac{\left[\frac{61282 - 0}{386}\right] \times 0.2}{1000} = 0.032\ \text{ppm}$$

Based on wet weight.

Result ppm corrected for % moisture.

$$\text{Conc.(ppm)dryweight} = \text{Conc. (ppm)} \times \left[\frac{1}{\left(1 - \left(\frac{\%\,\text{Moisture}}{100}\right)\right)}\right]$$

$$= 0.032 \times \left[\frac{1}{\left(1 - \left(\frac{15}{100}\right)\right)}\right] = 0.038 \text{ ppm}$$

5.4 Example of a Recovery Calculation

Compound: Imidacloprid
Sample I.D.: L1100-161+8
Matrix: Soil
Analysis Date: January 11, 2002
File Name: NB0111C004 (See Appendix 1)

$$\text{D.F} = \frac{2.0 \text{ mL FV}}{50 \text{ g soil}} = 0.040$$

$$\text{ppb} = \left[\frac{(20780 - 0)}{386}\right] \times 0.040 = 2.15 \text{ ppb}$$

$$\text{ppm} = \frac{2.15 \text{ ppb}}{1000} = 0.00215 \text{ ppm}$$

$$\%\,\text{Recovery} = \frac{\text{ppm found}}{\text{ppm spiked}} \times 100$$

$$= \frac{0.00215}{0.00200} \times 100 = 108\%$$

6.0 Discussion and Conclusion

The average verification recoveries and SD for Imidacloprid in runoff soil was 99±7.9%. See Table 1 for recoveries. The average verification recoveries and SD for Imidacloprid in field soil was 90±22%. See Table 2 for recoveries.

The average verification recoveries and SD for Imidacloprid, Hydroxy metabolite, and Olefin metabolite in clover flowers were 96±9.8%, 92±10%, and 96±15% respectively. See Table 3 for recoveries.

The average verification recoveries and SD for Imidacloprid, Hydroxy metabolite, and Olefin metabolite in clover leaves were 110±6.6%, 105±14%, and 92±7.3% respectively. See Table 4 for recoveries.

The average verification recoveries and SD for Imidacloprid, Hydroxy metabolite, and Olefin metabolite in goldenrod flowers were 112±3.6%, 112±4.3%, and 107±10% respectively. See Table 5 for recoveries.

The average verification recoveries for Imidacloprid, Hydroxy metabolite, and Olefin metabolite in pollen was 104±16%, 96±18%, and 89±15% respectively. See Table 7 for recoveries.

The average verification recoveries and SD for Imidacloprid, Hydroxy metabolite, and Olefin metabolite in honey were 101±9.3%, 85±19%, and 91±22% respectively. See Table 8 for recoveries.

The in-phase recoveries for Imidacloprid in field soil was 91%. See Table 2 for recoveries. Residues of Imidacloprid in treated field soil samples ranged from <0.002 ppm to 0.038 ppm. See Table 9 for results.

The in-phase recoveries for Imidacloprid in runoff soil was 98%. See Table 1 for recoveries. Residues of Imidacloprid in treated runoff soil samples ranged from <0.002 ppm to 0.0037 ppm. Samples RS-31, RS-34 and RS-37 were re-submitted by client and analyzed to confirm initial results. See Table 10 for results.

The in-phase recoveries for Imidacloprid, Hydroxy metabolite, and Olefin metabolite clover flowers were 87%, 85% and 90% respectively. See Table 3 for recoveries. No residues of Imidacloprid, Hydroxy metabolite, or Olefin metabolite were found in treated clover flower samples. See Table 11 for results.

The in-phase recoveries for Imidacloprid, Hydroxy metabolite, and Olefin metabolite in clover leaves were 88%, 90%, and 79%, respectively. See Table 4 for recoveries. Residues of Imidacloprid treated clover leaves samples ranged from <0.002 ppm to 0.0044 ppm. See Table 12 for results.

The in-phase recoveries for Imidacloprid, Hydroxy metabolite, and Olefin metabolite goldenrod flowers were 106%, 96% and 105% respectively. See Table 5 for recoveries. No residues of Imidacloprid, Hydroxy metabolite, or Olefin metabolite were found in treated fireweed or aster flower samples. See Table 14 for results.

The in-phase recoveries for Imidacloprid, Hydroxy metabolite, and Olefin metabolite in pollen were 96%, 94% and 74% respectively. See Table 7 for recoveries. No residues of Imidacloprid, Hydroxy metabolite, or Olefin metabolite were found in treated pollen samples. See Table 15 for results.

The in-phase recoveries for Imidacloprid, Hydroxy metabolite, and Olefin metabolite in honey were 101±9.3%, 85±19% and 91±22% respectively. See Table 8 for recoveries. No residues of Imidacloprid, Hydroxy metabolite, or Olefin metabolite were found in treated nectar and honey samples. See Table 17 for results.

Appendix I contains a complete analytical data set.

The method worked well and gave good recoveries at levels near the LOQ.

7.0 RECOVERY AND RESULTS DATA

TABLE 1: RECOVERY DATA FOR IMIDACLOPRID IN RUNOFF SOIL

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	EXTRACTION DATE (m/d/y)	ANALYSIS DATE (m/d/y)	FORT. LEVEL (ppm)	RECOVERY LEVEL (ppm)	% RECOVERY
Verification							
L1100-25-1	Control Runoff Soil	Imidacloprid	1/8/02	1/11/02	---	<0.002	---
L1100-25-2	Control Runoff Soil	Imidacloprid	1/8/02	1/11/02	---	<0.002	---
L1100-25+1	Control Runoff Soil	Imidacloprid	1/8/02	1/11/02	0.00200	0.00209	105
L1100-25+2	Control Runoff Soil	Imidacloprid	1/8/02	1/11/02	0.00200	0.00209	105
L1100-25+3	Control Runoff Soil	Imidacloprid	1/8/02	1/11/02	0.00200	0.00204	102
L1100-25+4	Control Runoff Soil	Imidacloprid	1/8/02	1/11/02	0.00200	0.0173	87
L1100-25+5	Control Runoff Soil	Imidacloprid	1/8/02	1/11/02	0.00200	0.0188	94
In-Phase							
L1100-25+6	Control Runoff Soil	Imidacloprid	1/8/02	1/11/02	0.00200	0.00204	102
L1100-25+7	Control Runoff Soil	Imidacloprid	1/8/02	1/11/02	0.0200	0.0186	93

		Recovery	SD
VERIFICATION RUNOFF SOIL	Imidacloprid	99%	±7.9
IN-PHASE RUNOFF SOIL	Imidacloprid	98%	

7.0 RECOVERY AND RESULTS TABLES

TABLE 2: RECOVERY DATA FOR IMIDACLOPRID IN FIELD SOIL

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	EXTRACTION DATE (m/d/y)	ANALYSIS DATE (m/d/y)	FORT. LEVEL (ppm)	RECOVERY LEVEL (ppm)	% RECOVERY
Verification							
L1100-16-2	FS-Control Field Soil	Imidacloprid	1/8/02	1/11/02	---	<0.002	---
L1100-16-3	FS-Control Field Soil	Imidacloprid	1/8/02	1/11/02	---	<0.002	---
L1100-16-+3	FS-Control Field Soil	Imidacloprid	1/8/02	1/11/02	0.00200	0.00167	84
L1100-16+4	FS-Control Field Soil	Imidacloprid	1/8/02	1/11/02	0.00200	0.00142	71
L1100-16+5	FS-Control Field Soil	Imidacloprid	1/8/02	1/11/02	0.00200	0.00139	70
L1100-16+6	FS-Control Field Soil	Imidacloprid	1/8/02	1/11/02	0.0200	0.0219	110
L1100-16+7	FS-Control Field Soil	Imidacloprid	1/8/02	1/11/02	0.0200	0.0232	116
In-Phase							
L1100-16+8	FS-Control Field Soil	Imidacloprid	1/8/02	1/11/02	0.00200	0.00215	108
L1100-16+9	FS-Control Field Soil	Imidacloprid	1/8/02	1/11/02	0.0200	0.0145	73
L1100-16+10	FS-Control Field Soil	Imidacloprid	1/22/02	1/24/02	0.00200	0.00144	72
L1100-16+11	FS-Control Field Soil	Imidacloprid	1/22/02	1/24/02	0.0200	0.0144	72

		Recovery	SD
VERIFICATION FIELD SOIL	Imidacloprid	90%	±22
IN-PHASE FIELD SOIL	Imidacloprid	81%	

7.0 RECOVERY AND RESULTS TABLES

TABLE 4: RECOVERY DATA FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN CLOVER LEAVES cont'd

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	EXTRACTION DATE (m/d/y)	ANALYSIS DATE (m/d/y)	FORT. LEVEL (ppm)	RECOVERY LEVEL (ppm)	% RECOVERY
In-Phase							
L1100-53+8	CL-Control Clover Leaves	Imidacloprid	1/9/02	1/15/02	0.00200	0.00152	76
		Hydroxy Met.		1/15/02	0.00200	0.00145	73
		Olefin Met.		1/15/02	0.00200	0.00129	64
L1100-53+9	CL-Control Clover Leaves	Imidacloprid	1/9/02	1/15/02	0.0200	0.0201	100
		Hydroxy Met.		1/15/02	0.0200	0.0211	106
		Olefin Met.		1/15/02	0.0200	0.0188	94

		Recovery	SD
VERIFICATION LEAVES	Imidacloprid	110%	±6.6
	Hydroxy Met.	105%	±14
	Olefin Met.	92%	±7.3
IN-PHASE LEAVES	Imidacloprid	88%	
	Hydroxy Met.	90%	
	Olefin Met.	79%	

7.0 RECOVERY AND RESULTS TABLES

TABLE 5: RECOVERY DATA FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN GOLDENROD FLOWERS

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	EXTRACTION DATE (m/d/y)	ANALYSIS DATE (m/d/y)	FORT. LEVEL (ppm)	RECOVERY LEVEL (ppm)	% RECOVERY
Verification							
L1100-65-1	GF- Control GoldenRod Fl	Imidacloprid	1/9/02	1/15/02	---	<0.002	---
		Hydroxy Met.		1/15/02	---	<0.002	---
		Olefin Met.		1/15/02	---	<0.002	---
L1100-65-2	GF- Control GoldenRod Fl	Imidacloprid	1/9/02	1/15/02	---	<0.002	---
		Hydroxy Met.		1/15/02	---	<0.002	---
		Olefin Met.		1/15/02	---	<0.002	----
L1100-65+1	GF- Control GoldenRod Fl	Imidacloprid	1/9/02	1/15/02	0.00200	0.00211	106
		Hydroxy Met.		1/15/02	0.00200	0.00234	117
		Olefin Met.		1/15/02	0.00200	0.00209	105
L1100-65+2	GF- Control GoldenRod Fl	Imidacloprid	1/9/02	1/15/02	0.00200	0.00224	112
		Hydroxy Met.		1/15/02	0.00200	0.00222	111
		Olefin Met.		1/15/02	0.00200	0.00181	91
L1100-65+3	GF- Control GoldenRod Fl	Imidacloprid	1/9/02	1/15/02	0.00200	0.00229	115
		Hydroxy Met.		1/15/02	0.00200	0.00210	105
		Olefin Met.		1/15/02	0.00200	0.00230	115
L1100-65+4	GF- Control GoldenRod Fl	Imidacloprid	1/9/02	1/15/02	0.0200	0.0227	114
		Hydroxy Met.		1/15/02	0.0200	0.0224	112
		Olefin Met.		1/15/02	0.0200	0.0230	115
L1100-65+5	GF- Control GoldenRod Fl	Imidacloprid	1/9/02	1/15/02	0.0200	0.0227	114
		Hydroxy Met.		1/15/02	0.0200	0.0226	113
		Olefin Met.		1/15/02	0.0200	0.0222	111

7.0 RECOVERY AND RESULTS TABLES

TABLE 6: RECOVERY DATA FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN FIREWEED AND ASTER FLOWERS

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	EXTRACTION DATE (m/d/y)	ANALYSIS DATE (m/d/y)	FORT. LEVEL (ppm)	RECOVERY LEVEL (ppm)	% RECOVERY
In-Phase Fireweed							
L1100-67+1	FW- Control Fireweed Fl	Imidacloprid	1/9/02	1/15/02	0.00200	0.00188	94
		Hydroxy Met.		1/15/02	0.00200	0.00142	71
		Olefin Met.		1/15/02	0.00200	0.00173	87
L1100-67+2	FW- Control Fireweed Fl	Imidacloprid	1/9/02	1/15/02	0.00200	0.00205	103
		Hydroxy Met.		1/15/02	0.00200	0.00209	105
		Olefin Met.		1/15/02	0.00200	0.00197	99
L1100-67+3	FW- Control Fireweed Fl	Imidacloprid	1/9/02	1/15/02	0.0200	0.0208	104
		Hydroxy Met.		1/15/02	0.0200	0.0201	100
		Olefin Met.		1/15/02	0.0200	0.0186	93

		Recovery
IN-PHASE Fireweed Flowers	Imidacloprid	100%
	Hydroxy Met.	92%
	Olefin Met.	93%

7.0 RECOVERY AND RESULTS TABLES

TABLE 6: RECOVERY DATA FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN FIREWEED AND ASTER FLOWERS cont'd

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	EXTRACTION DATE (m/d/y)	ANALYSIS DATE (m/d/y)	FORT. LEVEL (ppm)	RECOVERY LEVEL (ppm)	% RECOVERY
In-Phase Aster							
L1100-57+1	AF- Control Aster Flower	Imidacloprid	1/9/02	1/15/02	0.00200	0.00164	82
		Hydroxy Met.		1/15/02	0.00200	0.00169	85
		Olefin Met.		1/15/02	0.00200	0.00140	70
L1100-57+2	AF- Control Aster Flower	Imidacloprid	1/9/02	1/15/02	0.00200	0.00154	77
		Hydroxy Met.		1/15/02	0.00200	0.00141	71
		Olefin Met.		1/15/02	0.00200	0.00147	74
L1100-57+3	AF- Control Aster Flower	Imidacloprid	1/9/02	1/15/02	0.0200	0.0187	94
		Hydroxy Met.		1/15/02	0.0200	0.0202	101
		Olefin Met.		1/15/02	0.0200	0.0169	85

		Recovery
IN-PHASE ASTER FLOWERS	Imidacloprid	84%
	Hydroxy Met.	86%
	Olefin Met.	76%

7.0 RECOVERY AND RESULTS TABLES

TABLE 7: RECOVERY DATA FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN POLLEN cont'd

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	EXTRACTION DATE (m/d/y)	ANALYSIS DATE (m/d/y)	FORT. LEVEL (ppm)	RECOVERY LEVEL (ppm)	% RECOVERY
L1100-73+7	Control Pollen	Imidacloprid	1/9/02	1/15/02	0.0200	0.0179	90
		Hydroxy Met.		1/15/02	0.0200	0.0165	83
		Olefin Met.		1/15/02	0.0200	0.0167	84
In-Phase							
L1100-73+8	Control Pollen	Imidacloprid	1/9/02	1/15/02	0.00200	0.00223	112
		Hydroxy Met.		1/15/02	0.00200	0.00210	105
		Olefin Met.		1/15/02	0.00200	0.00151	76
L1100-73+9	Control Pollen	Imidacloprid	1/9/02	1/15/02	0.0200	0.0160	80
		Hydroxy Met.		1/15/02	0.0200	0.0164	82
		Olefin Met.		1/15/02	0.0200	0.0142	72

		Recovery	SD
VERIFICATION POLLEN	Imidacloprid	104%	±16
	Hydroxy Met.	96%	±18
	Olefin Met.	89%	±15
IN-PHASE POLLEN	Imidacloprid	96%	
	Hydroxy Met.	94%	
	Olefin Met.	74%	

7.0 RECOVERY AND RESULTS TABLES

TABLE 8: RECOVERY DATA FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN HONEY

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	EXTRACTION DATE (m/d/y)	ANALYSIS DATE (m/d/y)	FORT. LEVEL (ppm)	RECOVERY LEVEL (ppm)	% RECOVERY
Verification							
L1100-89-2	Control Honey (G.B.)	Imidacloprid	1/9/02	1/11/02	---	<0.002	---
		Hydroxy Met.		1/11/02	---	<0.002	---
		Olefin Met.		1/11/02	---	<0.002	---
L1100-89-3	Control Honey (G.B.)	Imidacloprid	1/9/02	1/11/02	---	<0.002	---
		Hydroxy Met.		1/11/02	---	<0.002	---
		Olefin Met.		1/11/02	---	<0.002	---
L1100-89+3	Control Honey (G.B.)	Imidacloprid	1/9/02	1/11/02	0.00200	0.00233	117
		Hydroxy Met.		1/11/02	0.00200	0.00219	110
		Olefin Met.		1/11/02	0.00200	0.00250	125
L1100-89+4*	Control Honey (G.B.)	Imidacloprid	1/9/02	1/11/02	0.00200	0.00203	102
		Hydroxy Met.		1/11/02	0.00200	0.00147	74
		Olefin Met.		1/11/02	0.00200	<0.002	0*
L1100-89+4**	Control Honey (G.B.)	Imidacloprid	1/9/02	1/24/02	0.00200	0.00176	88
		Hydroxy Met.		1/24/02	0.00200	0.00141	70
		Olefin Met.		1/24/02	0.00200	0.00148	74
L1100-89+5	Control Honey (G.B.)	Imidacloprid	1/9/02	1/11/02	0.00200	0.00204	102
		Hydroxy Met.		1/11/02	0.00200	0.00149	75
		Olefin Met.		1/11/02	0.00200	0.00199	100

* Questionable injection
** Re-injection of spike.

7.0 RECOVERY AND RESULTS TABLES

TABLE 8: RECOVERY DATA FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN HONEY cont'd

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	EXTRACTION DATE (m/d/y)	ANALYSIS DATE (m/d/y)	FORT. LEVEL (ppm)	RECOVERY LEVEL (ppm)	% RECOVERY
L1100-89+6	Control Honey (G.B.)	Imidacloprid	1/9/02	1/11/02	0.0200	0.0195	98
		Hydroxy Met.		1/11/02	0.0200	0.0120	110
		Olefin Met.		1/11/02	0.0200	0.0156	78
L1100-89+7	Control Honey (G.B.)	Imidacloprid	1/9/02	1/11/02	0.0200	0.0199	100
		Hydroxy Met.		1/11/02	0.0200	0.0142	71
		Olefin Met.		1/11/02	0.0200	0.0154	77
In-Phase							
L1100-89+8	Control Honey (G.B.)	Imidacloprid	1/9/02	1/11/02	0.0200	0.00213	107
		Hydroxy Met.		1/11/02	0.0200	0.00186	93
		Olefin Met.		1/11/02	0.0200	0.00199	100
L1100-89+9	Control Honey (G.B.)	Imidacloprid	1/9/02	1/11/02	0.0200	0.0199	100
		Hydroxy Met.		1/11/02	0.0200	0.0109	55
		Olefin Met.		1/11/02	0.0200	0.0141	71

	Compound	Recovery	SD
VERIFICATION HONEY	Imidacloprid	104%	±7.6
	Hydroxy Met.	88%	±20
	Olefin Met.	95%	
IN-PHASE HONEY	Imidacloprid	104%	
	Hydroxy Met.	74%	
	Olefin Met.	86%	

7.0 RECOVERY AND RESULTS TABLES

TABLE 9: RESIDUE RESULTS FOR IMIDACLOPRID IN FIELD SOIL (Based on Dry Wt.)

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS Dry weight (ppm)
L1100-1	Control Field Soil	Imidacloprid	7/24/01	1/8/02	1/11/02	<0.002
L1100-5	FS-01 Field Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	0.020
L1100-6	FS-02 Field Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	0.016
L1100-7	FS-03 Field Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	0.038
L1100-8	FS-011 Field Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	0.017
L1100-9	FS-12 Field Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	0.024
L1100-10	FS-13 Field Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	0.014
L1100-11	FS-14 Field Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	0.025
L1100-12	FS-15 Field Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	<0.002
L1100-13	FS-21 Field Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	0.038
L1100-14	FS-22 Field Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	0.027
L1100-15	FS-23 Field Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	0.031
L1100-16-4	FS-Control Field Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	<0.002
L1100-16-5	FS-Control Field Soil	Imidacloprid	10/25/01	1/2202	1/24/02	<0.002

7.0 RECOVERY AND RESULTS TABLES

TABLE 10: RESIDUE RESULTS FOR IMIDACLOPRID IN RUNOFF SOIL (Based on Dry Wt.)

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS Dry weight (ppm)
L1100-17	RS-31 Runoff Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	<0.002
L1100-18	RS-32 Runoff Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	<0.002
L1100-19	RS-33 Runoff Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	<0.002
L1100-20	RS-34 Runoff Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	0.0028
L1100-21	RS-35 Runoff Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	<0.002
L1100-22	RS-36 Runoff Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	<0.002
L1100-23	RS-37 Runoff Soil	Imidacloprid	10/25/01	1/8/02	1/15/02	0.022*
L1100-24	RS-38 Runoff Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	<0.002
L1100-25-3	Control Runoff Soil	Imidacloprid	10/25/01	1/8/02	1/11/02	<0.002
L1100-90	RRS 31	Imidacloprid	10/25/01	1/2202	1/24/02	<0.002
L1100-91	RRS 34	Imidacloprid	10/25/01	1/2202	1/24/02	0.0037
L1100-92	RRS 37	Imidacloprid	10/25/01	1/2202	1/24/02	<0.002

* Possible sample labelling error. See L1100-92 result.

