IR-4 Laboratory Guidance Document

Dr. David Soderlund
Chair, Project Management Committee (Date)

Dr. Mary Duryea
Chair, Administrative Advisers (Date)

Dr. Jerry Baron
Executive Director of IR-4 (Date)

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IR-4 Laboratory Guidance Document

Introduction

This Guidance Document has been developed to provide consistency and to facilitate communication among the IR-4 Laboratory Research Directors (LRDs), Regional Directors (RDs, management), Quality Assurance (QA), and the IR-4 Study Directors (SDs) and will serve as a resource for all facets of IR-4. It indicates how IR-4 operates, by designating responsibilities and providing guidelines for implementation of procedures, to assure that all studies conducted by IR-4 meet EPA GLP regulations. Once this document is approved by the IR-4 PMC, it becomes an official policy document for the conduct of studies in the IR-4 laboratories.

The main areas of attention in this document include personnel responsibilities in relation to IR-4 residue work, definitions and a significant section regarding lab operations with emphasis on sample handling and storage; sample processing; analytical method validation; sample analysis and extract storage; storage stability studies; communication with the study director; and the Analytical Summary Report. This document will provide guidance for contract labs and will be used as a training tool with regard to IR-4 analytical work.

Please note: paragraphs formatted with *italics* are taken directly from the "Operational Handbook of IR-4" Version 7.0

Original Committee members:
Daniel Kunkel, IR-4 Headquarters, Associate Director (Chair)
Debbie Carpenter, IR-4 Headquarters
Tom Hendricks, USDA-ARS, Tifton, GA
Matt Hengel, Western Regional Laboratory Coordinator, University of California
Wayne Jiang, North Central Regional Laboratory Coordinator, Michigan State University
Christopher Lam, North Eastern Regional Laboratory Coordinator, Cornell
Jim McFarland, Western Region QA Coordinator, University of California
Marion Miller, Western Region Director, University of California
Jau Yoh, Southern Regional Laboratory Coordinator, University of Florida

Review Conducted by Analytical Chemists Advisory Committee (AC-AC):
Debbie Carpenter, IR-4 Headquarters
Tom Hendricks, USDA-ARS, Tifton, GA
Matt Hengel, Western Regional Laboratory Coordinator, University of California
Todd Wixson, USDA-ARS, Yakima, WA
Sue Erhardt, North Central Regional Laboratory Coordinator, Michigan State University
Wlodzimierz (Wlodek) Borejsza-Wysocki, Southern Regional Laboratory Coordinator, University of Florida
Sherita Normington, Western Region QA Officer, University of California
David Soderlund, North Eastern Region Director, Cornell

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Responsibilities

IR-4 Headquarters (HQ) Staff *coordinate the program among the regions and USDA-ARS, and provide functions including:*

1) GLP oversight including Study Director and Quality Assurance.
2) *Prepare research protocols.*
3) *Review, analyze, and archive raw data.*
4) *Prepare, review, and submit petitions to establish and maintain tolerances.*
5) *Interact with EPA and cooperating registrants.*
6) *Maintain a database to track projects.*
7) *Oversee Manufacturer and Contract Laboratories*

The HQ office is administered by an Executive Director (Management Representative).

Regional Research Programs. *Each Regional Program is administered by a Regional Director who has overall responsibility for GLP compliance at the regional level. The Regional Director has Regional Laboratory, Field and QA Coordinators who work with state scientists within their region and provide them with research support.*

1) *Regional Laboratory Coordinator (RLC): Oversees and coordinates regional laboratories and their satellite laboratories, and some contract laboratories, conduct analyses to determine test substance residues on crop samples.*
2) *Regional QA Coordinator: Monitors the field and laboratory operations in each region to assure that they are meeting GLP requirements.*

ARS Programs Research Personnel *The ARS Program is administered by an ARS National IR-4 Director who has overall responsibility for GLP compliance at the ARS Facilities. The ARS National IR-4 Director supports USDA-ARS residue laboratories and scientists (Laboratory Research Directors) that conduct analyses and determine test substance residues on crop samples. QA for these facilities is provided by other IR-4 QA and contract QA.*

Definitions

GLP Definitions

Archives: *All raw data developed by the IR-4 program will be archived as required under 40 CFR 160.190. Archivists will be designated by the Executive Director for IR-4 HQ and an index of archived laboratory data from the RLCs will be sent periodically to the HQ Archivist.*

Protocol: *The regulations require an approved written protocol for each study. The SD is responsible for the development of the protocol, which is prepared in accordance with the information as outlined under 40 CFR 160.120. Protocols will contain both the field and laboratory phases of each study and detail the proposed sites for the research. The regulations require that the protocol be approved by the SD and sponsor by signing and dating.* The Project Management Committee (PMC, sponsor) delegates approval of the protocols to the Executive Director or his/her designee.