7.0 RECOVERY AND RESULTS TABLES

TABLE 11: RESIDUE RESULTS FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN CLOVER FLOWERS

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS (ppm)
L1100-3	Control Field Red Clover Flowers no in F.A.	Imidacloprid	7/24/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-26	CF-01 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-27	CF-02 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-28	CF-03 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-29	CF-11 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-30	CF-12 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-31	CF-13 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-32	CF-14 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002

7.0 RECOVERY AND RESULTS TABLES

TABLE 11: RESIDUE RESULTS FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN CLOVER FLOWERS cont'd

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS (ppm)
L1100-33	CF-15 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-34	CF-16 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-35	CF-17 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-36	CF-18 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-37	CF-19 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-38	CF-110 Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-39-4	CF-Control Clover Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002

7.0 RECOVERY AND RESULTS TABLES

TABLE 12: RESIDUE RESULTS FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN CLOVER LEAVES

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS (ppm)
L1100-2	Control Field Clover Leaves No in Furrow A	Imidacloprid	7/24/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-40	CL-11 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	0.0025
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-41	CL-12 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-42	CL-13 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-43	CL-14 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	0.0020
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-44	CL-15 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-45	CL-16 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002

7.0 RECOVERY AND RESULTS TABLES

TABLE 12: RESIDUE RESULTS FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN CLOVER LEAVES cont'd

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS (ppm)
L1100-46	CL-17 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-47	CL-18 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-48	CL-19 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-49	CL-110 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-50	CL-21 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	0.0044
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-51	CL-22 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-52	CL-23 Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-53-4	CL-Control Clover Leaves	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002

7.0 RECOVERY AND RESULTS TABLES

TABLE 13: RESIDUE RESULTS FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN GOLDENROD FLOWERS

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS (ppm)
L1100-4	CONT. FIELD GOLDENROD INFLORESCENCE NO IN F.A.	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-58	GF-31 Goldenrod Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-59	GF-32 Goldenrod Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-60	GF-33 Goldenrod Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-61	GF-34 Goldenrod Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-62	GF-35 Goldenrod Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-63	GF-36 Goldenrod Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002

7.0 RECOVERY AND RESULTS TABLES

TABLE 13: RESIDUE RESULTS FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN GOLDENROD FLOWERS cont'd

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS (ppm)
L1100-64	GF-37 Goldenrod Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-65-3	GF- Control GoldenRod Fl	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002

7.0 RECOVERY AND RESULTS TABLES

TABLE 14: RESIDUE RESULTS FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN ASTER AND FIREWEED FLOWERS

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS (ppm)
L1100-54	AF-31 Aster Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-55	AF-34 Aster Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-56	AF-37 Aster Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-57-1	AF-Control Aster Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-66	FW-31 Fireweed Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-67-1	FW-Control Fireweed Flowers	Imidacloprid	10/25/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002

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TABLE 15: RESIDUE RESULTS FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN POLLEN

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS (ppm)
L1100-68	BP-11 Pollen	Imidacloprid	10/31/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-69	BP-12 Pollen	Imidacloprid	10/31/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-70	BP-13 Pollen	Imidacloprid	10/31/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-71	BP-14 Pollen	Imidacloprid	10/31/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-72	BP-15 Pollen	Imidacloprid	10/31/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002
L1100-73-4	Control Pollen	Imidacloprid	10/31/01	1/9/02	1/15/02	<0.002
		Hydroxy Met.			1/15/02	<0.002
		Olefin Met.			1/15/02	<0.002

7.0 RECOVERY AND RESULTS TABLES

TABLE 16: RESIDUE RESULTS FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN NECTAR

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS (ppm)
L1100-74	BN-11 Nectar	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-75	BN-12 Nectar	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-76	BN-13 Nectar	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-77	BN-14 Nectar	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-78	BN-15 Nectar	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002

7.0 RECOVERY AND RESULTS TABLES

TABLE 17: RESIDUE RESULTS FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN HONEY

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS (ppm)
L1100-79	UH-11 Unripened Honey	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-80	UH-12 Unripened Honey	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-81	UH-13 Unripened Honey	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-82	UH-14 Unripened Honey	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-83	UH-15 Unripened Honey	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-84	UH-11.2 Unripened Honey	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-85	UH-12.2 Unripened Honey	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002

7.0 RECOVERY AND RESULTS TABLES

TABLE 17: RESIDUE RESULTS FOR IMIDACLOPRID, HYDROXY, AND OLEFIN METABOLITES IN HONEY cont'd

LAB SAMPLE #	SAMPLE I.D.	COMPOUND	DATE RECEIVED (m/d/y)	DATE EXTRACTED (m/d/y)	DATE ANALYZED (m/d/y)	RESULTS (ppm)
L1100-86	UH-13.2 Unripened Honey	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-87	UH-14.2 Unripened Honey	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-88	UH-15.2 Unripened Honey	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002
L1100-89-4	Control Honey (G.B.)	Imidacloprid	10/31/01	1/9/02	1/11/02	<0.002
		Hydroxy Met.			1/11/02	<0.002
		Olefin Met.			1/11/02	<0.002

APPENDIX I:
A COMPLETE ANALYTICAL DATA SET

MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:19
Calibration File: 011102Ccal Path: LinCir 8800 DATA La Cie 1250 BAYER Imidacloprid 011102
Comments: Imidacloprid (Field Soil Set#1)

Imidacloprid
No Internal Standard
256.5->175.0
Linear Thru' Zero
Intercept = 0
Slope = 386
Correlation Coeff. = 0.9986
Use Area

Filename	Filetype	Sample Name	Sample Desc.	Conc.	Area	Dil. Factor	Calc. Conc.	Accuracy
NB0111C001	Standard	IMIDACLOPRID	Imidacloprid 1 ppb	1.00	521	1.000	1.349	135
NB0111C002	Standard	IMIDACLOPRID	Imidacloprid 2 ppb	2.00	1122	1.000	2.905	145
NB0111C003	Sample	IMIDACLOPRID	L1100-16-4 1mL	n / a	194	0.020	0.010	n / a
NB0111C004	QC	IMIDACLOPRID	L1100-16-8 1mL	8.00	20780	0.040	2.153	108
NB0111C005	QC	IMIDACLOPRID	L1100-16+9 10mL	20.00	27985	0.200	14.497	72
NB0111C006	Standard	IMIDACLOPRID	Imidacloprid 5 ppb	5.00	1583	1.000	4.100	82
NB0111C007	Sample	IMIDACLOPRID	L1100-1 1mL (EF, NJ, JAN. 11/02)	n / a	76	0.040	0.008	n / a
NB0111C008	Sample	IMIDACLOPRID	L1100-5 10mL	n / a	31205	0.200	19.273	n / a
NB0111C009	Sample	IMIDACLOPRID	L1100-6 10mL	n / a	29257	0.200	15.156	n / a
NB0111C010	Standard	IMIDACLOPRID	Imidacloprid 10 ppb	10.00	3576	1.000	9.264	93
NB0111C011	Sample	IMIDACLOPRID	L1100-7 10mL	n / a	61282	0.200	31.746	n / a
NB0111C012	Sample	IMIDACLOPRID	L1100-8 10mL	n / a	30445	0.200	15.772	n / a
NB0111C013	Sample	IMIDACLOPRID	L1100-9 10mL	n / a	42050	0.200	21.783	n / a
NB0111C014	Standard	IMIDACLOPRID	Imidacloprid 20 ppb	20.00	7917	1.000	20.507	103
NB0111C015	Sample	IMIDACLOPRID	L1100-10 10mL	n / a	24510	0.200	12.697	n / a
NB0111C016	Sample	IMIDACLOPRID	L1100-11 10mL	n / a	44989	0.200	23.306	n / a
NB0111C017	Sample	IMIDACLOPRID	L1100-12 1mL (EE, NJ, JAN 12/02)	n / a	108	0.040	0.011	n / a
NB0111C018	Standard	IMIDACLOPRID	Imidacloprid 40 ppb	40.00	17671	1.000	45.771	114
NB0111C019	Sample	IMIDACLOPRID	L1100-13 10mL	n / a	61225	0.200	31.717	n / a
NB0111C020	Sample	IMIDACLOPRID	L1100-14 10mL	n / a	44518	0.200	23 062	n / a
NB0111C021	Sample	IMIDACLOPRID	L1100-15 10mL	n / a	49368	0.200	25 575	n / a
NB0111C022	Standard	IMIDACLOPRID	Imidacloprid 75 ppb	75.00	30310	1.000	78.509	105
NB0111C023	Standard	IMIDACLOPRID	Imidacloprid 100 ppb	100.00	40535	1.000	104.993	105
NB0111C024	Standard	IMIDACLOPRID	Imidacloprid 150 ppb	150.00	55353	1.000	143.375	96

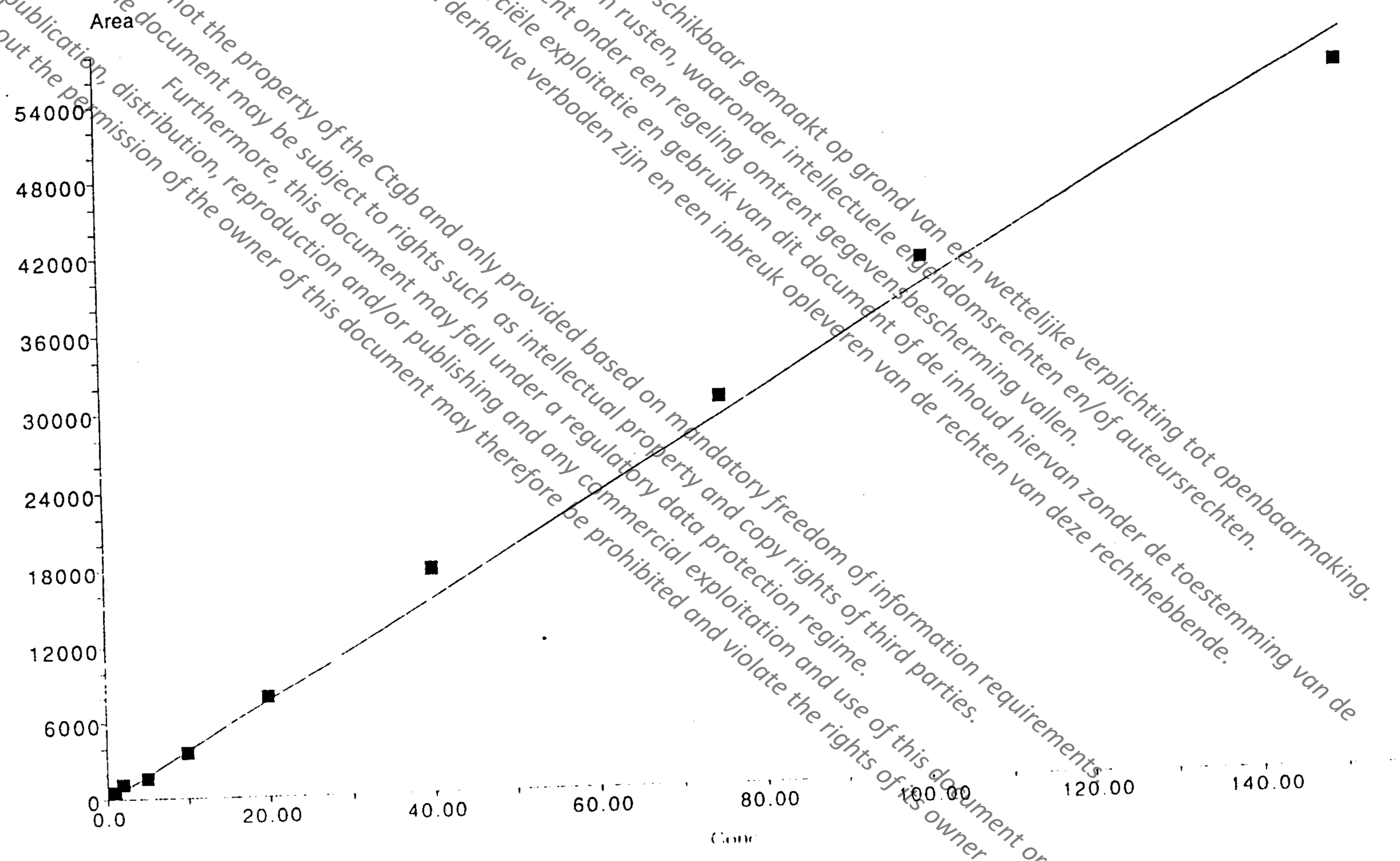
$$\text{Calc. Conc. (ppm)} = \left(\frac{\text{Area} - \text{Intercept}}{\text{Slope}} \right) \times \text{Dilution Factor} \times 1000$$

$$\text{Dilution Factor (D.F.)} = \frac{1 \text{ mL Final Volume}}{50 \text{ g}} = 0.020$$

NJ, JAN. 12/2002

MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:20
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102:
Comments: Imidacloprid (Field Soil Set# 1)

Imidacloprid 256.5->175.0 No Internal Standard
Linear Thru' Zero
Intercept = 0
Slope = 386
Correlation Coeff. = 0.9986



Study Plan: ETL01BAY03
% Moisture Calculation Worksheet

Lab sample No.	Client I.D.	Result (ppm)	% Moisture	Result (ppm) (dry weight)
L1100-1	CONTROL FIELD SOIL NO IN FURROW ADMIRE	<0.002	14	<0.002
L1100-5	FS-01 FIELD SOIL	0.019	6.0	0.020
L1100-6	FS-02 FIELD SOIL	0.015	8.0	0.016
L1100-7	FS-03 FIELD SOIL	0.032	15	0.038
L1100-8	FS-011 FIELD SOIL	0.016	8.0	0.017
L1100-9	FS-12 FIELD SOIL	0.022	7.0	0.024
L1100-10	FS-13 FIELD SOIL	0.013	7.0	0.014
L1100-11	FS-14 FIELD SOIL	0.023	7.0	0.025
L1100-12	FS-15 FIELD SOIL	<0.002	13	<0.002
L1100-13	FS-21 FIELD SOIL	0.032	15	0.038
L1100-14	FS-22 FIELD SOIL	0.023	14	0.027
L1100-15	FS-23 FIELD SOIL	0.026	15	0.031
L1100-16	FS-VALIDATION C.CONTROL FIELD SOIL	<0.002	8.0	<0.002
L1100-17	RS-31 RUNOFF SOIL	<0.002	14	<0.002
L1100-18	RS-32 RUNOFF SOIL	<0.002	13	<0.002
L1100-19	RS-33 RUNOFF SOIL	0.0026	7.0	0.0028
L1100-20	RS-34 RUNOFF SOIL	<0.002	8.0	<0.002
L1100-21	RS-35 RUNOFF SOIL	<0.002	8.0	<0.002
L1100-22	RS-36 RUNOFF SOIL	0.020	7.0	0.022
L1100-23	RS-37 RUNOFF SOIL	<0.002	9.0	<0.002
L1100-24	RS-38 RUNOFF SOIL	<0.002	13	<0.002
L1100-25	VALIDATION C.CONTROL RUNOFF SOIL		8.0	

*Moisture Correction: Conc. (ppm) x (1 / (1 - % Moisture / 100))

Enviro-Test Laboratories
Analysis of Imidacloprid in Soils by LC/MS/MS

Method Ref.: MS 254.00
Protocol no.: ETL01BAY03.PRO
Method Ref.: Bayer Rep. No. 106428

Set No.:Field Soil Set 1

Page 1 of 2

I. Sample Fortification:

Sample ID (ETL#)	Client ID	Analyte	STD Conc (ppm)	Fort Vol (mL)	Fort.Lev (ppm)
L1100-16+8	FS-VALIDATION C.CONTROL FIELD SOIL	Imidacloprid	1.00	0.100	0.00200
L1100-16+9	FS-VALIDATION C.CONTROL FIELD SOIL	Imidacloprid	10.0	0.100	0.0200

Pipette # used: #5 05-JAN-02

Spiking Std. ID.: D 279 a/c	Prep. Date: 21-NOV-01	Analyst: [redacted]	Date:
Witness for Fortification (Name, initials, date): [redacted] 08-JAN-02			

II. Samples Extracted:

Sample ID (ETL#)	Client ID	Smp. Wt. (g) 50.0	Extraction Vol. (mL)	Final Vol (mL)	Dilution	Dilution	Dil'n Date & Initials
L1100-16-4	FS-VALIDATION C.CONTROL FIELD SOIL	50.0	380	1.0	1:5 (EE), NB, Jan		JAN.11/02, NB
L1100-16+8	FS-VALIDATION C.CONTROL FIELD SOIL	50.0	"	1.0	1:2		JAN.11/02 NB
L1100-16+9	FS-VALIDATION C.CONTROL FIELD SOIL	50.0	"	1.0	1:10		JAN.11/02 NB
L1100-1	CONT.L FIELD SOIL NO IN FURROW ADMIRE	50.0	"	1.0	1:2		JAN.11/02 NB
L1100-5	FS-01 FIELD SOIL	50.0	"	1.0	1:10		JAN.11/02 NB
L1100-6	FS-02 FIELD SOIL	50.0	"	1.0	1:10		JAN.11/02 NB
L1100-7	FS-03 FIELD SOIL	50.0	"	1.0	1:10		JAN.11/02 NB
L1100-8	FS-011 FIELD SOIL	50.0	"	1.0	1:10		JAN.11/02 NB
L1100-9	FS-12 FIELD SOIL	50.0	"	1.0	1:10		JAN.11/02 NB
L1100-10	FS-13 FIELD SOIL	50.0	"	1.0	1:10		JAN.11/02 NB
L1100-11	FS-14 FIELD SOIL	50.0	"	1.0	1:10		JAN.11/02 NB
L1100-12	FS-15 FIELD SOIL	50.0	"	1.0	1:2		JAN.11/02 NB
L1100-13	FS-21 FIELD SOIL	50.0	"	1.0	1:10		JAN.11/02 NB
L1100-14	FS-22 FIELD SOIL	50.0	"	1.0	1:10		JAN.11/02 NB
L1100-15	FS-23 FIELD SOIL	50.0	"	1.0	1:10		JAN.11/02 NB

Container: 250 mL centr. bottle	
Balance I.D. #5	
Date: Jan 04/02	[redacted]
Signature: [redacted]	08-JAN-02

Enviro-Test Laboratories

Analysis of Imidacloprid in Soils by LC/MS/MS

Method Ref.: MS 254.00
Protocol no.: ETL01BAY03.PRO
Method Ref.: Bayer Rep. No. 106428

Set No.:Field Soil Set 1

III. Method Summary

Soil Extraction

Weigh 50 g of soil sample into a 250 mL centrifuge bottle.

1 Fortify recovery sample prior to the extraction and allow sample to equilibrate.
2 Add 20 mL of 0.01 M calcium chloride solution. (AG # 2057) Place in ultrasonic bath for 2 min.
3 Add 180 mL of ACN and shake it on a platform shaker for two hours.
4 Centrifuge and decant into a sep. funnel.
5 Re-extract soil with 200 mL of 9:1 ACN / Water (AG# 2066) and shake for 1 hr.
6 Centrifuge and decant into the same sep. funnel.
7 To the sep. funnel add about 1 L of water and ~ 50 g of NaCL and partition twice with 150 mL DCM.
8 Combine the DCM extracts intoa 250 mL round-bottom flask through regular Na_2SO_4.
9 Rinse the Na_2SO_4 with DCM.
10 Evaporate the sample on a rotary evaporator (~40°C).
11 Transfer with DCM to a vial and take it to dryness using the Nitrogen evaporator.
12 Dissolve the residue in 20%ACN/0.1%Acetic acid solution and bring to a final volume of 1.0 mL.
13 Analyze by HPLC-MS/MS (SIM).

	Initials:	Date:
1	DE	08-JAN-02
2	DE	"
0	DE	"
3	DE	"
4	DE	"
5	DE	"
6	DE	"
7	DE	"
8	DE	"
9	DE	"
10	DE	"
11	DE	"
12	DE	"
13	NB	JAN. 11/02

Deviations from Method:

Comments:

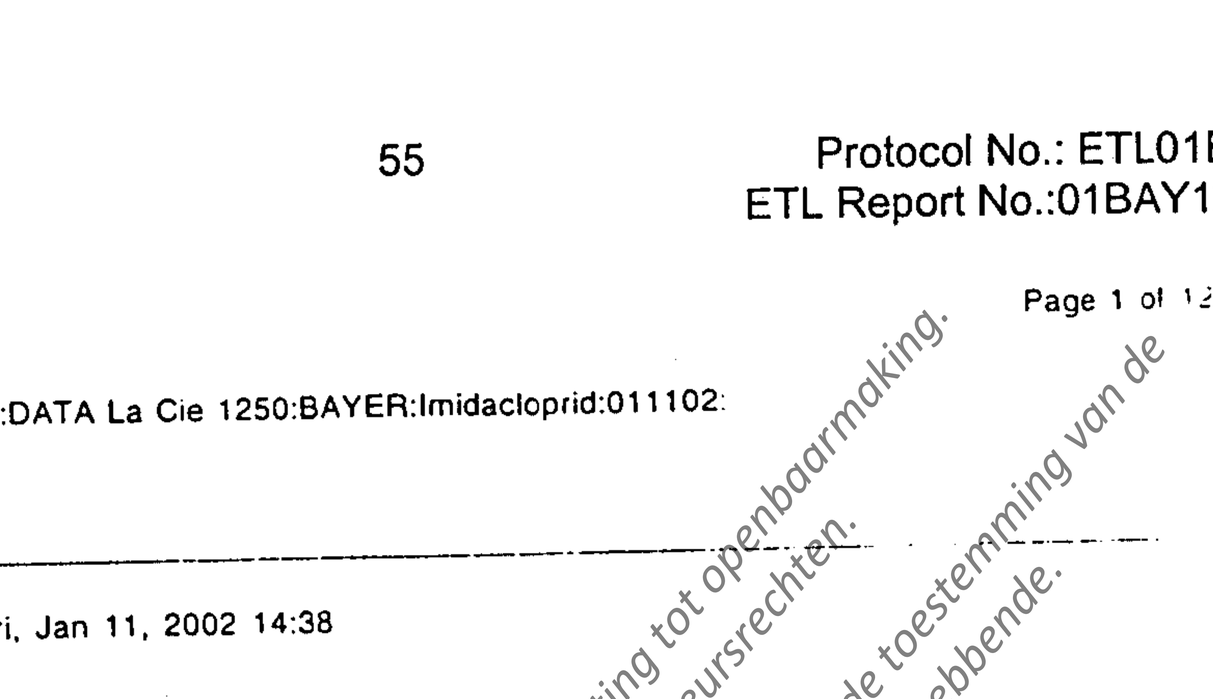
MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:21
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102:
Comments: Imidacloprid (Field Soil Set#1)

NB0111C001 **IMIDACLOPRID** Fri, Jan 11, 2002 14:38
Imidacloprid 1 ppb

5:32 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:01 MRM. 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	521
Height	73
Start Time	2:27
End Time	2:44
Integration Width	0:17.1
Retention Time	2:37
Integration Type	A - BV

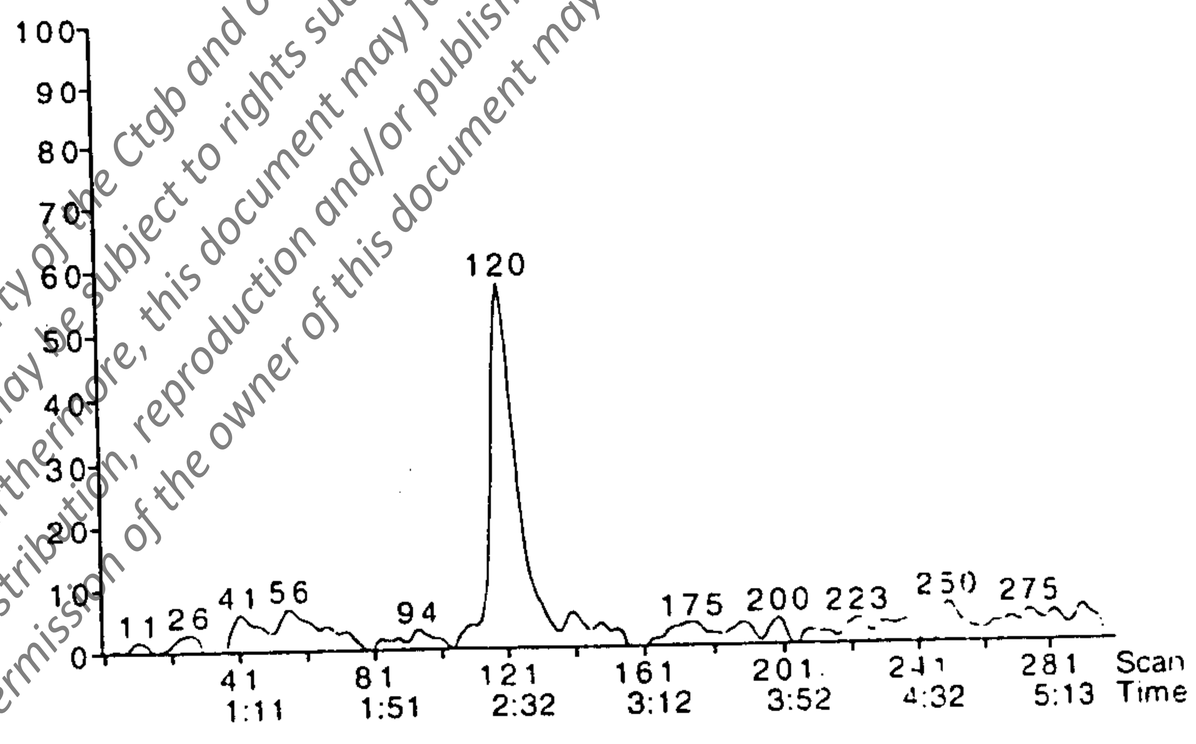


NB0111C002 **IMIDACLOPRID** Fri, Jan 11, 2002 14:45
Imidacloprid 2 ppb

5:32 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:01 MRM. 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	1.0
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	1122
Height	136
Start Time	2:19
End Time	2:45
Integration Width	0:26.2
Retention Time	2:31
Integration Type	A - BB



NB, JAN. 12/2002
PAGES 1 to 12

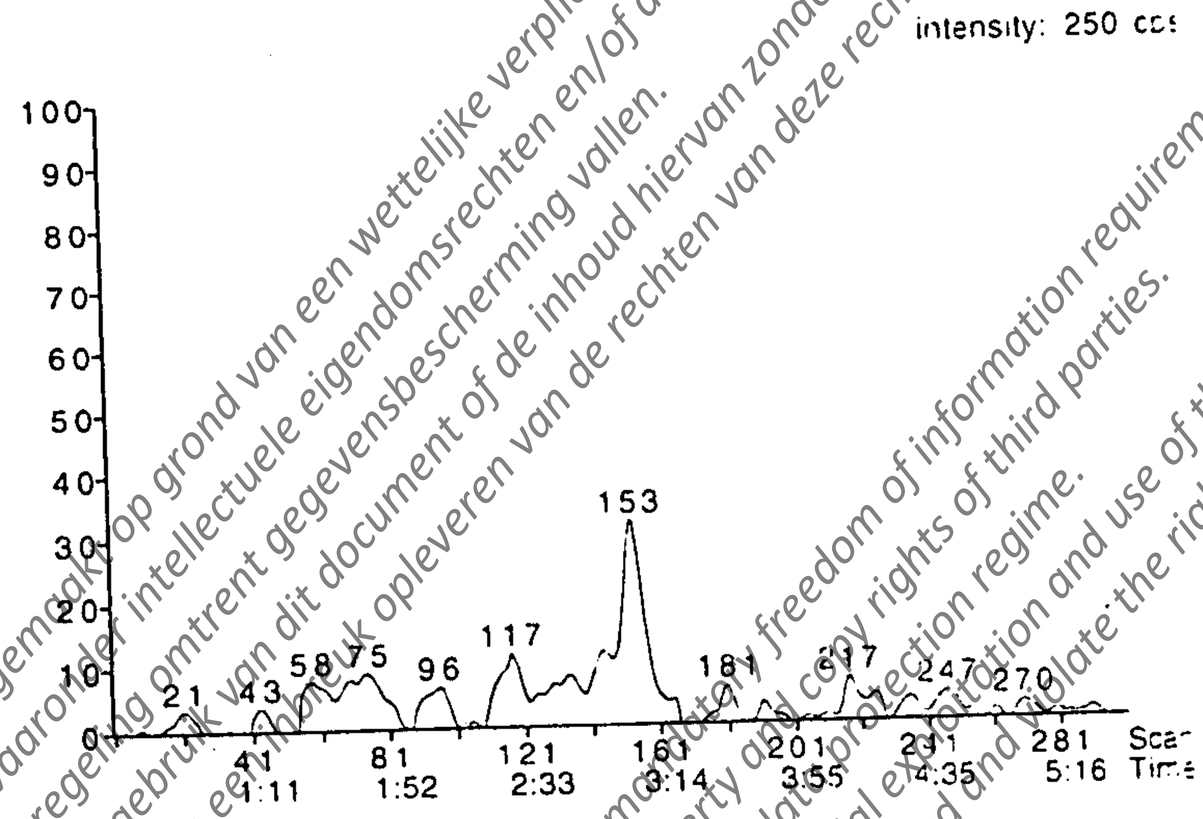
MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:21
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102:
Comments: Imidacloprid (Field Soil Set#1)

NB0111C003 **IMIDACLOPRID** Fri, Jan 11, 2002 14:51
L1100-16-4 1mL

5:35 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	194
Height	25
Start Time	2:21
End Time	2:33
Integration Width	0:12.2
Retention Time	2:29
Integration Type	A - BV

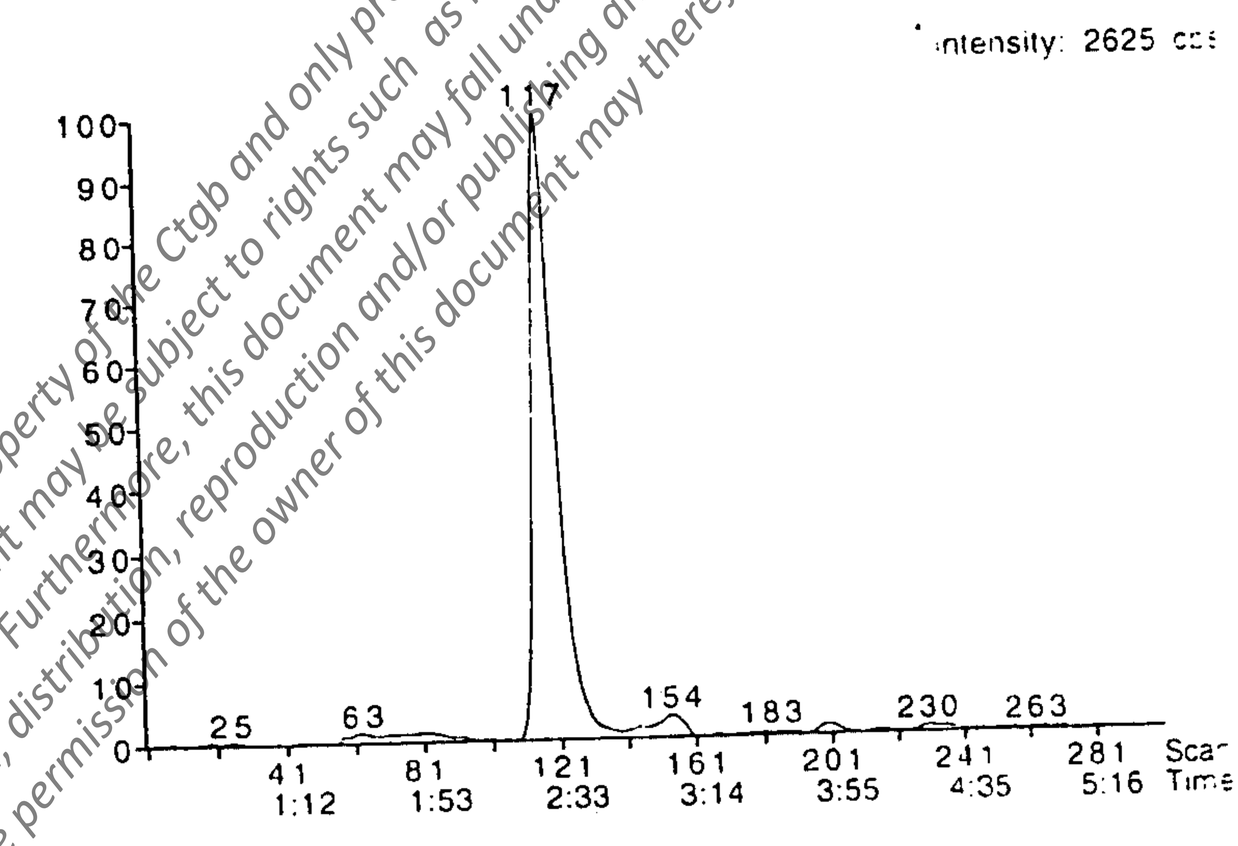


NB0111C004 **IMIDACLOPRID** Fri, Jan 11, 2002 14:58
L1100-16/8 1mL

5:36 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	20780
Height	2623
Start Time	2:21
End Time	2:52
Integration Width	0:30.2
Retention Time	2:29
Integration Type	A - BV



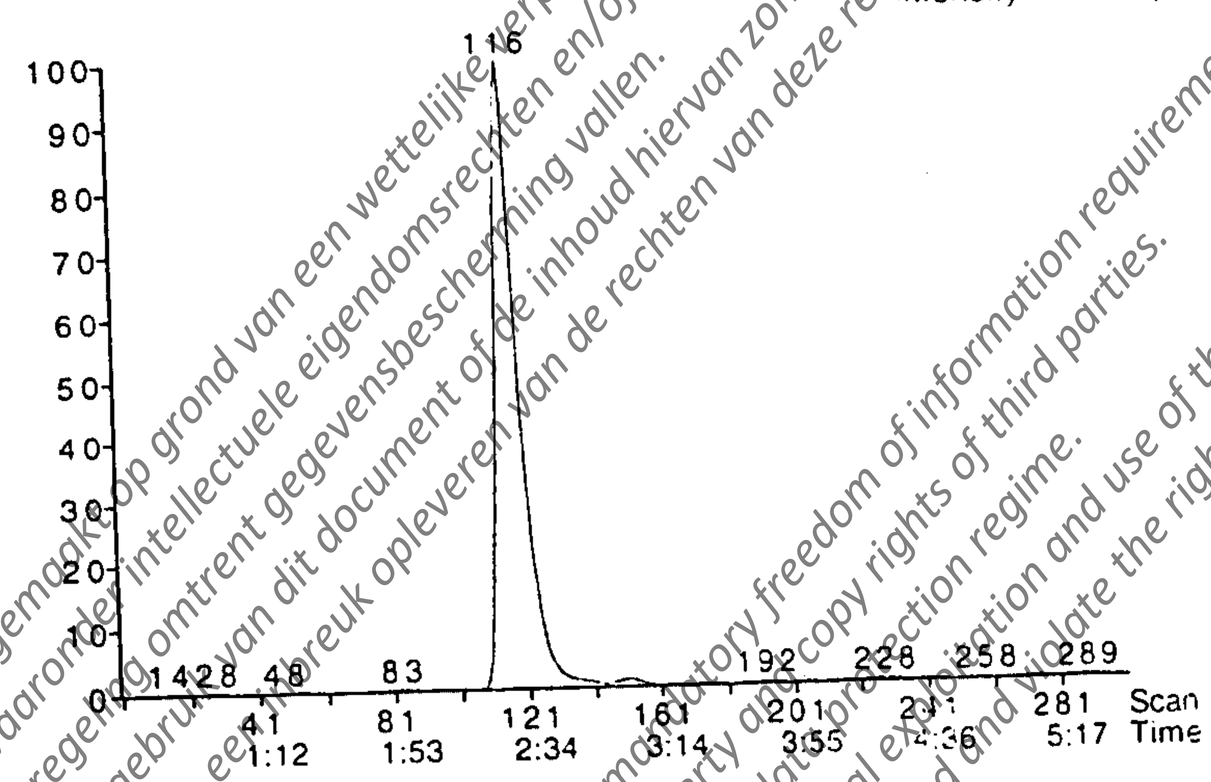
MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:21
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102
Comments: Imidacloprid (Field Soil Set#1)

NB0111C005 **IMIDACLOPRID** Fri, Jan 11, 2002 15:04
L1100-16+9 10mL

5:36 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM. 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	27985
Height	3410
Start Time	2:20
End Time	2:57
Integration Width	0:36.7
Retention Time	2:28
Integration Type	A - BV

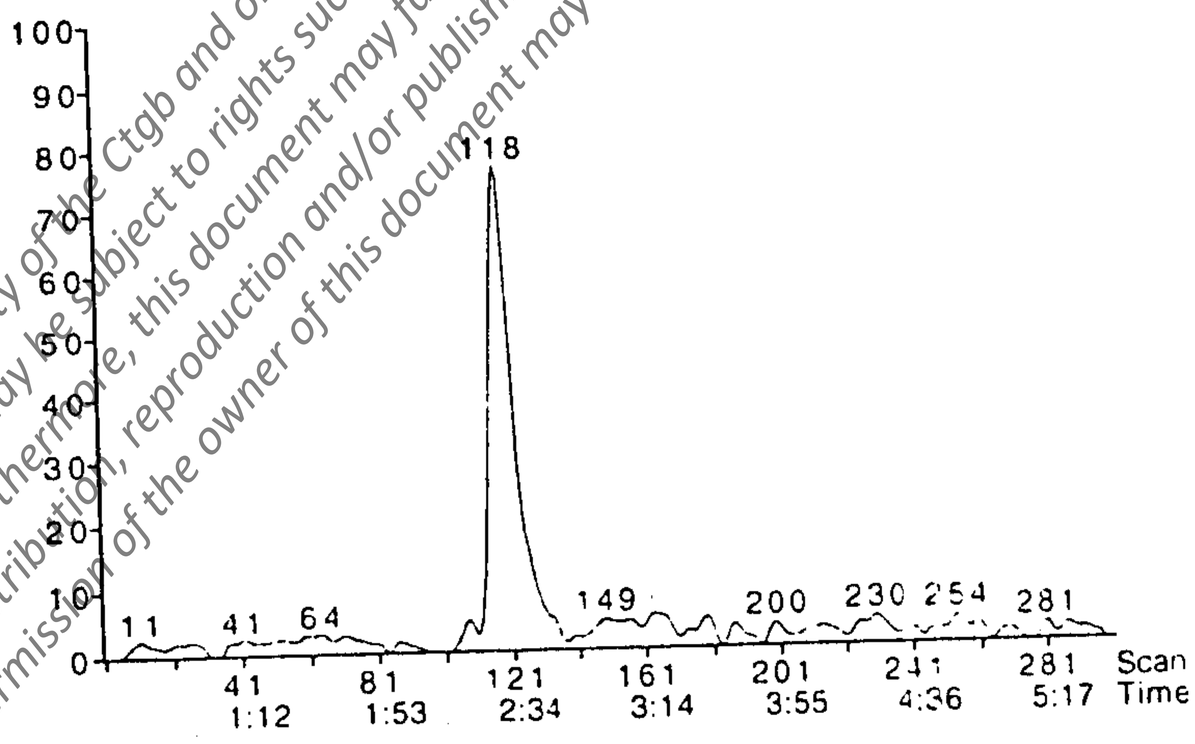


NB0111C006 **IMIDACLOPRID** Fri, Jan 11, 2002 15:11
Imidacloprid 5 ppb

5:36 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM. 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	1583
Height	188
Start Time	2:24
End Time	2:48
Integration Width	0:24.5
Retention Time	2:31
Integration Type	A - VB



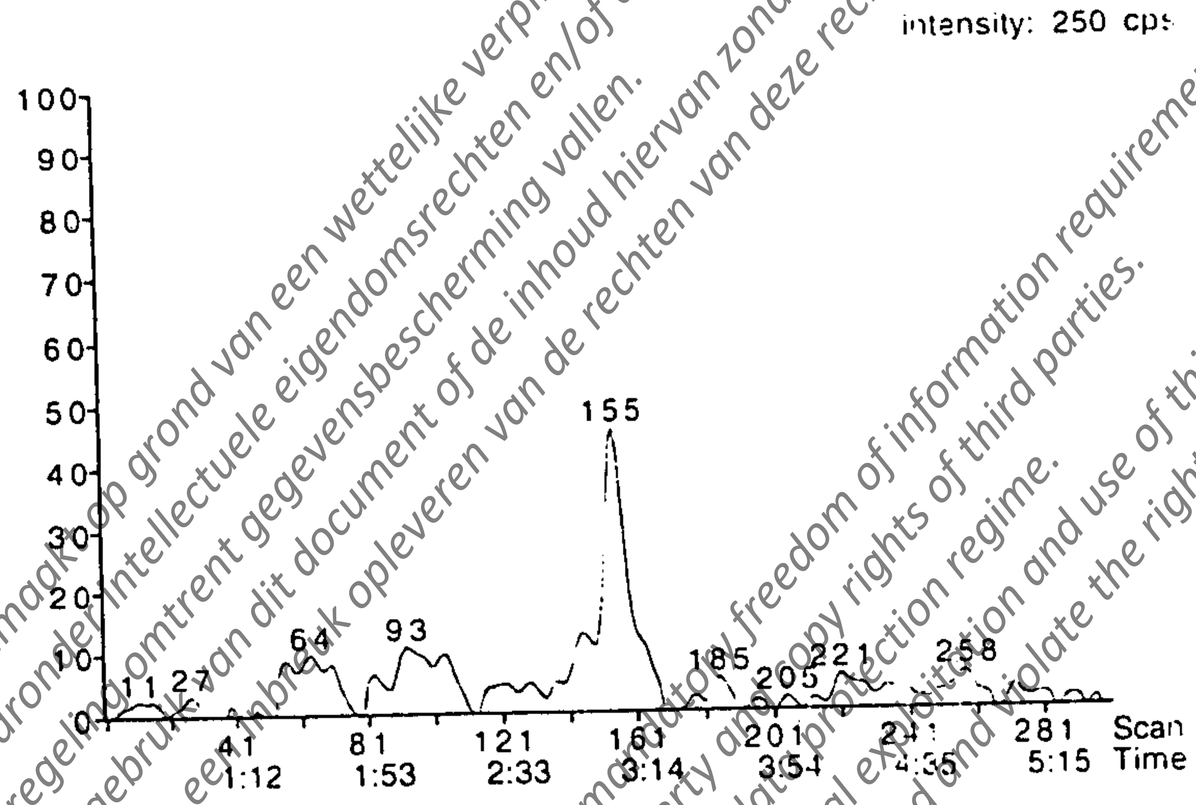
MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:21
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102
Comments: Imidacloprid (Field Soil Set#1)

NB0111C007 **IMIDACLOPRID** Fri, Jan 11, 2002 15:17
L1100-1 1mL

5:33 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:03 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0	
Quant Thres.	0.5	
Min. Width	5	
Mult. Width	4	
Base. Width	50	
RT Win. (secs)	20	
Smooth	5	
Expected RT	2:31	
Area	76	
Height	9	
Start Time		2:26
End Time		2:37
Integration Width		0:11.1
Retention Time		2:29
Integration Type		A - BV

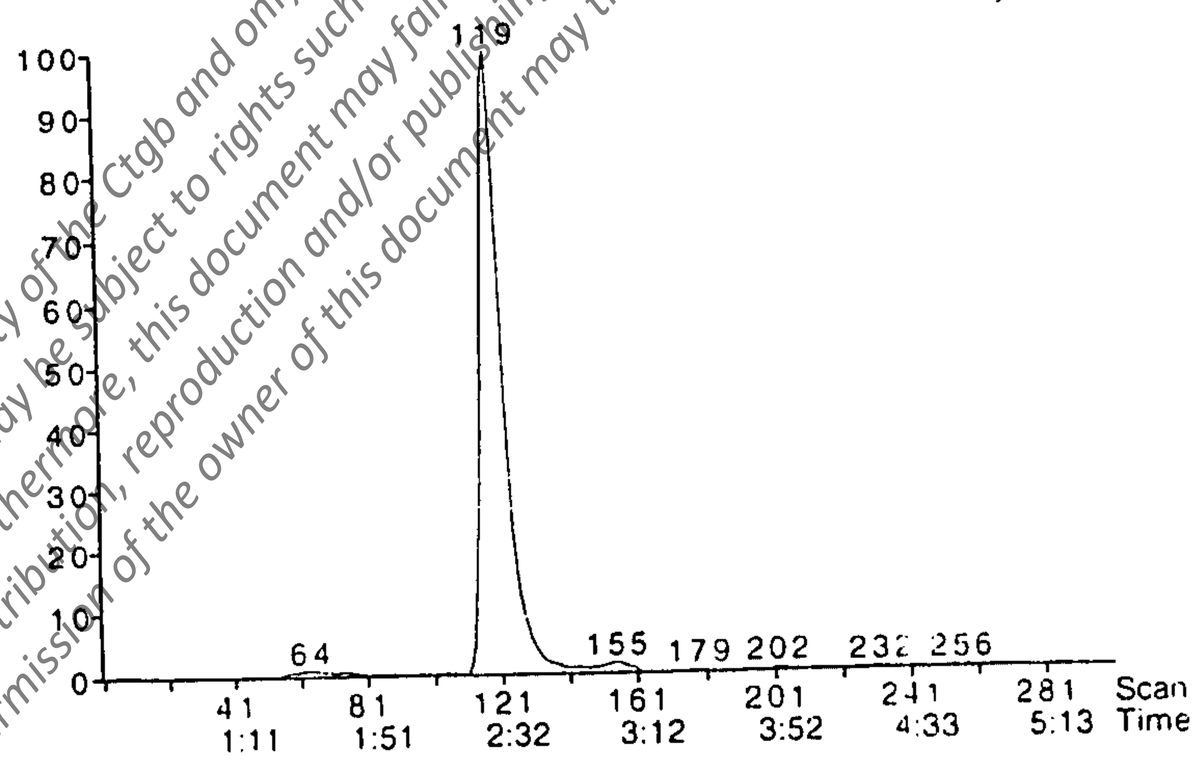


NB0111C008 **IMIDACLOPRID** Fri, Jan 11, 2002 15:24
L1100-5 10mL

5:32 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:01 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0	
Quant Thres.	0.5	
Min. Width	5	
Mult. Width	4	
Base. Width	50	
RT Win. (secs)	20	
Smooth	5	
Expected RT	2:31	
Area	37205	
Height	4749	
Start Time		2:22
End Time		2:56
Integration Width		0:34.2
Retention Time		2:30
Integration Type		A - BV