Quality Assurance Unit: *The QA unit will conduct facility inspections at all IR-4 test locations and conduct critical phase inspections of each study at intervals adequate to ensure study integrity. All QA audits from facility and critical phase inspections will be provided to the appropriate SD and Management (IR-4 Executive Director) for review, appropriate response and corrective action, and signature. Those reports that require action may be forwarded to the Regional Directors as necessary. The HQ QA Manager will maintain a copy of the Master Schedule for all IR-4 studies.*

Sponsor: *The sponsor is the person who initiates and provides financial or other support for a study. The IR-4 Project Management Committee acts as sponsor for IR-4 studies and has designated the Executive Director as sponsor for the purposes of GLP. The Executive Director may delegate individuals to act as Sponsor Representative to sign the protocol, etc.*

Study: *An experiment conducted at the IR-4 Research Facilities (or contract facilities) to determine the magnitude of the residue (test substance) in or on a given commodity to provide the sponsor with residue chemistry data to support a pesticide tolerance.*

Study Director: *The SD represents the single point of study control, and is responsible for the overall conduct of the study. The accountability provided by a single SD (who plans, oversees, and controls the interpretation, analysis, documentation, and reporting of the results) is one of the most important aspects of the GLP standards. For IR-4 studies, the SD oversees the research of FRD and LRDs who are responsible for carrying out the field and analytical duties. The RLCs, RFCs, and ARS National IR-4 Director assist the SDs in meeting their responsibilities.*

Testing Facility: *IR-4 HQ serves as the testing facility for the purposes of GLP. The Executive Director will represent testing facility management, and the SDs and QAU will report to the Executive Director.*

IR-4 Definitions

Laboratory Research Director: *A person with sufficient training and experience to be able to conduct the laboratory analysis and appoint adequate personnel to assure this function will be carried out for all studies. The LRD will report all deviations from the protocol or the SOPs to the SD.*

Quality Assurance Coordinator (QAC) and Officers (QAO): *These persons, designated by the Regional Director or Executive Director, report the findings of their audits to the SD, to the Executive Director (Testing Facility Management) and to other research associated personnel. The QAC/QAO will monitor studies, including facilities, equipment, personnel, methods, practices, records and controls, for compliance with GLP. The QAU reviews the final report to assure that it accurately reflects the raw data of the study and prepares and signs a Quality Assurance Statement noting dates the inspections and findings were reported to the SD and SD Management.*

Regional Laboratory Coordinator: This person assigns laboratory-testing sites within his/her region for residue analyses conducted by the regional laboratory, its satellites, and private contract laboratories.

References

Good science is key to successfully completing the analytical portion of any study. However, it is just as important for SDs and LRDs to be aware of the impact of the following references. These references provide a framework for all IR-4 study related work.

Operational Handbook of IR-4 (current version).
Good Laboratory Practice Standards, 40 CRF Part 160, August 17, 1989.
Food and Feed Crops of the United States, Markle et al. 1998.
OPPTS 860 Residue Chemistry Test Guidelines including:

- OPPTS 860.1000, Background
- OPPTS 860.1340, Residue Analytical Method
- OPPTS 860.1380, Storage Stability Data
- OPPTS 860.1500, Crop Field Trials
- OPPTS 860.1520, Processed Food/Feed

Lab Operations

Standard Operating Procedures: The development of a comprehensive set of SOPs that address the development, monitoring, and reporting of data from specific study phases conducted at the research test site is the responsibility of each Laboratory Research Director at that site. RLCs and the ARS National IR-4 Director and/or ARS Facility Research Leader, or designee provide guidance for and approval of SOPs. This guidance document will take precedence over SOPs and they may therefore need modification after this document is put in place.