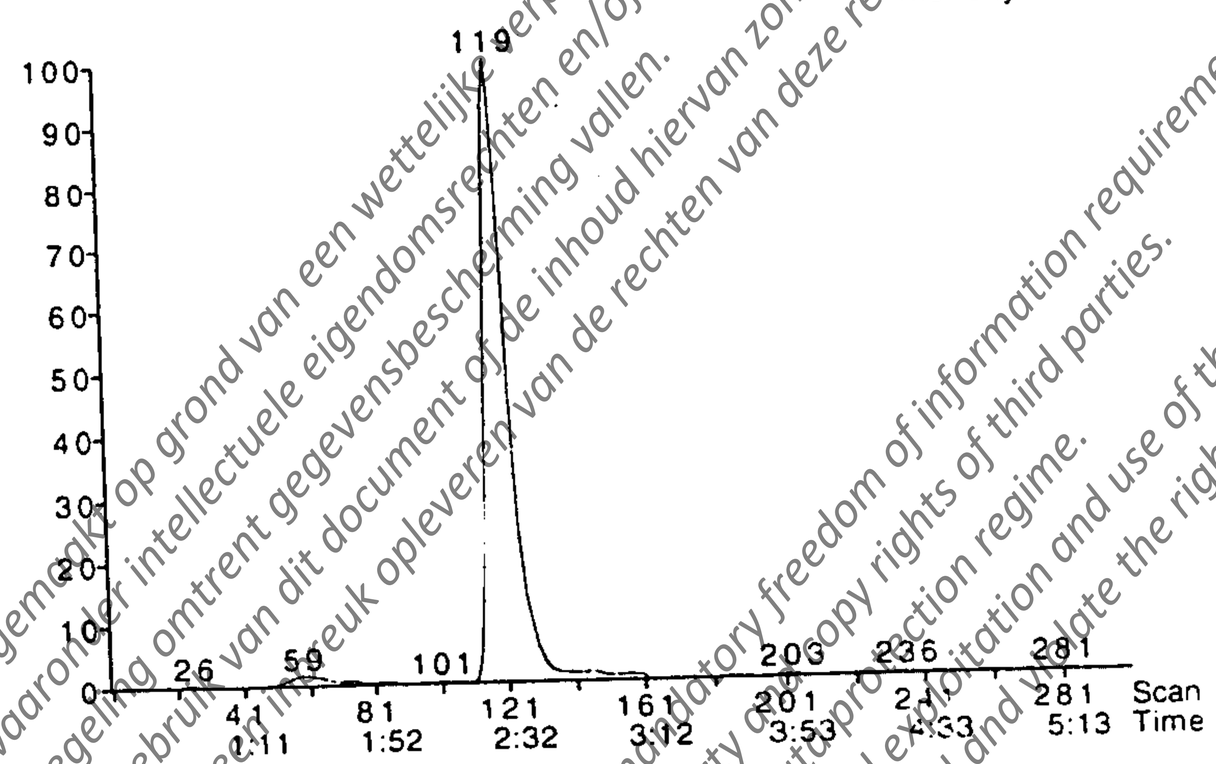
MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:21
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102
Comments: Imidacloprid (Field Soil Set#1)

NB0111C009 **IMIDACLOPRID** Fri, Jan 11, 2002 15:30
L1100-6 10mL

5:33 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:01 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	29257
Height	3741
Start Time	2:22
End Time	2:52
Integration Width	0:30.2
Retention Time	2:30
Integration Type	A - BV

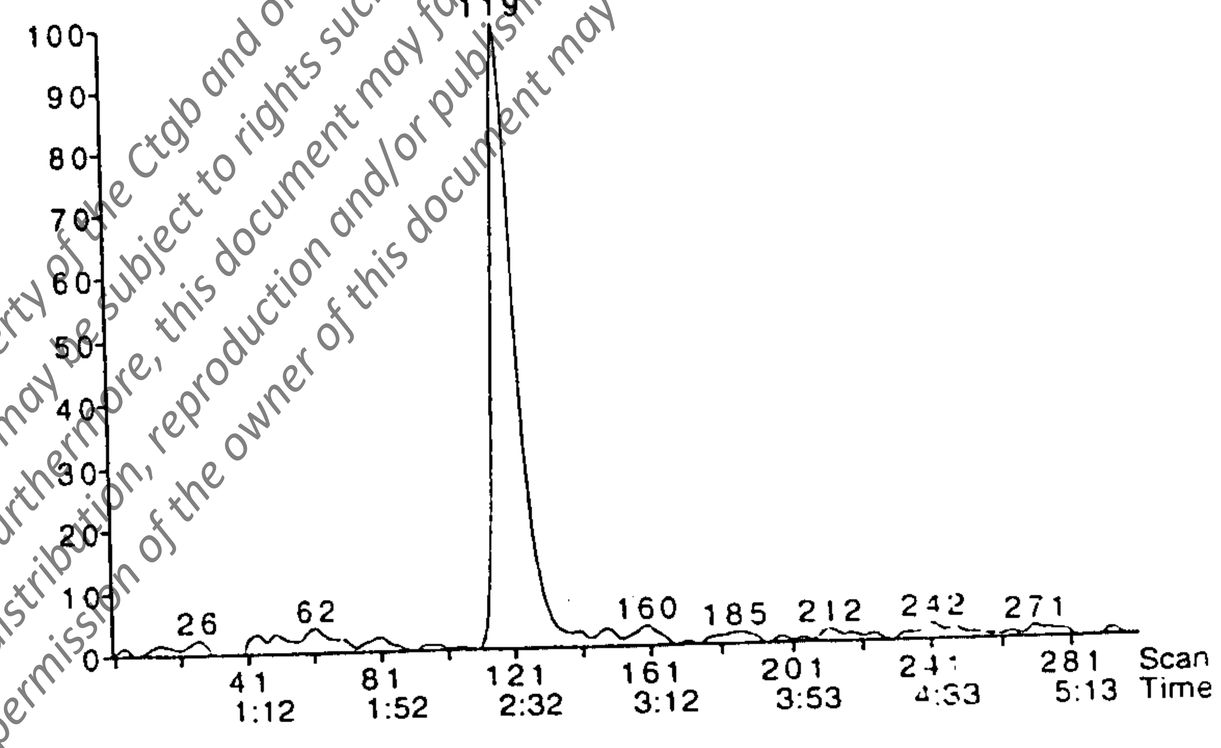


NB0111C010 **IMIDACLOPRID** Fri, Jan 11, 2002 15:37
Imidacloprid 10 ppb

5:32 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:01 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	3576
Height	437
Start Time	2:22
End Time	2:53
Integration Width	0:31.2
Retention Time	2:30
Integration Type	A - BV



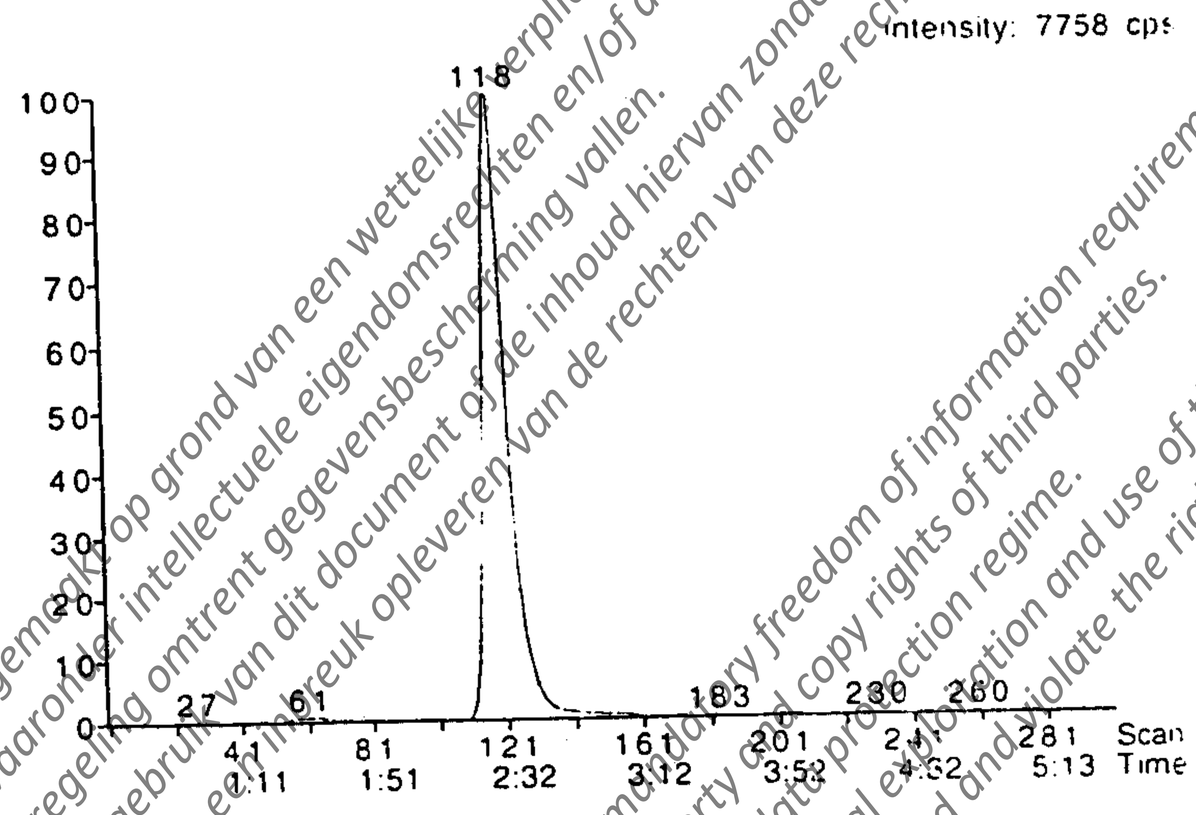
MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:21
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102:
Comments: Imidacloprid (Field Soil Set#1)

NB0111C011 IMIDACLOPRID Fri, Jan 11, 2002 15:43
L1100-7 10mL

5:32 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:01 MRM, 300 scans
256.5->175.0

Noise Thres.	7.0
Quant Thres.	7.0
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area 61282	
Height 7699	
Start Time	2:22
End Time	2:53
Integration Width	0:31.2
Retention Time	2:29
Integration Type	A - BB

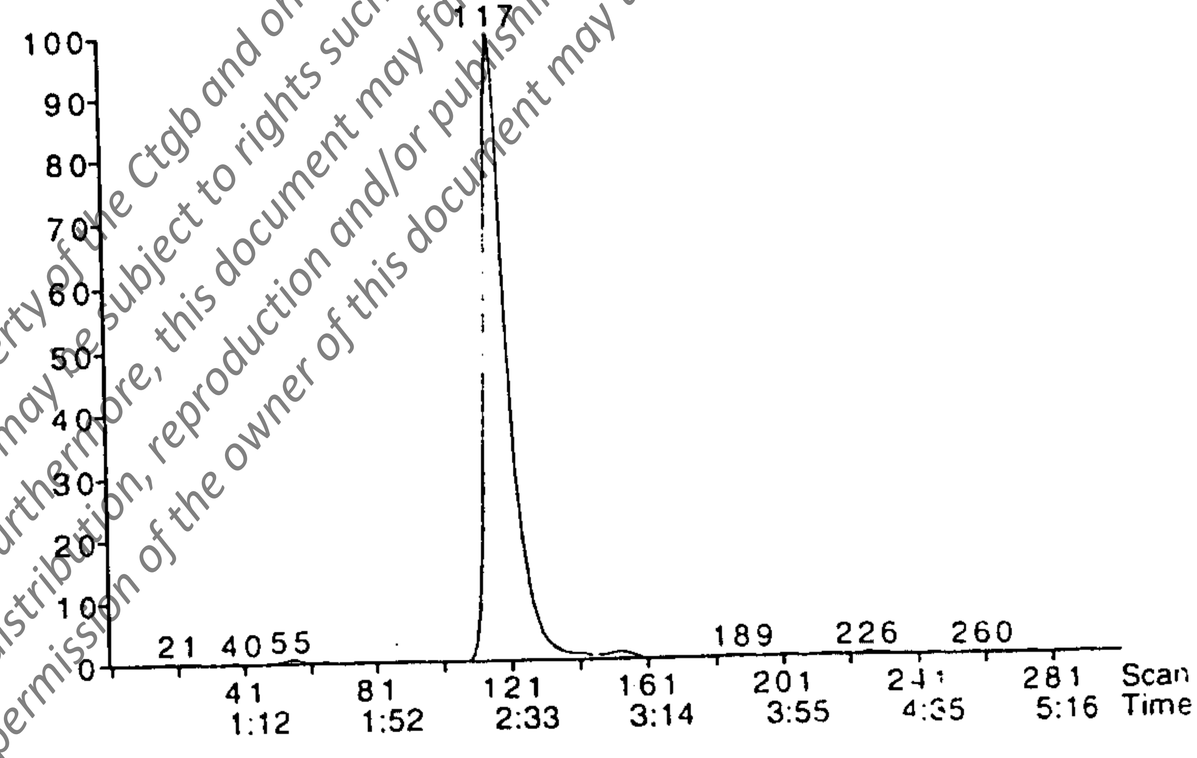


NB0111C012 IMIDACLOPRID Fri, Jan 11, 2002 15:50
L1100-8 10mL

5:36 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area 30445	
Height 3750	
Start Time	2:21
End Time	2:58
Integration Width	0:36.7
Retention Time	2:29
Integration Type	A - BV



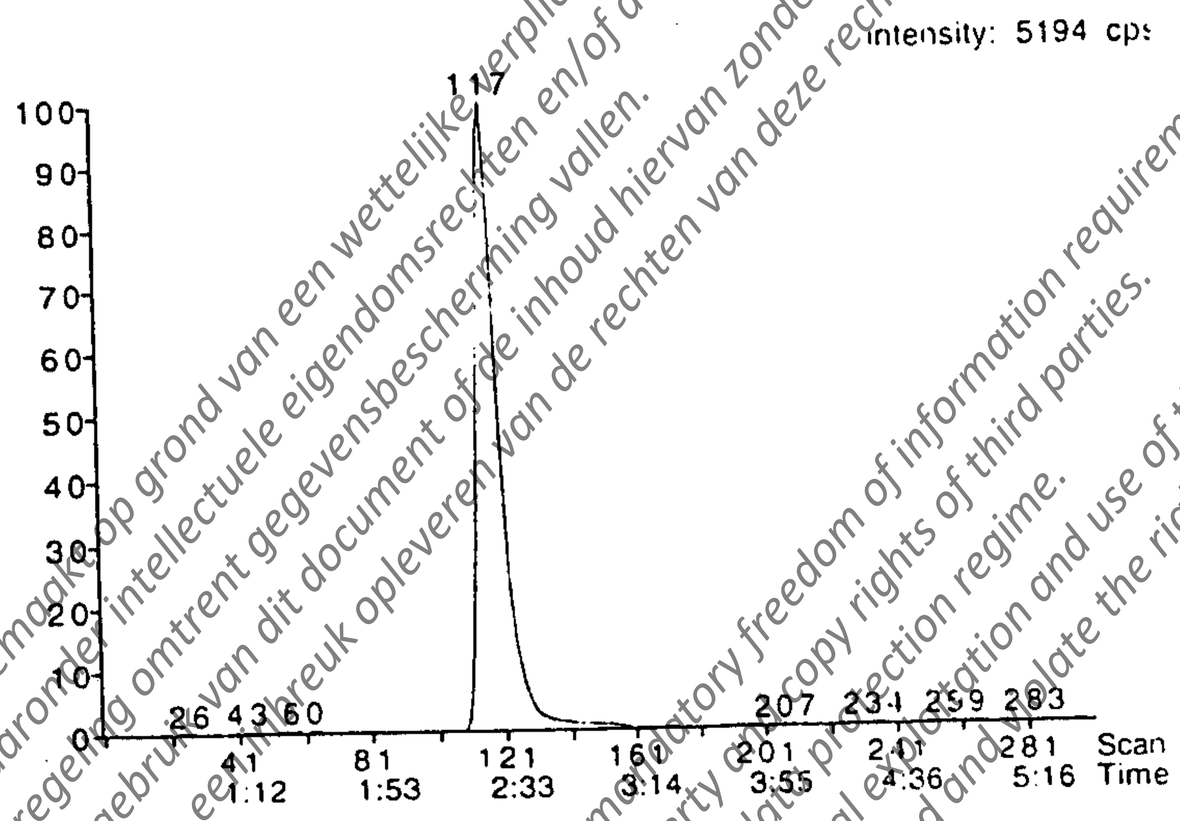
MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:21
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102
Comments: Imidacloprid (Field Soil Set#1)

NB0111C013 **IMIDACLOPRID** Fri, Jan 11, 2002 15:56
L1100-9 10mL

5:35 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	42050
Height	5194
Start Time	2:21
End Time	3:12
Integration Width	0:50.9
Retention Time	2:29
Integration Type	A - BB

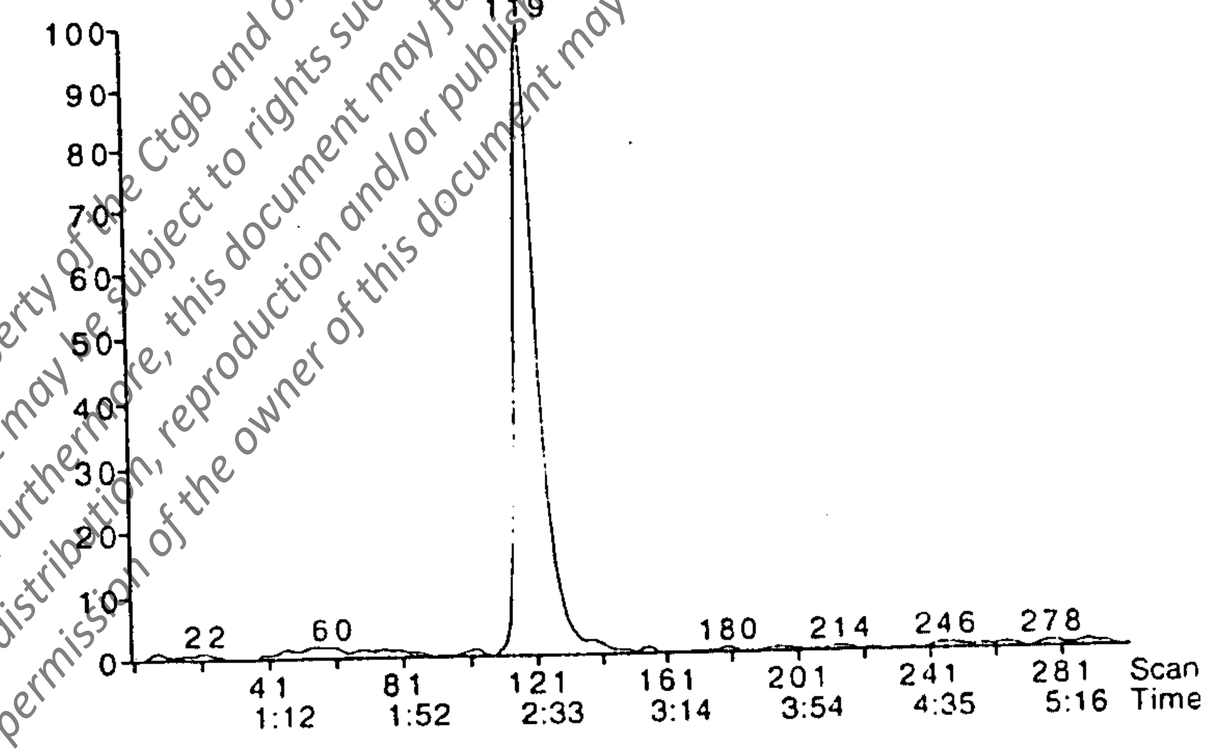


NB0111C014 **IMIDACLOPRID** Fri, Jan 11, 2002 16:03
Imidacloprid 20 ppb

5:35 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:04 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	7917
Height	988
Start Time	2:21
End Time	3:01
Integration Width	0:40.3
Retention Time	2:31
Integration Type	A - BB



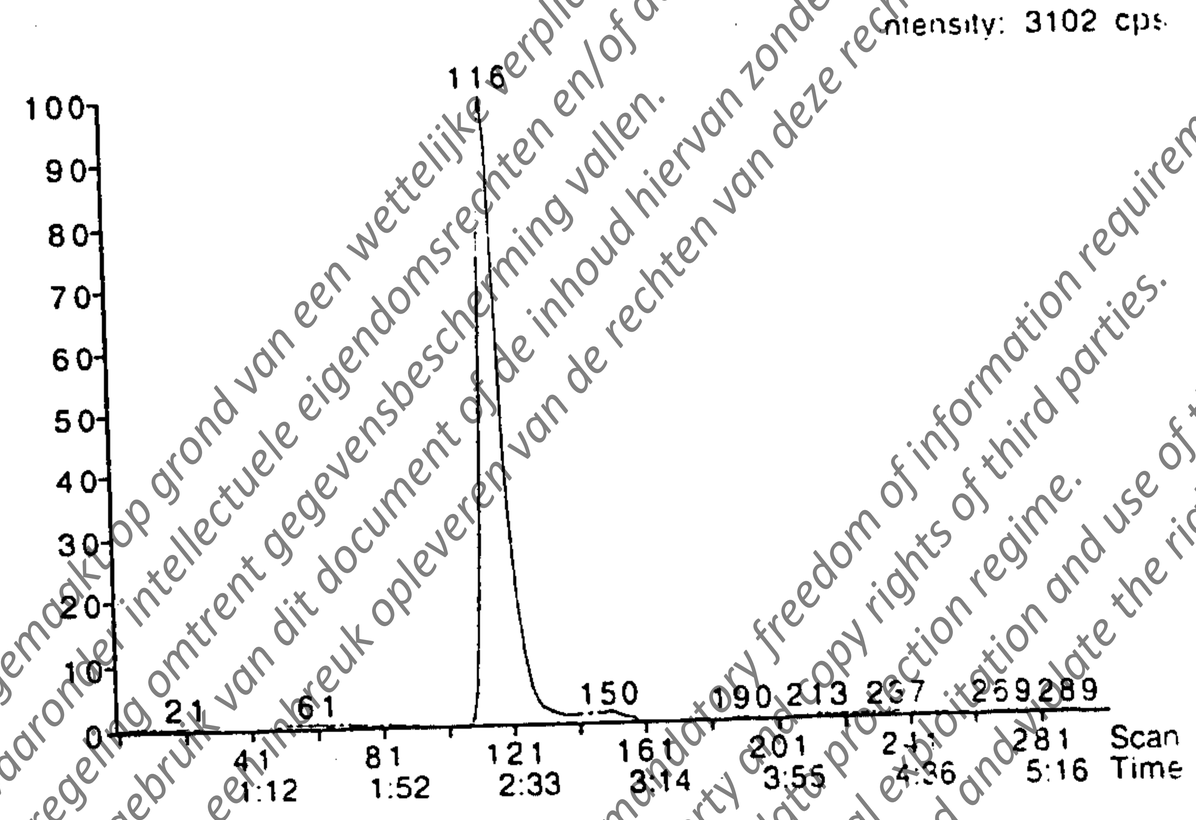
MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:21
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102
Comments: Imidacloprid (Field Soil Set#1)

NB0111C015 **IMIDACLOPRID** Fri, Jan 11, 2002 16:09
L1100-10 10mL

5:36 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	24510
Height	3100
Start Time	2:20
End Time	2:50
Integration Width	0:29.5
Retention Time	2:28
Integration Type	A - BV

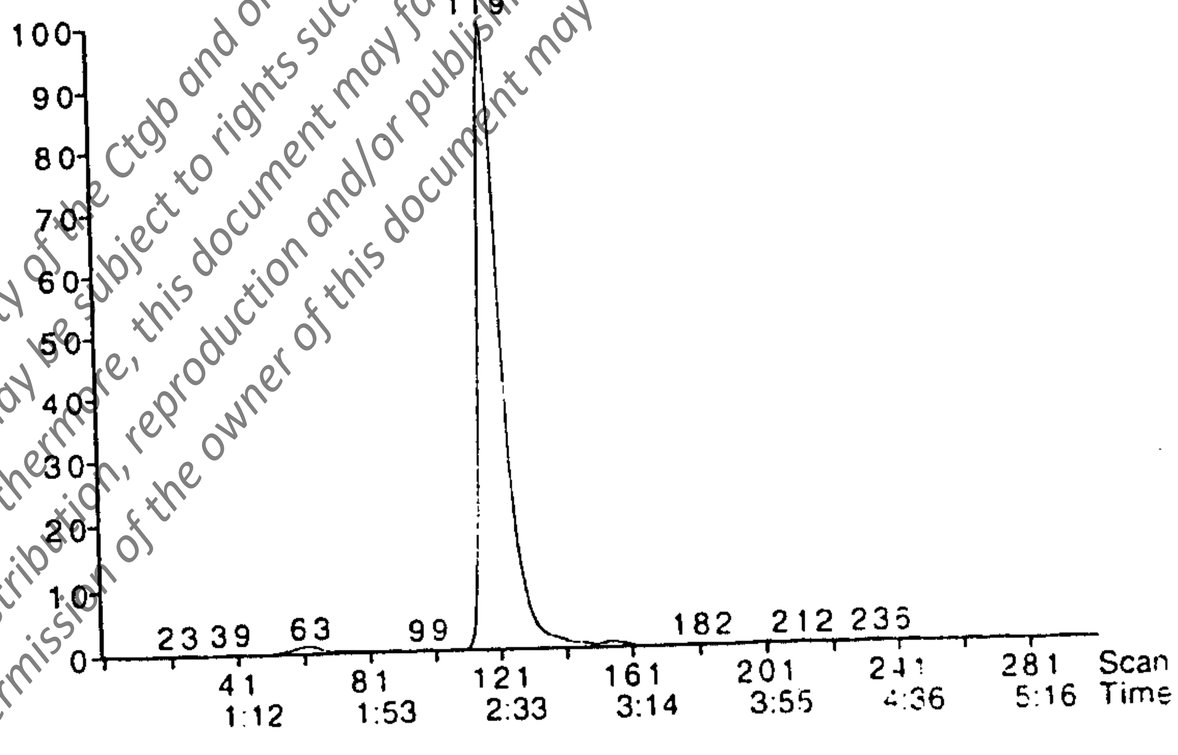


NB0111C016 **IMIDACLOPRID** Fri, Jan 11, 2002 16:16
L1100-11 10mL

5:35 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	44989
Height	5592
Start Time	2:23
End Time	3:02
Integration Width	0:38.8
Retention Time	2:31
Integration Type	A - BV



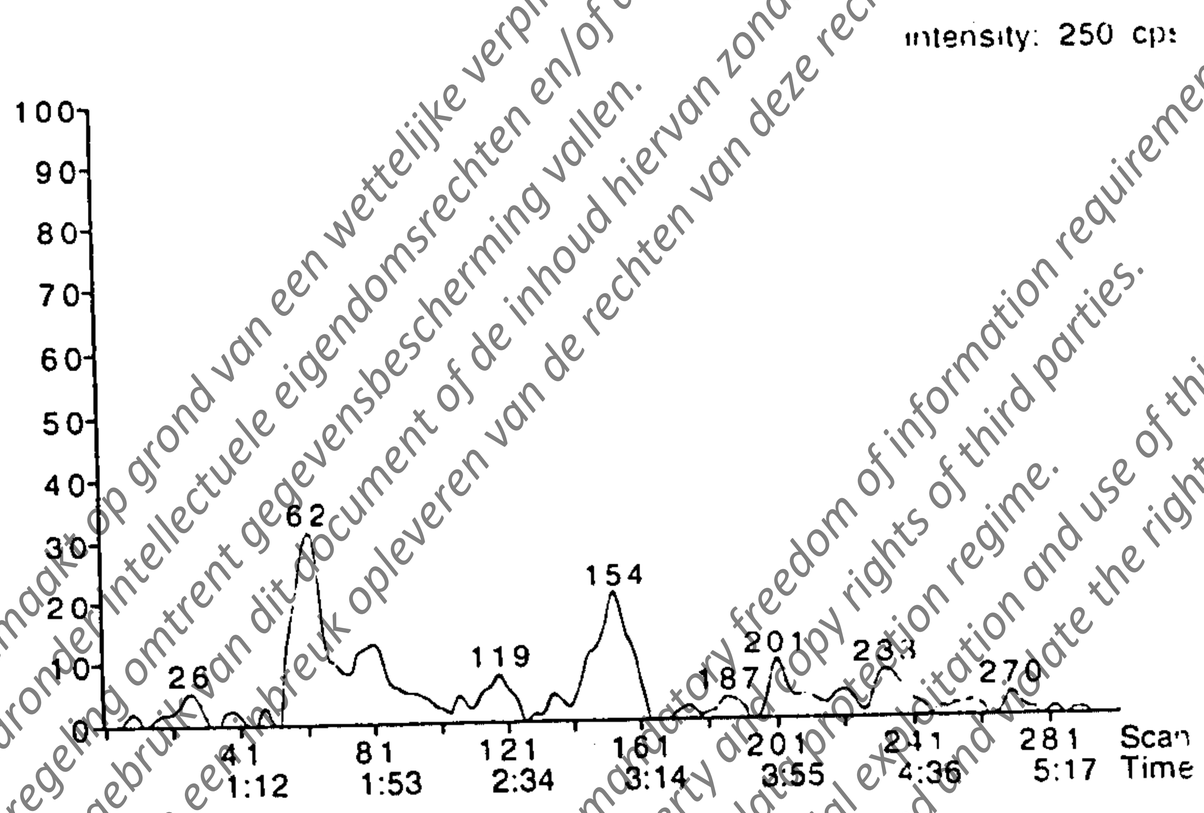
MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:21
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102
Comments: Imidacloprid (Field Soil Set#1)

NB0111C017 **IMIDACLOPRID** Fri, Jan 11, 2002 16:22
L1100-12 1mL

5:37 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM. 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area 108	
Height 14	
Start Time	2:22
End Time	2:37
Integration Width	0:14.1
Retention Time	2:32
Integration Type	A - VB

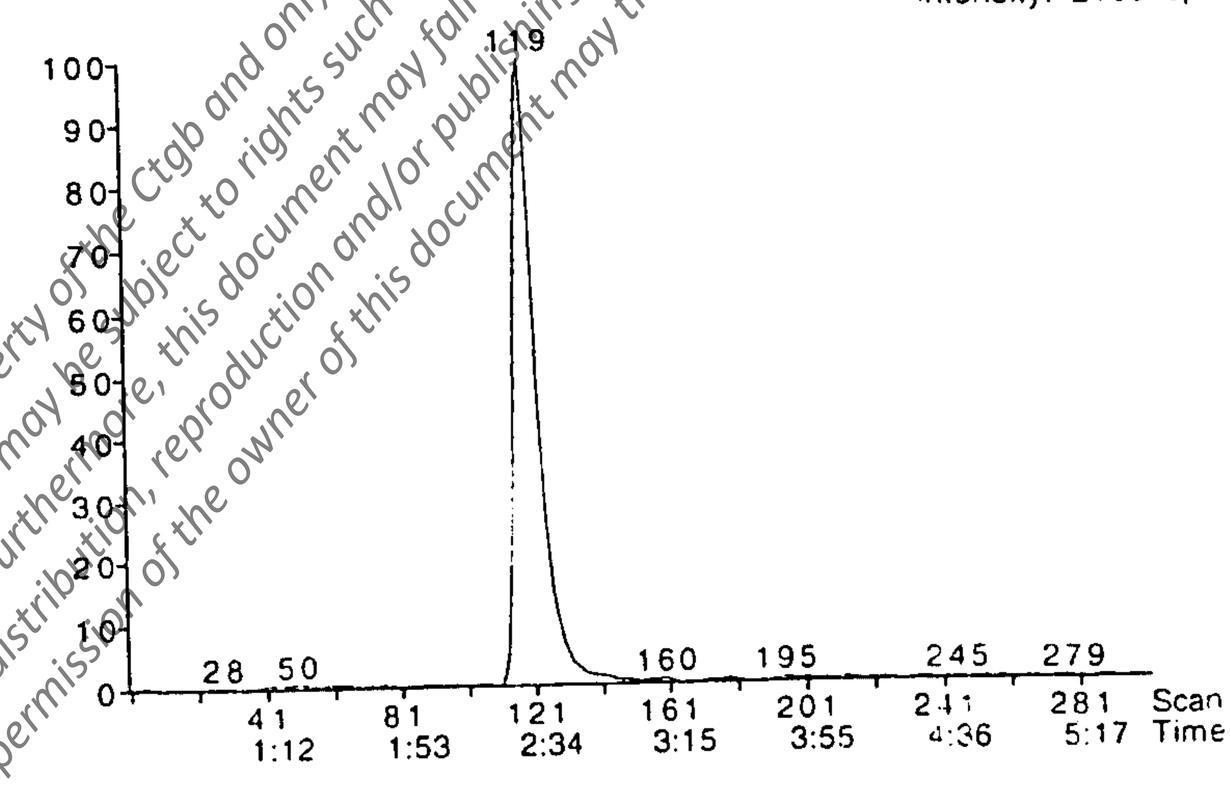


NB0111C018 **IMIDACLOPRID** Fri, Jan 11, 2002 16:29
Imidacloprid 40 ppb

5:37 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM. 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area 17671	
Height 2184	
Start Time	2:24
End Time	3:05
Integration Width	0:41.9
Retention Time	2:32
Integration Type	A - BV



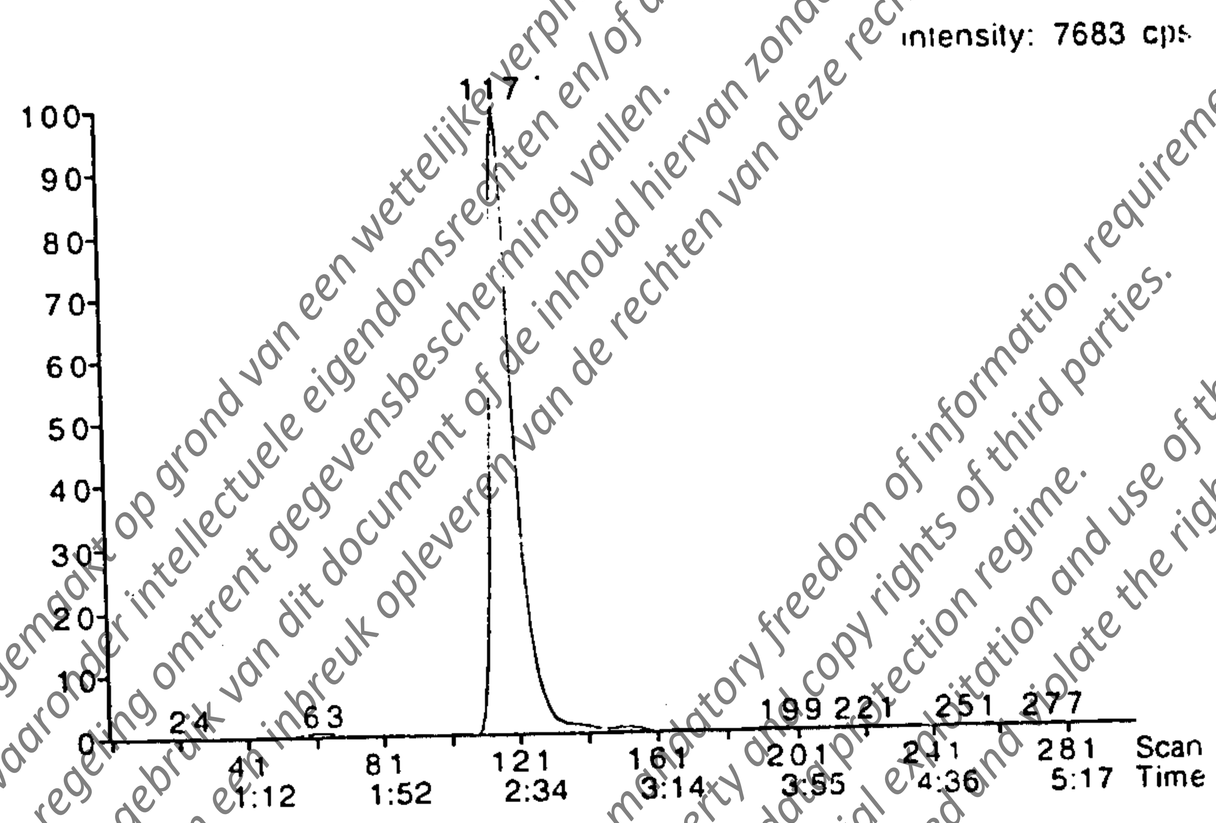
MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:21
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102:
Comments: Imidacloprid (Field Soil Set#1)

NB0111C019 **IMIDACLOPRID** Fri, Jan 11, 2002 16:35
L1100-13 10mL

5:36 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM, 300 scans
256.5->175.0

Noise Thres.	7.0
Quant Thres.	7.0
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	61225
Height	7669
Start Time	2:21
End Time	2:55
Integration Width	0:33.6
Retention Time	2:29
Integration Type	A - BB

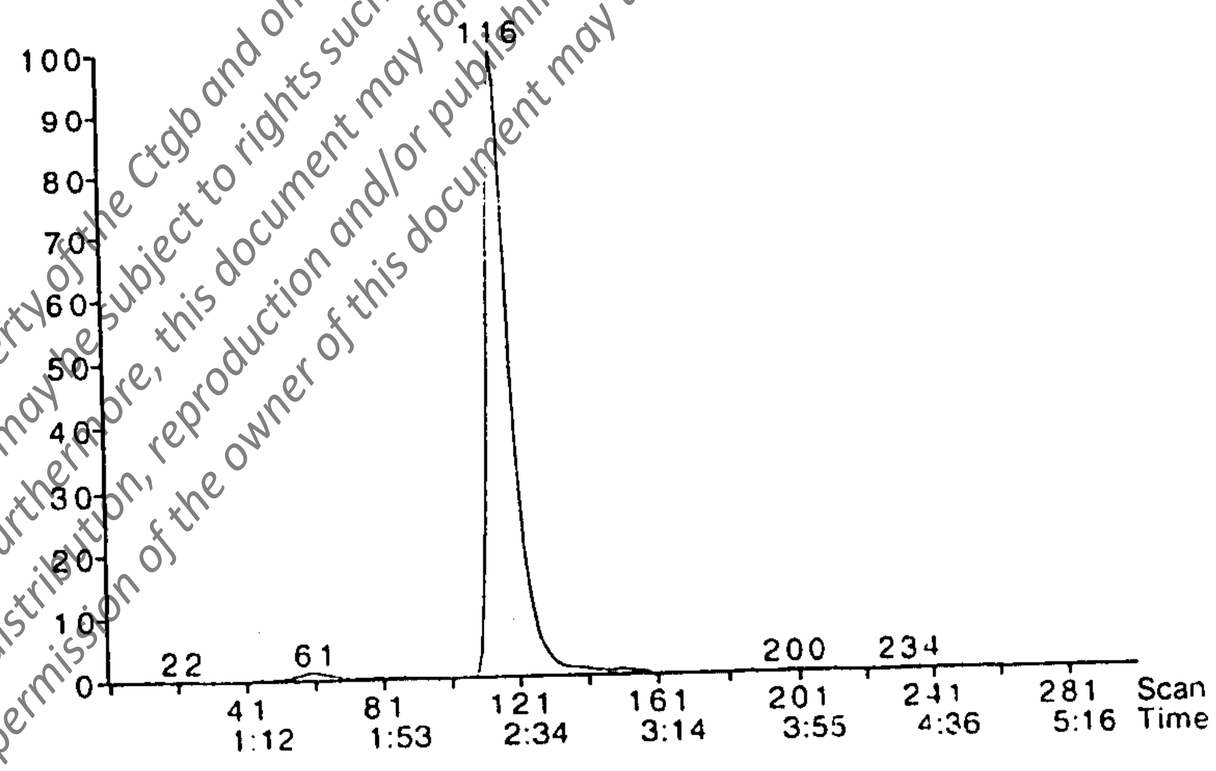


NB0111C020 **IMIDACLOPRID** Fri, Jan 11, 2002 16:42
L1100-14 10mL

5:36 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM, 300 scans
256.5->175.0

Noise Thres.	10
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	44518
Height	5512
Start Time	2:20
End Time	2:60
Integration Width	0:39.6
Retention Time	2:28
Integration Type	A - BV



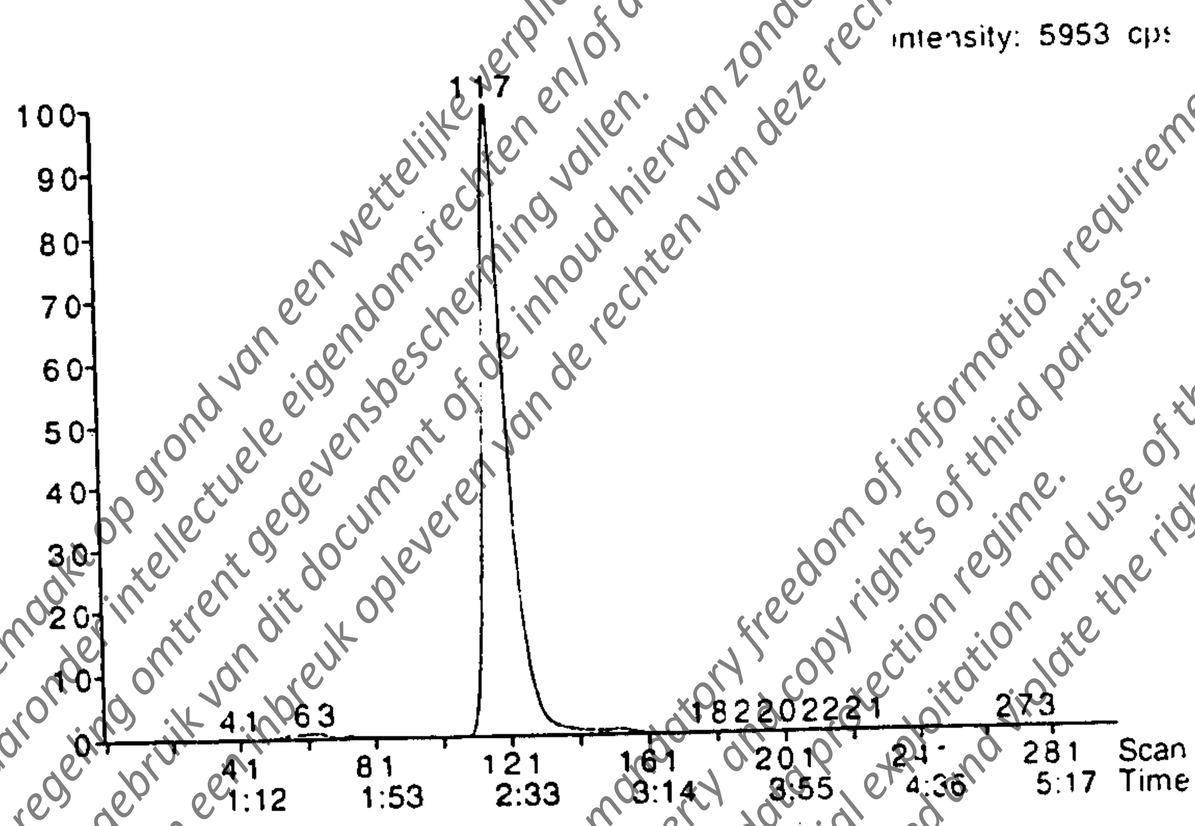
MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:21
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102
Comments: Imidacloprid (Field Soil Set#1)