Standards and Solutions

Obtaining Standards: IR-4s current policy is that all reference standards must be characterized under GLP preferably before the analysis begins, but definitely before the study is completed (signed by the study director). Due in part to the large number of registrants IR-4 works with, obtaining GLP standards can be difficult. It is recommended that the LRD initiate discussions with the cooperating registrant. If standards cannot be acquired in sufficient time frame, then the LRD is directed to contact the SD or Registrations Manager at IR-4 HQ to seek assistance in obtaining standards. Purity value given on the Certificate of Analysis should be used for all calculations of the standard concentration. In the case where a non-GLP standard is required to complete the analytical phase of the project, IR-4 management, in concert with the SD, will be contacted for approval.

Characterization of Substances: ***Analytical Reference Standards;*** *Documentation of the characterization of the standards used in the analytical trial should be obtained by the Laboratory Research Director and a copy sent to the SD along with the Analytical Summary Report of the trial.*

Reagents and Solutions: *The GLP standards require all reagents and solutions in the laboratory area to be labeled to indicate identity, titer or concentration, storage requirements, and expiration date. This requirement can be difficult to accomplish when there is a mix of IR-4 and non-IR-4 personnel utilizing the laboratory and sharing the chemicals or when the chemical is stable and has a long shelf life. The following is to be used as a guide for meeting the labeling requirement:*

1) *Identity can be the common name(s), CAS number or chemical name of the reagent or reagents in solution or mixture.*
2) *If the labeling of the original container provides the identity, concentration, storage requirements (if any) and expiration date or shelf life, no additional information is needed. If the labeling does not contain this information, than a supplemental label containing the missing information should be permanently attached to the container where it does not obscure other critical information.*
3) *All mixtures of chemicals prepared by laboratory personnel for use in IR-4 trials should have labels with the information as shown in 2 above.*
4) *Expiration dates for stable chemicals should be determined by the Laboratory Research Director following methods outlined in their SOPs.*
5) *Adequate precautions should be taken to avoid contamination of reagents and solutions so that purity of their content is preserved.*

Sample Receipt, Processing and Storage

Maintaining a representative sample and maintaining sample integrity are the important considerations to keep in mind during sample receipt storage, processing, and extraction/analysis (see Appendix 1.).

Sample Receipt: Samples are generally received from either ACDS, Fed Ex or other carriers. For receipt of samples from an overnight air express carrier such as FedEx, it is critical that the lab know a shipment is in transit. If the shipment is not received as expected, laboratory personnel will follow-up to track the shipment.

When samples are received, laboratory personnel check the condition of the samples; verify receipt of the correct samples by checking sample identification and matrices against the shipping papers. Unique laboratory numbers are assigned and recorded with cross reference to field samples IDs. Shipping forms (8B) received with the samples may be used to record the cross reference or custom forms may be used. At a minimum, custom forms must contain the same information required on the FDB forms, and must show that protocol conditions have been met (for example, acknowledging that forms 8B and 8C were shipped with the samples). The SD, RFC, and FRD are notified when samples are received, and any problems with the shipment are to be brought to their attention.

Sample Processing: For information regarding sample preparation, size and providing homogeneous representative samples see Attachment 1. Great care is taken in the field to collect samples from all areas of the plot, so that the sample is representative of the whole field. When processing the samples, the total sample must be processed, and thoroughly mixed. If this is not possible, guidance from the Study Director and/or Registration Manager must be sought. Sample integrity is generally maintained by processing samples with dry ice. The study analytical data must document how representative samples were prepared.

Storage: Storage of samples is in accordance with the protocol requirements and SOP's. To prepare for the loss of power or a freezer failure, consideration should be given to the availability of backup freezers and dry ice, generators (power backup) and spare parts. Temperature monitors, alarms, and established lines of notification are methods for providing the LRD with information on the temperature of the storage areas. For a longer term power outage, samples may need to be transported to another location to maintain sample integrity. These samples represent a significant investment and their integrity should be safeguarded accordingly.

Working Method, Validation and Modifications: IR-4 methods are provided initially by the cooperating registrant (reference method). Given the number of commodities IR-4 works with, it is likely that each method will require some modification to work effectively. It should be noted that once these methods are modified for other commodities, these methods become the enforcement method for EPA. Significant changes to the initial working method may trigger an independent laboratory validation (ILV, OPPTS 860.1340), and thus are not encouraged unless needed to develop an adequate method for the specific matrix. The LRD should discuss "significant changes" with the SD and/or gatekeeper prior to making the change.

Other considerations*:* Approval for significant changes to the reference method must be requested from the SD, HQ gatekeeper and registrant. Depending on the number of proposed changes and familiarity with the method, the laboratory should keep in mind that such changes will need to be dealt with well in advance of analysis, so that when the samples are received analysis may proceed without delay.

Extraction*:* In most cases the extraction solvent and procedures must remain the same as the reference method. Sample weights and extraction volumes must stay proportional to the original method. However, in some cases, the ratio of extraction solvent to sample weight can be increased to improve extraction efficiency (e.g. extracting high K_{ow} pesticides from high fat/oil content crops). Exchange of equipment can be made only when the equipment is basically carrying out the same function as noted in the method (for example tissuemizer and polytron). Other substitutions (from tissuemizer to shaker tray) should be discussed with the registrant providing the reference method and in consultation with the SD (and the gatekeeper[1] at HQ).

[1] The role of the Gatekeeper (Debbie Carpenter, Dan Kunkel or Bill Barney) is to provide greater consistency from IR-4 HQ by utilizing personnel with greater chemistry experience.

Clean-up steps*:* EPA has noted that as long as the extraction procedures are the same, clean-up steps maybe added or removed. It should also be noted that removing an excessive number of steps may result in excessive wear and tear on the column and instrument. The impacts of removing clean-up steps from the method, such as matrix enhancement effects, must be evaluated as chromatography must be clean and sharp. Modifications should be discussed with the SD, "IR-4 Gatekeeper[2]" as well as the registrant so they can share their experiences. Chemist should also consider cost and time relating to removal of cited clean-up steps.

Detector*:* Using LC-MS/MS has generally become the norm and essentially all of the IR-4 laboratories have at least one instrument. It is likely that any new equipment purchases will be directed toward using this technology. Therefore, replacing the detectors noted in the reference method with LC-MS/MS should have minimal effect on the method while providing better quantitation and confirmation.

Working method approval and validation data*:* Current minimum protocol requirements indicate that the LRD will send the SD the working method and recovery data from the method validation. If the recovery data are within 70 to 120% (reported as nearest whole number) then weathered sample analysis may proceed. However, it is expected that the SD take an active role in this process and acknowledge that the method and data are acceptable. If the recovery data are not within the 70 to 120% range, the SD must acknowledge that he/she is aware the data are out of range and if the data are acceptable. If the validation recoveries are within the 70 – 120% range it is at the discretion of the LRD to request SD approval prior to analysis of the field (weathered) samples in order to note SD responsibility. If study director approval is needed or requested, the study director should make every effort to respond within 2 working days. Recognizing that study directors have other responsibilities, including traveling, the lab will need to provide time for the study director to respond in these situations. For urgent needs, or situations where the SD is not able to respond within 2 working days, approval to proceed may be sought from the Gatekeeper[2]. However, the SD must provide approval when he/she becomes available.

Sample Analysis and Extracts

Sample Analysis: As noted in the protocol, each analytical set will have at least one concurrent recovery sample. Typically the fortification levels will reflect the expected residues in the treated samples. In the case where no residues are expected, fortifications should be at the lowest level of method validation (LLMV).

IR-4 laboratories agree that double injections for each weathered sample should be used. If there is a study with a large number of samples, the LRD may consider doing single injections: however, it should be noted that double injections provide a number of benefits such as instrument stability and detection of "bad injections" in real time, allowing the chemist to respond to situations more quickly and efficiently. LRDs will have the appropriate SOPs in place to define pass or fail criteria for poorly reproducing injections.

[2] The role of the Gatekeeper (Debbie Carpenter, Dan Kunkel or Bill Barney) is to provide greater consistency from IR-4 HQ by utilizing personnel with greater chemistry experience.

Laboratory personnel should be mindful when unusual results are obtained and notify the SD immediately. (Lab personnel may want to re-extract and re-analyze samples to confirm prior to notification of SD). Examples of unusual situations are samples that have no residues compared to other weathered (field samples) samples from treated plots, or decline samples where no residues are detected, samples from untreated control plots with residues or if residues from samples taken from the same treated plot have measurable residues and the values for each sample vary by a factor of 5X or more.

Extracts: "Registrants are advised to routinely include the storage of extracts, unless their standard laboratory practice is to analyze extracts on the same day as they are obtained "(860.1380). Always run samples with concurrent recoveries to demonstrate extract stability.