NB0111C021 IMIDACLOPRID Fri, Jan 11, 2002 16:48
L1100-15 10mL

5:36 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:06 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	49368
Height	5952
Start Time	2:21
End Time	2:60
Integration Width	0:39.0
Retention Time	2:29
Integration Type	A - BV

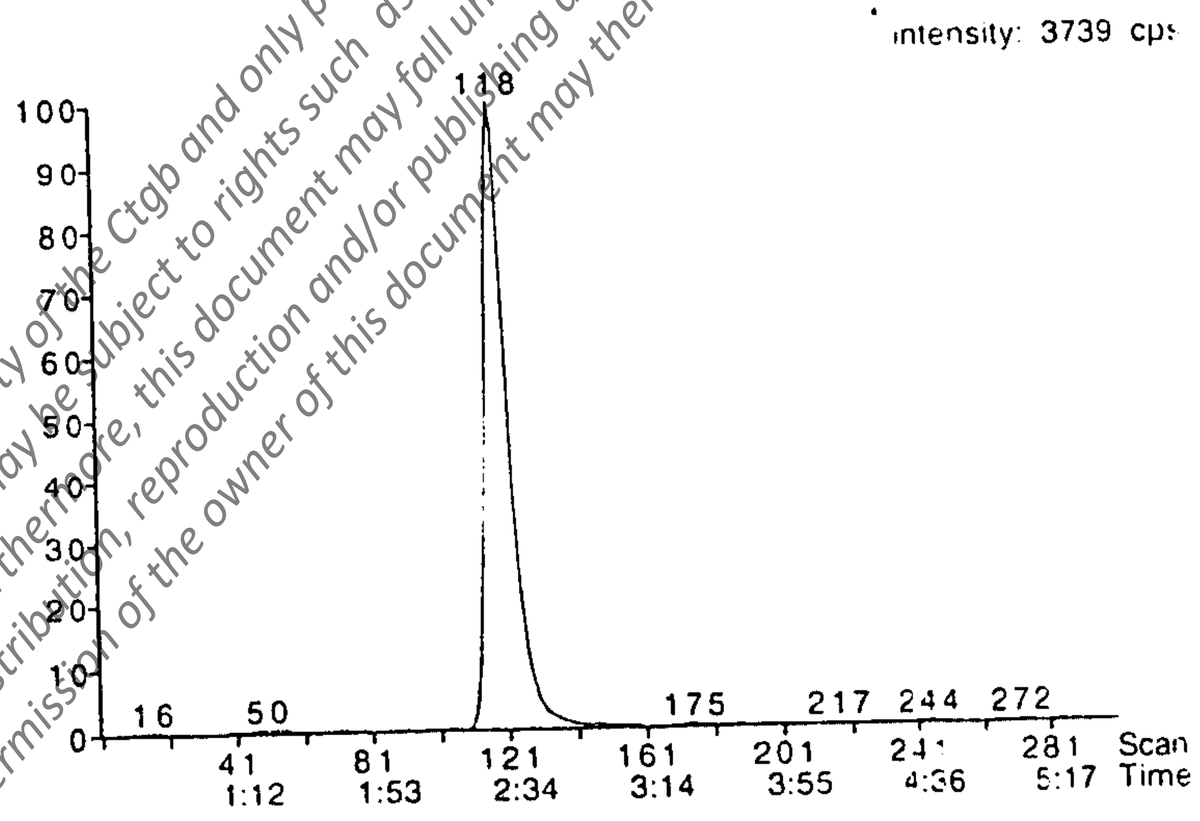


NB0111C022 IMIDACLOPRID Fri, Jan 11, 2002 16:55
Imidacloprid 75 ppb

5:37 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	30310
Height	3739
Start Time	2:21
End Time	3:12
Integration Width	0:51.0
Retention Time	2:30
Integration Type	A - BB



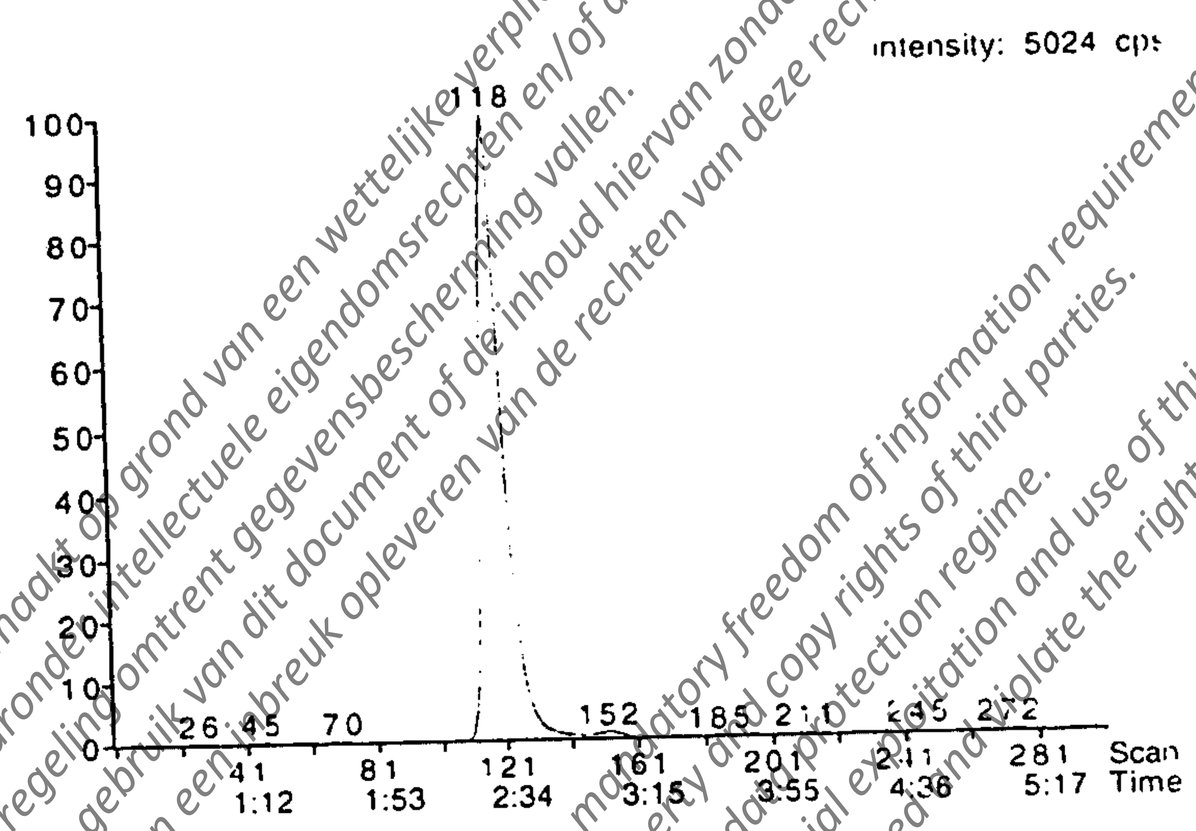
MacQuan, version 1.6
Printed: Sat, Jan 12, 2002 13:21
Calibration File: 011102Ccal Path: LaCie 8800:DATA La Cie 1250:BAYER:Imidacloprid:011102
Comments: Imidacloprid (Field Soil Set#1)

NB0111C023 **IMIDACLOPRID** Fri, Jan 11, 2002 17:01
Imidacloprid 100 ppb

5:36 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:05 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	,5
Expected RT	2:31
Area	40535
Height	5023
Start Time	2:22
End Time	2:58
Integration Width	0:36.7
Retention Time	2:31
Integration Type	A - BV

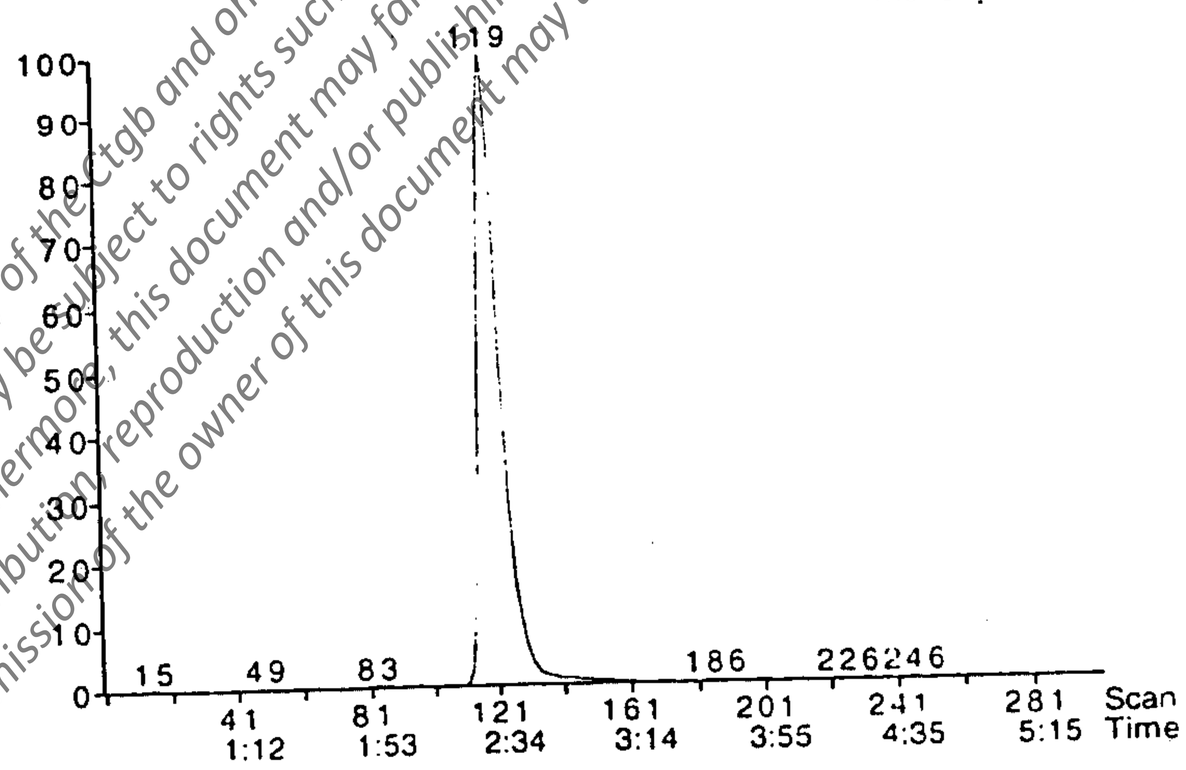


NB0111C024 **IMIDACLOPRID** Fri, Jan 11, 2002 17:08
Imidacloprid 150 ppb

5:34 in 1 period
Imidacloprid
No Internal Standard
Use Area

1: 5:03 MRM, 300 scans
256.5->175.0

Noise Thres.	1.0
Quant Thres.	0.5
Min. Width	5
Mult. Width	4
Base. Width	50
RT Win. (secs)	20
Smooth	5
Expected RT	2:31
Area	55353
Height	6738
Start Time	2:23
End Time	3:14
Integration Width	0:50.9
Retention Time	2:32
Integration Type	A - BB



NB, JAN. 12/2002.

Appendix C

Soil Analysis

Report

Soil Analysis Report

[redacted]

Soil & Feed Testing Laboratory
PEI Department of Agriculture & Forestry
440 University Avenue
PO Box 1600, Charlottetown, PEI
C1A 7N3
Fax: (902) 368-6299
Telephone: (902) 368-5628

[redacted]

Prince Edward Island CANADA

Client:	2875
Accession:	9073
Samples Reported:	08/22/2001
Samples Received:	08/20/2001

Sample Information		Soil Test Values and Ratings							
Lab Sample #	Field Number	Organic Matter (%)	pH	Phosphate P_2O_5 (ppm)	Potash K_2O (ppm)	Calcium Ca (ppm)	Magnesium Mg (ppm)	Boron B (ppm)	Copper Cu (ppm)
1	01	2.5	5.8	587 H+	259 H+	929 M	70 M	.4 L	1.4 M
2	02	2.6	6.3	247 M+	89 M	1026 M	112 M	.3 L	1.8 M+
3	03	3.3	5.9	304 H	123 H	859 M	75 M	.4 L	2.1 M+
4	11	2.4	5.8	623 H+	149 H	679 L	87 M	.2 L-	2.9 H
5	12	2.5	6.2	542 H+	205 H+	989 M	84 M	.3 L	2.9 H

Lab Sample #	Field Number	Zinc Zn (ppm)	Sulfur S (ppm)	Manganese Mn (ppm)	Iron Fe (ppm)	Sodium Na (ppm)	Aluminum Al (ppm)	Lime Index	Conduct-ivity mS/cm
1	01	2.9 L	30 H+	53 H+	182 H+	26		6.6	
2	02	1.2 L	24 H	113 H+	156 H+	29		6.9	
3	03	1.7 L	28 H+	37 M+	169 H+	35		6.5	
4	11	1.8 L	37 H+	43 H	181 H+	30		6.6	
5	12	8.1 H+	28 H+	35 M+	183 H+	30		6.6	

L-: Low L: Low M: Medium M+: Above Medium H: High H+: Very High

To convert HECTARES into ACRES multply by 2.47				To convert T/HECTARE into T/ACRE multiply by 0.45			To convert Kg/Ha into lbs/ACRE; multiply by 0.9		
Sample Information				Limestone application (T/Ha) to achieve			Required Applications (Kg/Ha)		
Lab Sample #	Field Number	Field Size (Ha)	Crop to be grown	pH 5.5	pH 6.0	pH 6.5	Nitrogen N	Phosphate P_2O_5	Potash K_2O
1	01		Unknown			3			
2	02		Unknown						
3	03		Unknown			3			
4	11		Unknown			3			
5	12		Unknown			3			

Lab Sample #	Field Number	Man	Sod	CEC (Meq/100g)	Base Saturation					Total % Base Saturation
					% K	% Mg	% Ca	% H	% Na	
1	01	0	0	11	5.2	5.5	43.4	44.9	1.1	54.1
2	02	0	0	8	2.5	12.3	67.7	15.8	1.7	82.5
3	03	0	0	11	2.3	5.5	37.9	52.9	1.3	45.7
4	11	0	0	9	3.4	7.7	36.2	51.2	1.4	47.3
5	12	0	0	11	4.0	6.4	44.9	43.6	1.2	55.3

Comments:

Methods: Water pH (1:1, soil:water)
Lime Index: SMP Buffer Method
Nutrient Extraction: Melich III
% Organic Matter: Combustion Furnace

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Analysis Approved By: [redacted]

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Soil Analysis Report

[redacted]

Soil & Feed Testing Laboratory
PEI Department of Agriculture
& Forestry
440 University Avenue
PO Box 1600, Charlottetown, PEI
C1A 7N3
Fax: (902) 368-6299
Telephone: (902) 368-5628

Client: 2875
Accession: 9073
Samples Reported: 08/22/2001
Samples Received: 08/20/2001

Sample Information		Soil Test Values and Ratings							
Lab Sample #	Field Number	Organic Matter (%)	pH	Phosphate P_2O_5 (ppm)	Potash K_2O (ppm)	Calcium Ca (ppm)	Magnesium Mg (ppm)	Boron B (ppm)	Copper Cu (ppm)
6	13	2.8	5.5	674 H+	239 H+	779 L	97 M	.3 L	1.8 M+
7	14	2.4	6.0	456 H+	296 H+	828 L	81 M	.3 L	1.5 M+
8	15	3.2	6.2	319 H	56 M	972 M	77 M	.2 L-	1.5 M+
9	21	2.8	6.5	270 H	73 M	1095 M	112 M	.4 L	1.1 M
10	22	2.3	5.8	714 H+	227 H+	551 L	107 M	.2 L-	1.5 M+

Lab Sample #	Field Number	Zinc Zn (ppm)	Sulfur S (ppm)	Manganese Mn (ppm)	Iron Fe (ppm)	Sodium Na (ppm)	Aluminum Al (ppm)	Lime Index	Conduct-ivity mS/cm
6	13	2.4 L	47 H+	52 H+	256 H+	29		6.5	
7	14	1.6 L	29 H+	36 M+	158 H+	29		6.7	
8	15	1.1 L	28 H+	18 M	143 H+	35		6.8	
9	21	1.2 L	24 H	66 H+	272 H+	29		6.7	
10	22	2.4 L	33 H+	55 H+	208 H+	27		6.4	

L-: Low L: Low M: Medium M+: Above Medium H: High H+: Very High

To convert HECTARES into ACRES multply by 2.47 | **To convert T/HECTARE into T/ACRE multiply by 0.45** | **To convert Kg/Ha into lbs/ACRE; multiply by 0.9**

Sample Information				Limestone application (T/Ha) to achieve			Required Applications (Kg/Ha)		
Lab Sample #	Field Number	Field Size (Ha)	Crop to be grown	pH 5.5	pH 6.0	pH 6.5	Nitrogen N	Phosphate P_2O_5	Potash K_2O
6	13		Unknown		2	3			
7	14		Unknown			2			
8	15		Unknown			2			
9	21		Unknown						
10	22		Unknown			4			

Lab Sample #	Field Number	Man	Sod	CEC (Meq/100g)	Base Saturation % K	% Mg	% Ca	% H	% Na	Total % Base Saturation
6	13	0	0	11	4.5	7.1	34.3	52.9	1.1	45.9
7	14	0	0	9	6.9	7.4	45.1	39.2	1.4	59.4
8	15	0	0	8	1.5	7.9	59.5	29.4	1.9	68.9
9	21	0	0	9	1.8	10.9	63.9	21.9	1.5	76.6
10	22	0	0	11	4.2	7.8	24.1	62.9	1.0	36.1

Comments:

Methods: Water pH (1:1, soil:water)
Lime Index: SMP Buffer Method
Nutrient Extraction: Melich III
% Organic Matter: Combustion Furnace

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Analysis Approved By: [redacted]

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Soil Analysis Report

[redacted]

Soil & Feed Testing Laboratory
PEI Department of Agriculture & Forestry
440 University Avenue
PO Box 1600, Charlottetown, PEI
C1A 7N3
Fax: (902) 368-6299
Telephone: (902) 368-5628

Edward Island CANADA

Client: 2875
Accession: 9073
Samples Reported: 08/22/2001
Samples Received: 08/20/2001

Sample Information		**Soil Test Values and Ratings**							
Lab Sample #	**Field Number**	**Organic Matter (%)**	**pH**	**Phosphate P_2O_5 (ppm)**	**Potash K_2O (ppm)**	**Calcium Ca (ppm)**	**Magnesium Mg (ppm)**	**Boron B (ppm)**	**Copper Cu (ppm)**
11	23	3.4	5.8	177 M	75 M	619 L	82 M	.2 L-	.3 L
12	31	3.0	5.9	141 L	91 M+	778 L	114 M	.3 L	.6 L
13	32	2.0	5.2	206 M	98 M+	673 L	134 M	.2 L-	.6 L
14	33	2.4	5.7	257 M+	119 H	659 L	95 M	.2 L-	1.0 M
15	34	2.3	5.6	262 M+	105 M+	781 L	104 M	.3 L	.4 L

Lab Sample #	**Field Number**	**Zinc Zn (ppm)**	**Sulfur S (ppm)**	**Manganese Mn (ppm)**	**Iron Fe (ppm)**	**Sodium Na (ppm)**	**Aluminum Al (ppm)**	**Lime Index**	**Conduct-ivity mS/cm**
11	23	1.1 L	27 H+	27 M	275 H+	19		6.4	
12	31	1.4 L	26 H+	21 M	286 H+	31		6.6	
13	32	1.3 L	25 H	47 H	262 H+	21		6.6	
14	33	1.0 L	28 H+	17 M	135 H+	26		6.6	
15	34	1.4 L	28 H+	20 M	572 H+	36		6.8	

L-: Low L: Low M: Medium M+: Above Medium H: High H+: Very High

To convert HECTARES into ACRES multply by 2.47				**To convert T/HECTARE into T/ACRE multiply by 0.45**			**To convert Kg/Ha into lbs/ACRE; multiply by 0.9**		
Sample Information				**Limestone application (T/Ha) to achieve**			**Required Applications (Kg/Ha)**		
Lab Sample #	**Field Number**	**Field Size (Ha)**	**Crop to be grown**	**pH 5.5**	**pH 6.0**	**pH 6.5**	**Nitrogen N**	**Phosphate P_2O_5**	**Potash K_2O**
11	23		Unknown			4			
12	31		Unknown			3			
13	32		Unknown	1	2	3			
14	33		Unknown		2	3			
15	34		Unknown		1	2			

Lab Sample #	**Field Number**	**Man**	**Sod**	**CEC (Meq/100g)**	**Base Saturation % K**	**% Mg**	**% Ca**	**% H**	**% Na**	**Total % Base Saturation**
11	23	0	0	11	1.4	6.1	27.6	64.2	.7	35.1
12	31	0	0	10	2.0	9.5	39.0	48.1	1.4	50.5
13	32	0	0	10	2.2	11.7	35.1	50.1	1.0	49.0
14	33	0	0	9	2.7	8.6	35.6	51.9	1.2	46.9
15	34	0	0	8	3.0	11.5	51.7	31.8	2.1	66.2

Comments:

Methods: Water pH (1:1, soil:water)
Lime Index: SMP Buffer Method
Nutrient Extraction: Melich III
% Organic Matter: Combustion Furnace

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Analysis Approved By: [redacted]

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Soil Analysis Report

[redacted]

Soil & Feed Testing Laboratory
PEI Department of Agriculture & Forestry
440 University Avenue
PO Box 1600, Charlottetown, PEI
C1A 7N3
Fax: (902) 368-6299
Telephone: (902) 368-5628

Prince Edward Island CANADA

Client: 2875
Accession: 9073
Samples Reported: 08/22/2001
Samples Received: 08/20/2001