Storage Stability: IR-4 does not carry out guideline storage stability studies as outlined in 860.1380. Our purpose is to show the samples are stable under the storage conditions used. Currently, storage stability, with analysis of one time point is carried out for most studies. For many compounds, the registrant may have adequate storage stability data available. IR-4 is working with EPA and the manufacturer to determine this. IR-4 will transition to fewer storage stability, where possible. However, analysis at two time points is often included for compounds new to IR-4. The first time point will be when the method is validated (3 samples) and another after an appropriate storage period (another 3 samples). A minimum of nine samples will be spiked at 10x LLMV. If the samples cover 90% of the storage time (from sample date to extraction date), this is sufficient, as per the protocol. In some cases the SD may be able to waive the storage stability analysis. Documentation of the waiver by the SD is required.

Communication of Results with SD:

Response Needed to Proceed:

There may be instances where the lab needs to communicate study related activities to the study director, and a response is needed to proceed. One example includes out of range recoveries. If the recovery data are not within the 70 to 120% range, the SD must acknowledge that he/she is aware the data are out of range, accepts the recoveries, and that the analysis may proceed. If study director approval is needed or requested, the study director should make every effort to respond within 2 working days. Recognizing that study directors have other responsibilities including traveling, the lab will need to provide time for the study director to respond in these situations. For urgent needs, or situations where the SD is not able to respond within 2 working days, approval to proceed may be sought from the gatekeepers, Debbie Carpenter, Dan Kunkel or Bill Barney. However, the SD must provide approval when he/she becomes available.

Routine Results: The LRD (or designate) will provide routine updates to the SD (e.g. residue analysis spreadsheet, residue result summaries) on a regular basis, along with background information and assessment of the data. The lab will decide the frequency of updates, based on their own operations. At a minimum, it is expected that the residue results will be shared with the study director as soon as possible, once all samples for the study have been analyzed. Acknowledgment of their receipt from the study director is expected.

Analytical Summary Report: a sample ASR is provided in Attachment 2.

Training. This document will be used as a training tool for new Laboratory Coordinators, IR-4 chemists, QA officers and Study Directors.

Contract/Company Labs. This document may also be used as a tool to provide guidance for contract and company laboratories used by IR-4 for residue analysis.

Guideline Document review: Target review is for every three years. Please note that significant material has been taken from the "Operational Handbook of IR-4" and updates to that document will affect this document as well.

Explanation of Attachments:

Attachment 1: Guidelines for the Preparation of Raw Agricultural Commodity Samples For Residue Analysis

This instructional guideline has been prepared to aid in insuring uniformity and consistency among IR-4 analytical facilities when preparing raw agricultural commodities (RAC) for *Magnitude of the Residue* determinations. The attachment provides information regarding sample preparation, size and providing homogeneous representative samples. Great care is taken in the field to collect samples from all areas of the plot, so that the sample is representative of the whole field and this guideline will help to insure that samples remain representative when processed in the IR-4 laboratories.

Attachment 2: Sample Analytical Summary Report.

This example report is provided to illustrate a typical IR-4 Analytical Summary Report and the critical elements that must be in a report. The tables etc have been updated to help aid final report preparation. Recently, EPA has begun to request that metabolite residues be expressed as parent equivalents, please refer to the protocol for specific reporting requirements.

Note that residues from weathered samples are to be reported using a minimum of 2 significant figures.

Also, it is imperative that all of the pages of the ASR be readable. For electronic copies of this example please go to IR-4 Laboratory Guidance Document

Attachment 3: Checklist for Review of Analytical Summary Reports

This checklist (version 1.1, 2/5/2013) is being provided as reference informaiton to assist in the internal quality evaluation of analytical data. The checklist can be used to identify and insure that appropriate information is included in the final reports submitted to EPA. The checklist identifies items which must be brought to the study director's attention in order for the study director to carry out his/her responsibilities under GLP.

Attachment 1

Sample Processing Document

GUIDELINES FOR THE PREPARATION OF RAW AGRICULTURAL COMMODITY SAMPLES FOR RESIDUE ANALYSIS

PURPOSE:

This instructional guideline has been prepared to aid in insuring uniformity and consistency among IR-4 analytical facilities when preparing raw agricultural commodities (RAC) for *Magnitude of the Residue* determinations.