Sample Information		Soil Test Values and Ratings							
Lab Sample #	Field Number	Organic Matter (%)	pH	Phosphate P_2O_5 (ppm)	Potash K_2O (ppm)	Calcium Ca (ppm)	Magnesium Mg (ppm)	Boron B (ppm)	Copper Cu (ppm)
16	35	2.5	6.0	468 H+	213 H+	991 M	111 M	.3 L	1.0 M
17	36	2.3	6.3	627 H+	160 H	1013 M	123 M	.3 L	1.4 M
18	37	2.2	6.4	188 M	96 M+	1110 M	137 M+	.2 L-	.5 L
19	CF/CL 16	5.8	6.8	98 L-	45 L	1346 M	200 H	.3 L	5.4 H+
20	CF/CL 17	4.1	5.8	665 H+	174 H	889 M	155 M+	.4 L	5.4 H+

Lab Sample #	Field Number	Zinc Zn (ppm)	Sulfur S (ppm)	Manganese Mn (ppm)	Iron Fe (ppm)	Sodium Na (ppm)	Aluminum Al (ppm)	Lime Index	Conductivity mS/cm
16	35	2.3 L	39 H+	44 H	171 H+	29		6.8	
17	36	1.2 L	53 H+	45 H	189 H+	28		6.9	
18	37	.8 L-	26 H+	37 M+	188 H+	28		6.9	
19	CF/CL 16	1.5 L	24 H	87 H+	171 H+	31		6.9	
20	CF/CL 17	3.3 M	30 H+	36 M+	269 H+	26		6.4	

L-: Low L: Low M: Medium M+: Above Medium H: High H+: Very High

To convert HECTARES into ACRES multply by 2.47				To convert T/HECTARE into T/ACRE multiply by 0.45			To convert Kg/Ha into lbs/ACRE; multiply by 0.9		
Sample Information				Limestone application (T/Ha) to achieve			Required Applications (Kg/Ha)		
Lab Sample #	Field Number	Field Size (Ha)	Crop to be grown	pH 5.5	pH 6.0	pH 6.5	Nitrogen N	Phosphate P_2O_5	Potash K_2O
16	35		Unknown			2			
17	36		Unknown						
18	37		Unknown						
19	CF/CL 16		Unknown						
20	CF/CL 17		Unknown			4			

Lab Sample #	Field Number	Man	Sod	CEC (Meq/100g)	Base Saturation % K	% Mg	% Ca	% H	% Na	Total % Base Saturation
16	35	0	0	9	5.1	10.4	55.9	27.1	1.4	71.4
17	36	0	0	8	4.4	13.2	65.3	15.5	1.6	82.9
18	37	0	0	8	2.5	13.9	67.5	14.6	1.5	83.9
19	CF/CL 16	0	0	10	.9	16.1	65.0	16.7	1.3	82.0
20	CF/CL 17	0	0	13	2.8	9.6	33.1	53.6	.8	45.5

Comments:

Methods: Water pH (1:1, soil:water)
Lime Index: SMP Buffer Method
Nutrient Extraction: Melich III
% Organic Matter: Combustion Furnace

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Sample Information		Soil Test Values and Ratings							
Lab Sample #	Field Number	Organic Matter (%)	pH	Phosphate P_2O_5 (ppm)	Potash K_2O (ppm)	Calcium Ca (ppm)	Magnesium Mg (ppm)	Boron B (ppm)	Copper Cu (ppm)
21	CF/CL 18	3.5	6.6	646 H+	193 H+	1540 M+	170 M+	.7 M	4.4 H+
22	CF/CL 19	3.0	5.8	509 H+	213 H+	703 L	75 M	.3 L	4.3 H+
23	CF/CL 110	4.1	6.6	482 H+	152 H	1510 M	281 H	.5 M	3.1 H+

Lab Sample #	Field Number	Zinc Zn (ppm)	Sulfur S (ppm)	Manganese Mn (ppm)	Iron Fe (ppm)	Sodium Na (ppm)	Aluminum Al (ppm)	Lime Index	Conductivity mS/cm
21	CF/CL 18	4.2 M	26 H+	52 H+	244 H+	29		6.8	
22	CF/CL 19	2.6 L	27 H+	46 H	230 H+	21		6.5	
23	CF/CL 110	2.4 L	26 H+	44 H	284 H+	33		6.8	

L-: Low L: Low M: Medium M+: Above Medium H: High H+: Very High

To convert HECTARES into ACRES multiply by 2.47 | **To convert T/HECTARE into T/ACRE multiply by 0.45** | **To convert Kg/Ha into lbs/ACRE: multiply by 0.9**

Sample Information				Limestone application (T/Ha) to achieve			Required Applications (Kg/Ha)		
Lab Sample #	Field Number	Field Size (Ha)	Crop to be grown	pH 5.5	pH 6.0	pH 6.5	Nitrogen N	Phosphate P_2O_5	Potash K_2O
21	CF/CL 18		Unknown						
22	CF/CL 19		Unknown			3			
23	CF/CL 110		Unknown						

Lab Sample #	Field Number	Man	Sod	CEC (Meq/100g)	Base Saturation % K	% Mg	% Ca	% H	% Na	Total % Base Saturation
21	CF/CL 18	0	0	12	3.4	11.7	63.8	20.1	1.0	78.9
22	CF/CL 19	0	0	11	4.3	5.8	32.9	56.1	.9	43.0
23	CF/CL 110	0	0	13	2.5	18.1	58.3	20.0	1.1	78.9

Comments:

Methods: Water pH (1:1, soil:water)
Lime Index: SMP Buffer Method
Nutrient Extraction: Melich III
% Organic Matter: Combustion Furnace

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Appendix D

Field Dimensions

And

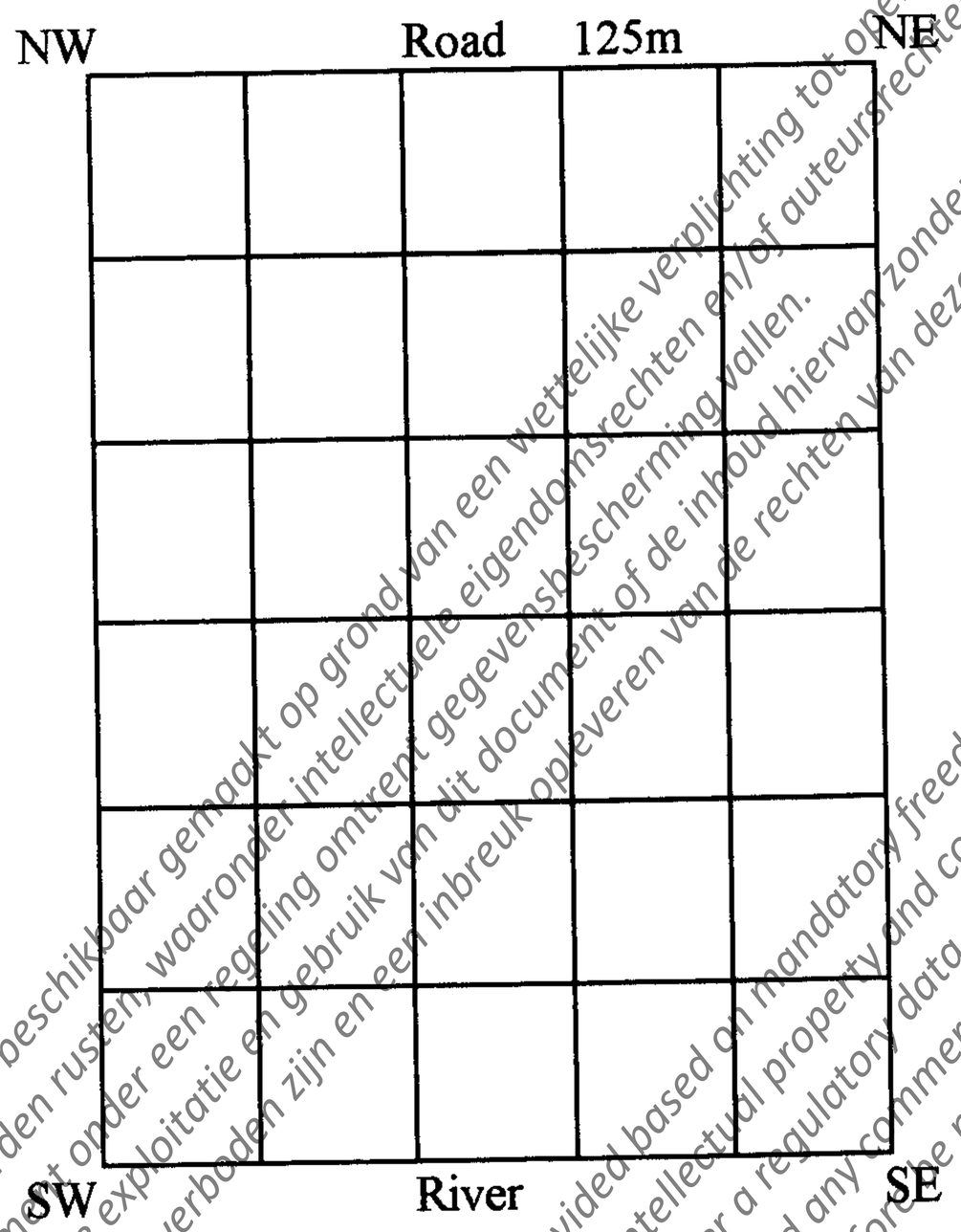
Locations

X, Y coordinates of the centroid of study sites in PEI and NB.

Site No.	Decimal Degrees	
	West Latitude (X)	North Longitude (Y)
01	63.84893	46.40509
02	63.76560	46.40360
03	63.47773	46.20790
11	63.66971	46.32987
12	63.85623	46.42185
13	63.73406	46.36518
14	63.83641	46.40495
15	63.35015	46.16347
21	63.17417	46.31000
22	63.35108	46.16472
23	63.29167	46.24825
31	63.29100	46.27733
32	63.31383	46.32108
33	63.53692	46.25792
34	63.29813	46.25078
35	63.85267	46.43917
36	63.53775	46.26042
37	63.29275	46.19014
16	61.70607	46.14381
17	61.56417	46.31362
18	61.59682	46.42447
19	61.59400	46.48506
110	61.55975	46.37425

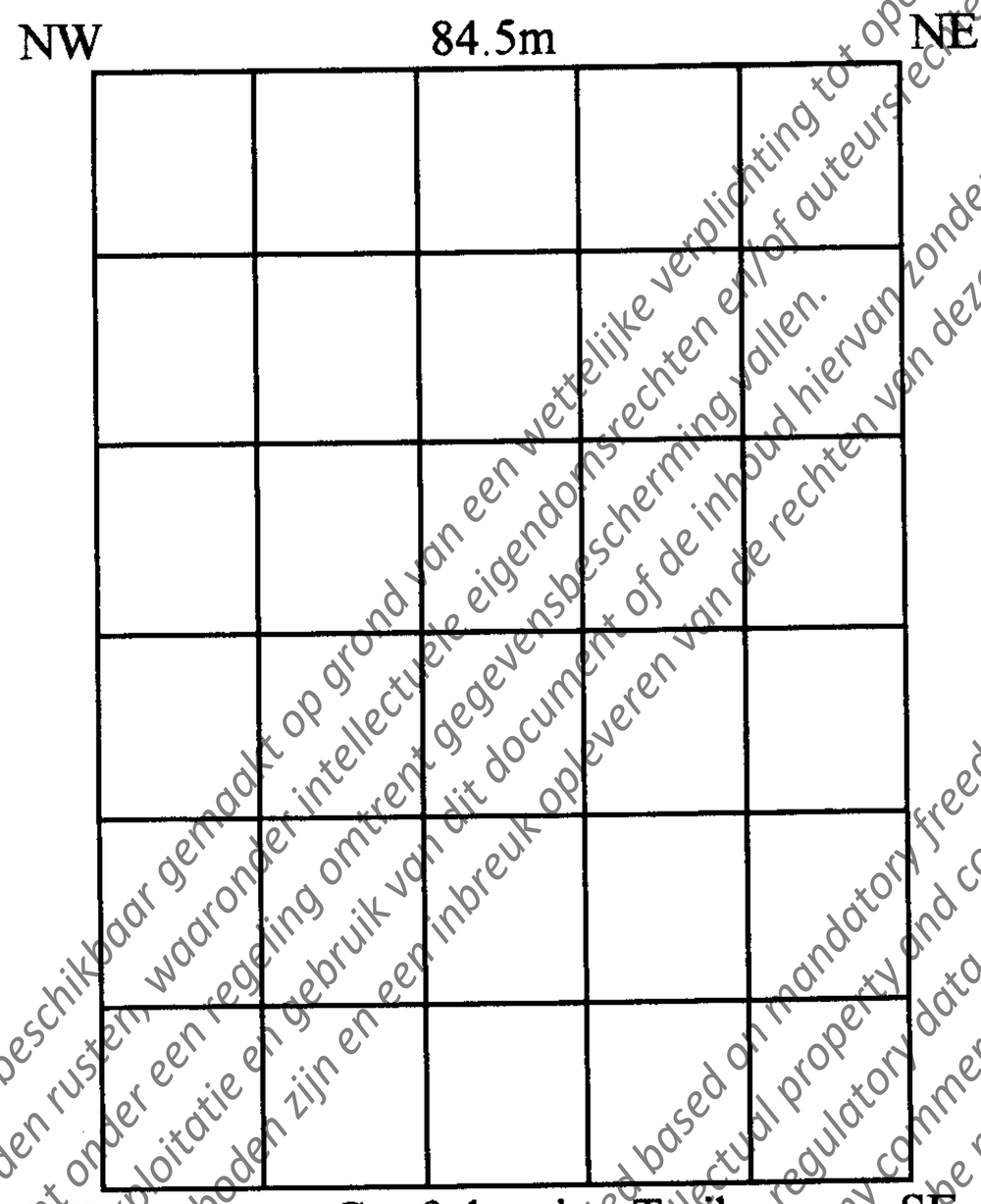
Field 01

Dimensions: 161m x 125m



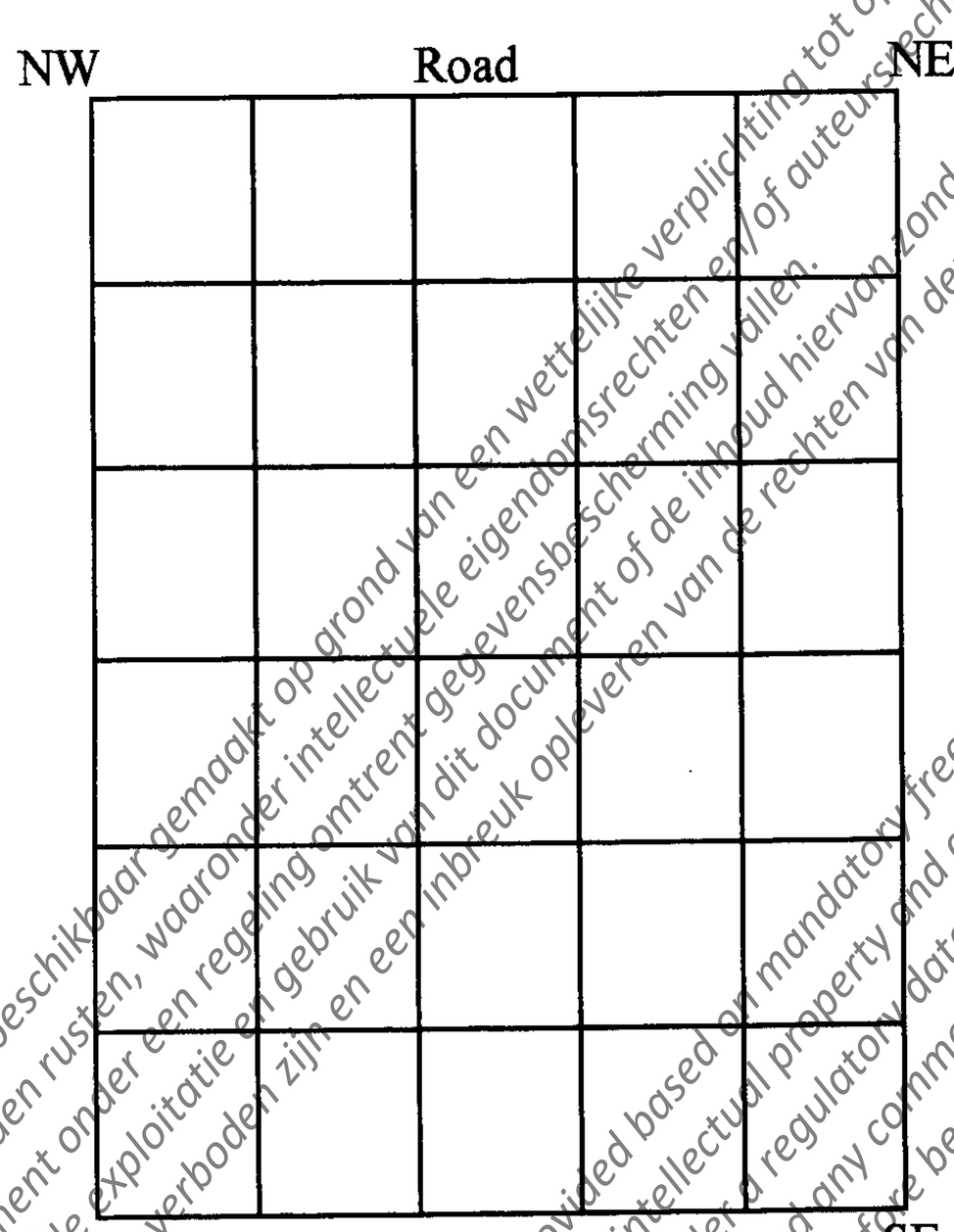
Field 02

Dimensions: 238m x 84.5m



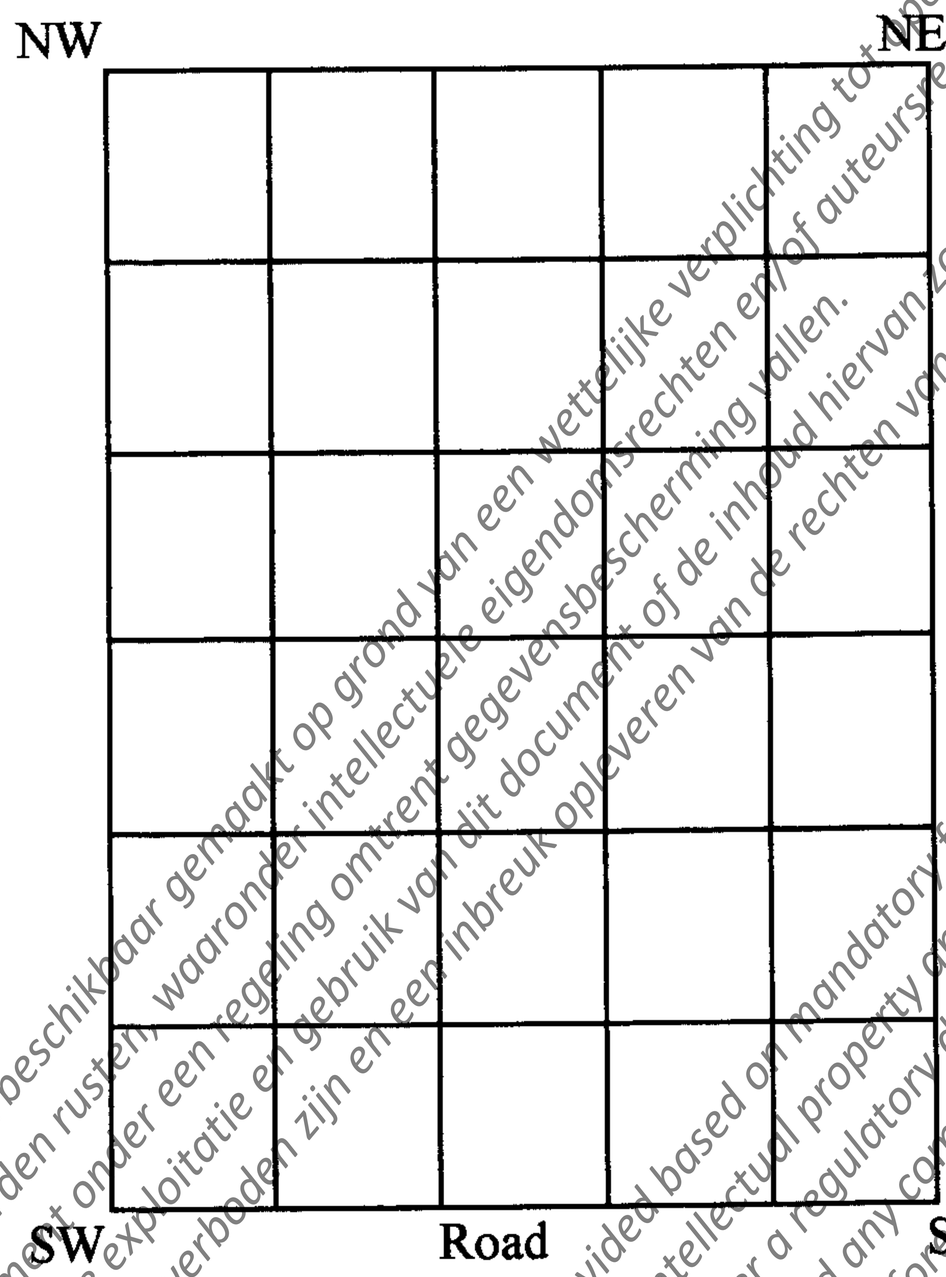
Field 03

Dimensions: 88.8m x 226.5m



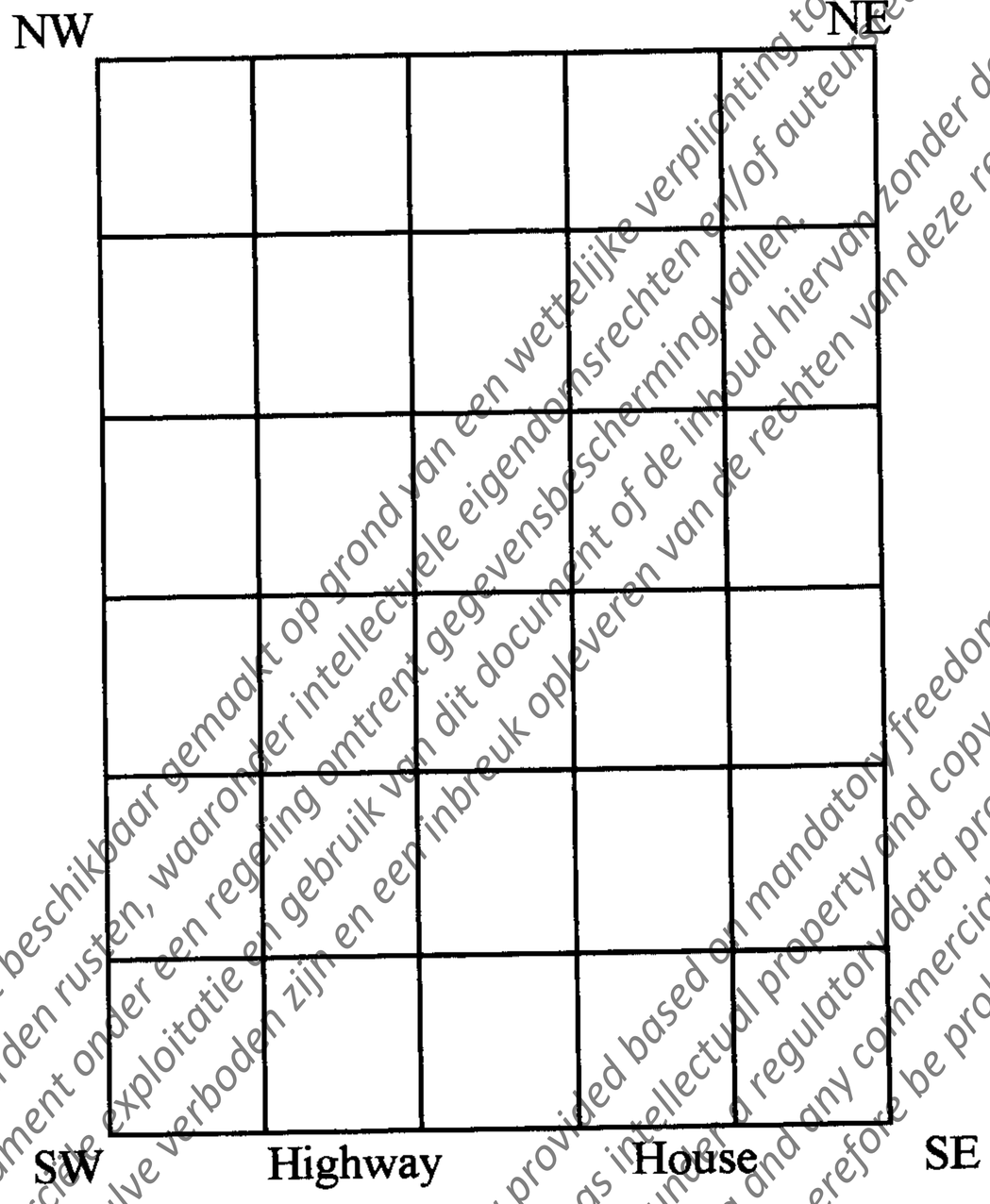
Field 11

Dimensions: 111.5m x 180m



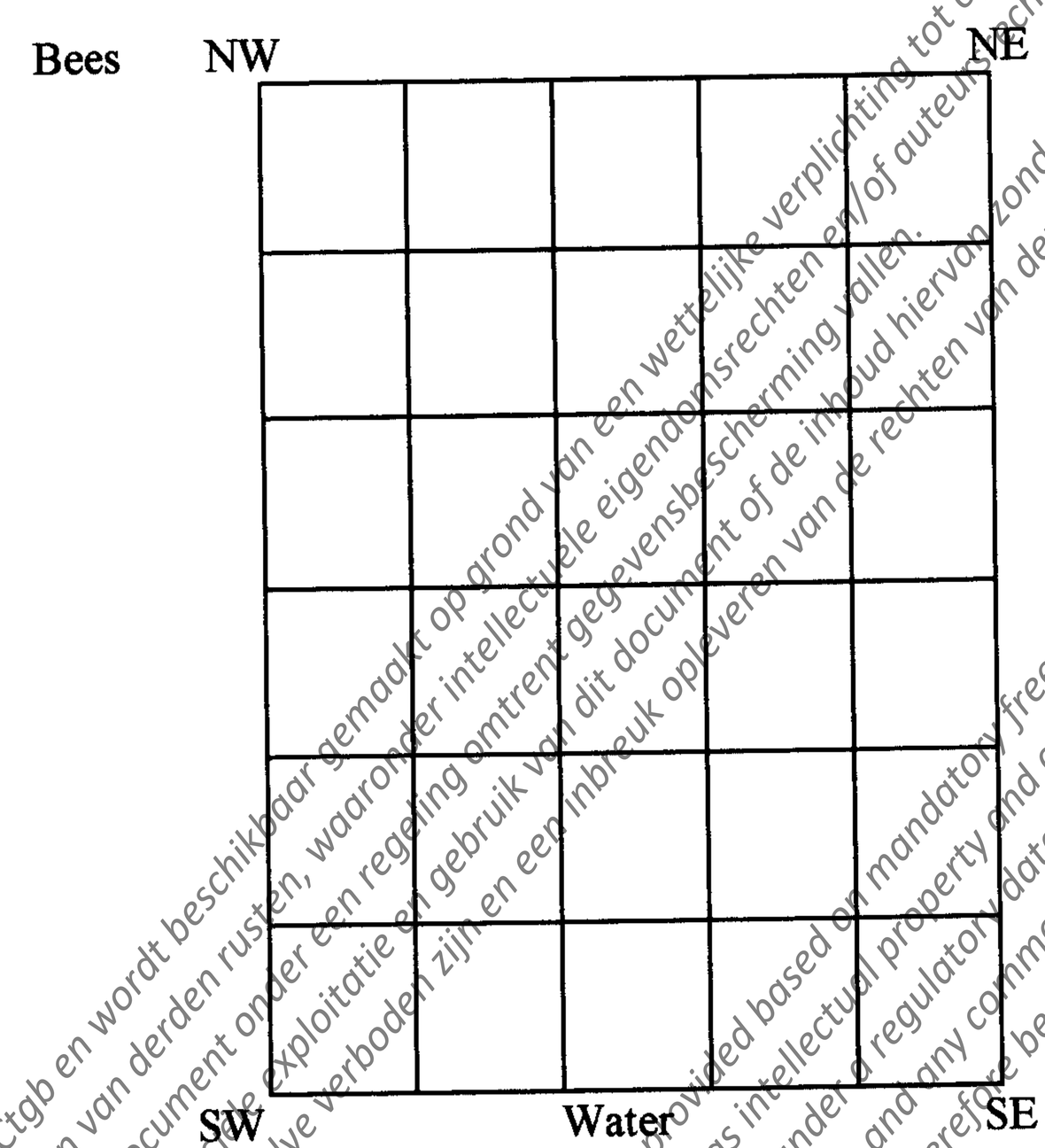
Field 12

Dimensions: 150m x 134m



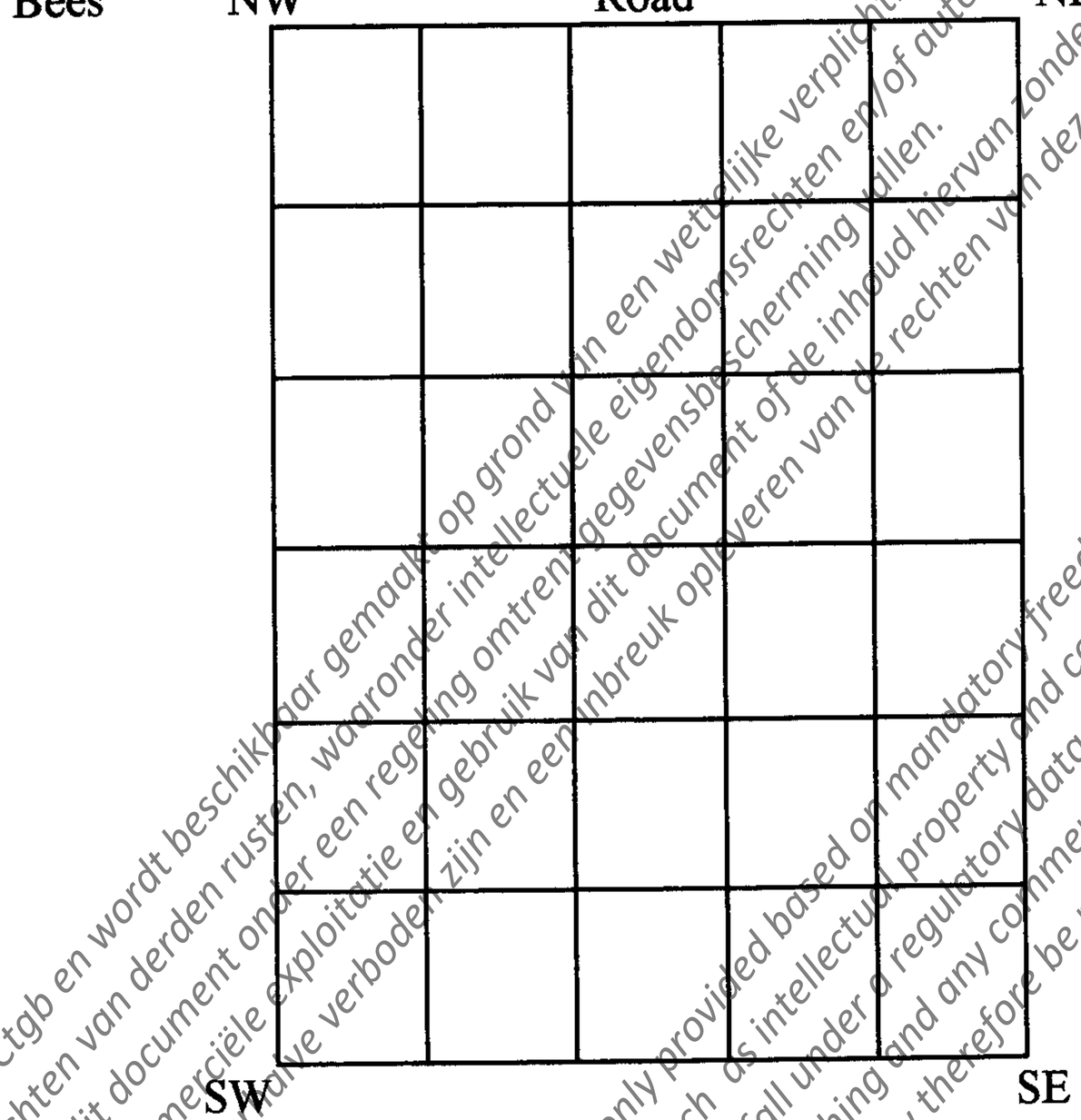
Field 13

Dimensions: 97m x 207.3m



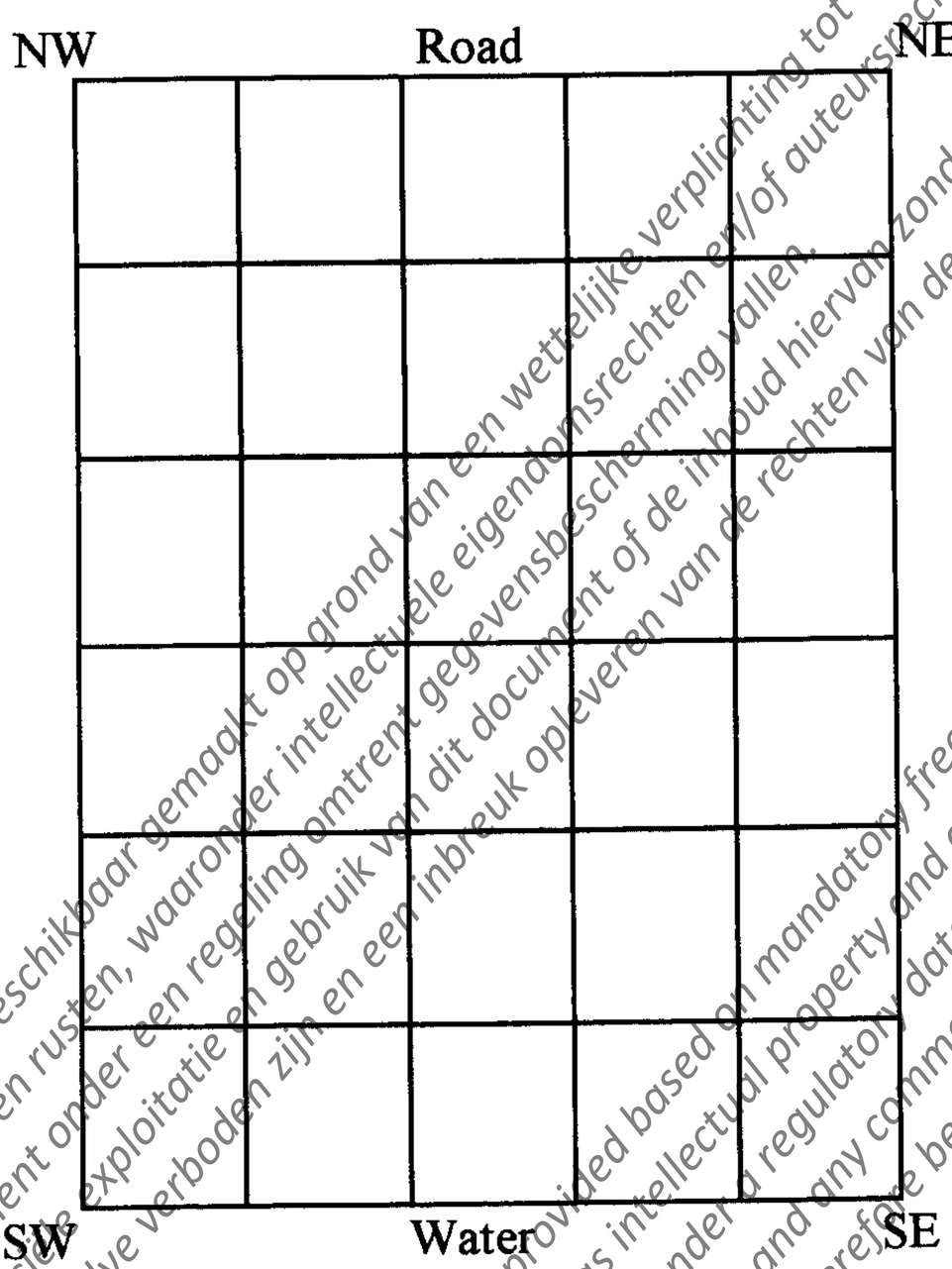
Field 14

Dimensions: 84m x 239.4m



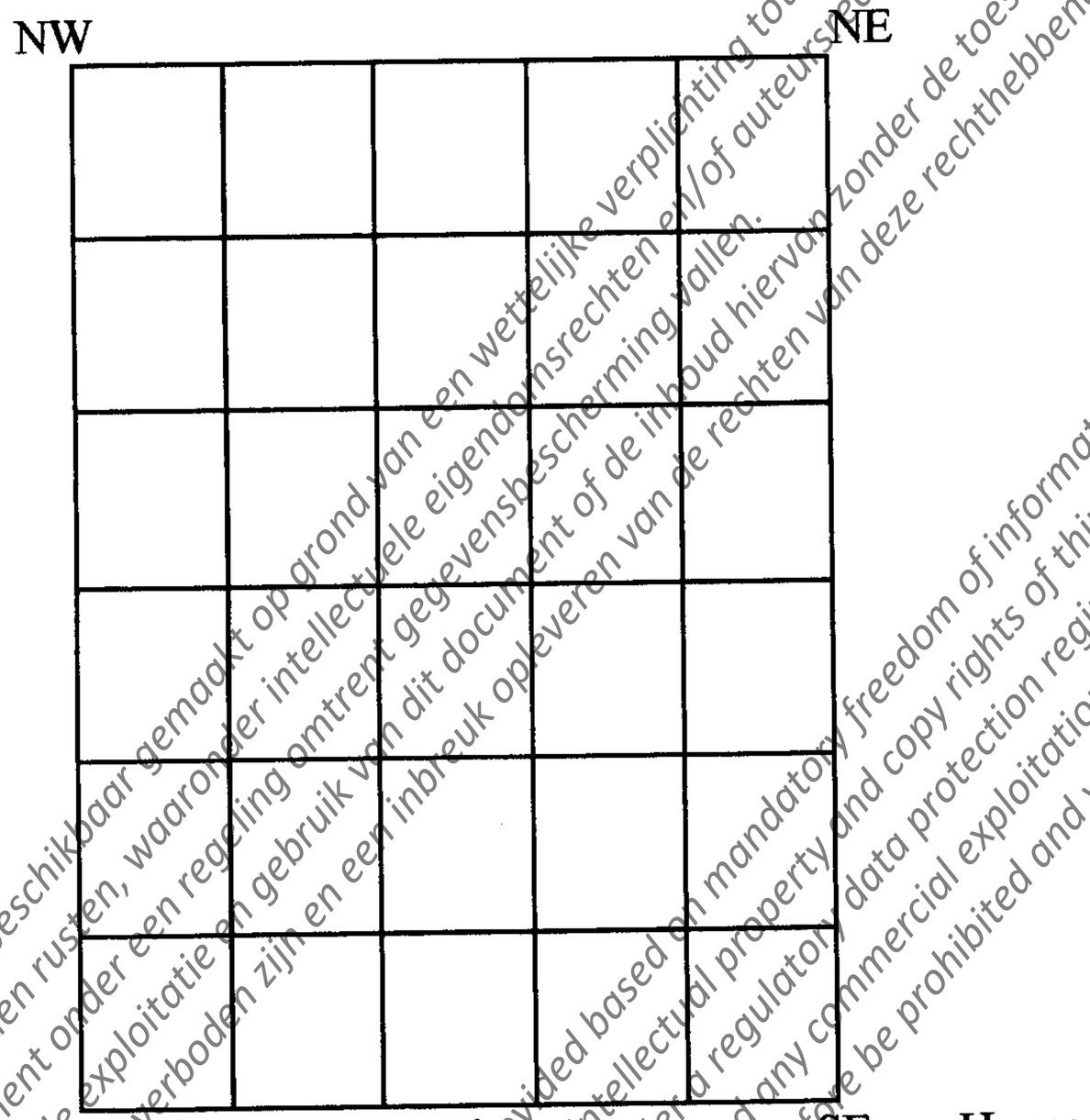
Field 15

Dimensions: 88.8m x 226.5m



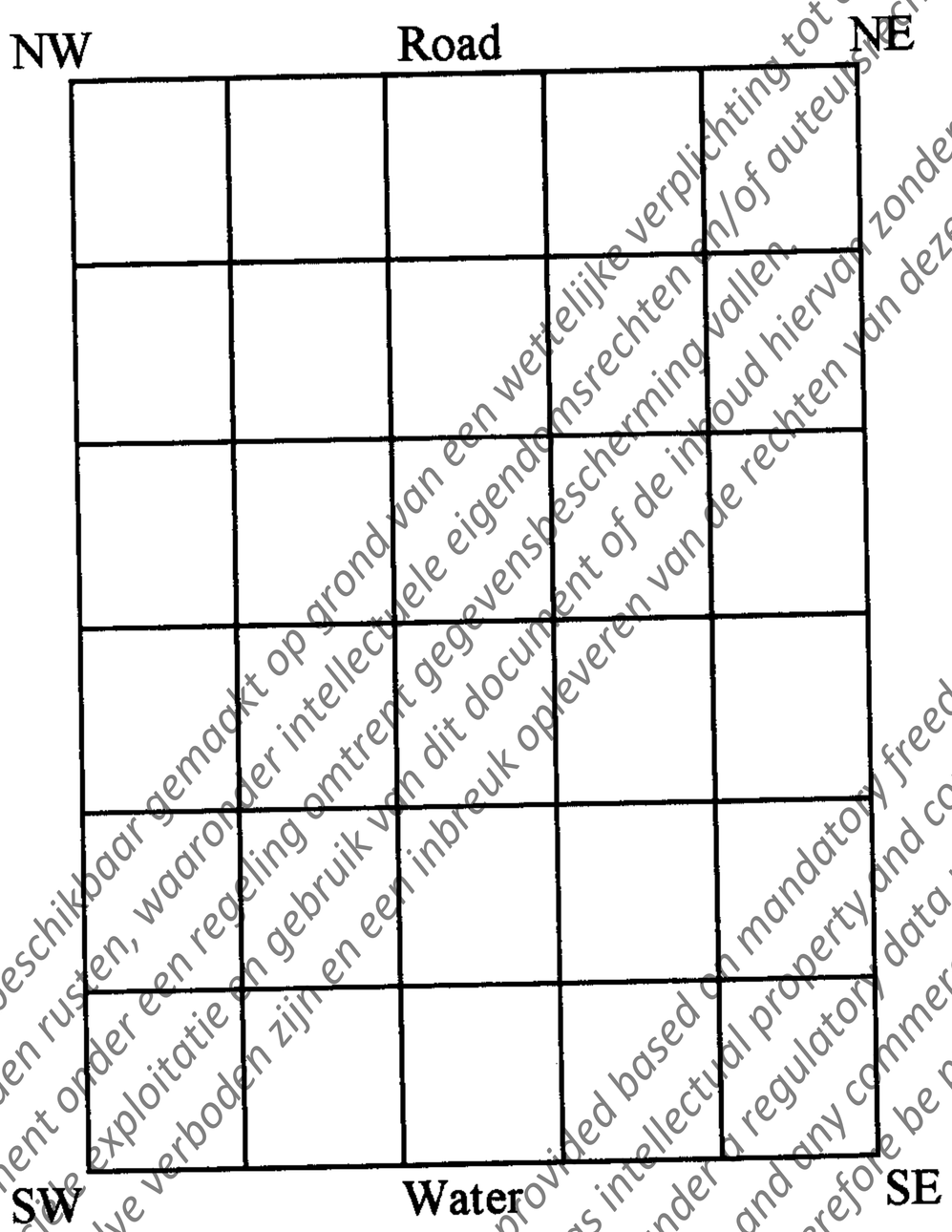
Field 21

Dimensions: 136m x 147.9m



Field 22

Dimensions: 100.6m x 200m



Field 23

Dimensions: 57.5m x 221.5m