This guideline contains general directions for:

- obtaining in a safe manner homogeneous RAC sub-samples with minimum risk of residue cross-contamination ("General Procedures" section A)
- processing guidelines for specific crop groupings with specific instructions on inspecting and what portion of the RAC is to be prepared for residue determination ("Guidelines for Determining Portion of RAC to be Analyzed" section B)
- uniform sample preparation and comminuting procedures (i.e., pulverizing/ reduce to powder) for whole and sub-sampled RACs ("Guidelines for Sample Preparation" section C)

Definitions of Terms Used in this Guideline:

Raw Agricultural Commodity

Fresh fruits, whether or not they have been washed and colored or otherwise treated in their unpeeled natural form; vegetables in their raw or natural state, whether or not they have been stripped of their outer leaves, waxed, prepared into fresh green salads, etc.; grains, nuts, eggs, raw milk, meats, and similar agricultural produce. It does not include foods that have been processed, fabricated, or manufactured by cooking, freezing, dehydrating, or milling (40 CFR 180)

Sample

The amount of individual agricultural commodity units (e.g. specific number of fruits or tubers, a set weight of grain, etc.) randomly selected from a plot which may be composited for pesticide analysis (OPPTS 860.1500)

PROCEDURE:

A. General Guidelines

Persons given responsibility for processing agricultural crops (Processor) will be fully trained in properly processing agricultural commodities and also in the safe use of processing equipment and cryogenic materials. Proper ventilation is mandatory when working with cryogenic materials such as liquid nitrogen and carbon dioxide. It is the responsibility of the Processor to immediately notify her/his immediate supervisor and/or the Laboratory Research Director or if unsafe working conditions exist.

Processing equipment often operate at high revolutions to pulverize/powder the RAC. This equipment can be hazardous and should be routinely checked for proper operation before processing agricultural commodities.

The sample should not be brushed, stripped, trimmed, or washed except to the extent that these are commercial practices before shipment or to the extent allowable (see 40 CFR 180) or the Pesticide Assessment Manual (PAM). Details for cleaning or trimming specific crop types are outlined under "**Guidelines for Determining Portion of RAC to be Analyzed" section B** and Appendix 1. **In each case, the protocol and Study Director will be consulted to clarify any potential problems prior to sample processing.**

The total sample should be processed whenever feasible. If the sample size is too large to process, a representative sub-sample of each component part should be taken (e.g., 1/4 of each cantaloupe from the original residue sample bag for maceration). Sub-sampling of the component parts will be done in a manner to represent the residue distribution to be found on all surfaces of the whole vegetative part. Details for specific crop types are outlined under "**Guidelines for Sample Preparation**" **section C. If sub-sampling must occur, due to large sample size or unit size, the Study Director will be consulted prior to sample processing.**

The order in which samples are processed should be chosen to minimize the potential for residue cross-contamination. For each trial location, untreated samples should always be processed first. Treated samples with the lowest application rate and the longest pre-harvest interval (PHI) should follow. Samples with the highest application rate and the shortest PHI should be processed last. In addition, crop fractions should also be considered (e.g. nut meat fractions should be processed before hull fractions).

If cryogenic materials are required, the pulverized sample can quickly liquefy and separate at room temperature soon after processing. All attempts should be made to immediately transfer the sample to a properly labeled sampling bag and place in frozen storage.

Processing equipment should be thoroughly washed and rinsed with distilled water and acetone or methanol before attempting to process the next sample irrespective if the next sample is a replicate from the same treatment location or a replicated control sample.

B. Guidelines for Determining Portion of RAC to be Analyzed

40 CFR 180 specifies that the sample taken should be of the whole raw agricultural commodity (RAC) as it moves through interstate commerce. In certain cases, the portion to be analyzed for a residue tolerance may not represent the whole RAC. Instructions on what portion of the RAC should be analyzed are provided for nine individual food commodities (*e.g.*, bananas) and crop group commodities (*e.g.*, root vegetables) in this regulatory guideline. To fill this void, the FDA has provided additional guidance for RACs that fall under a more complete crop groupings list (see 40 CFR 180.34 (f)). The portion of the sample to be analyzed as described under PAM Volume 1 takes into

account practical considerations of sample preparation. Appendix 1 on page 4 (Table 102-a: *Portion of Raw Agricultural Commodity to be Analyzed for Pesticide Residues*) provides a compilation of EPA regulations and FDA directions to be followed for RAC preparation. If sample processing procedures for a particular RAC are not specified under the above crop grouping guidelines, or in the protocol, additional guidance from the Laboratory Research Director and IR-4 Study Director approval will be sought before preparing samples for residue determination.

C. Guidelines for Sample Preparation

The relatively small 2.5 to 100 gram laboratory sample taken from the whole RAC must represent the entire treated or control sample. Often these samples are *bulky* or can be comprised of *a few large units or many smaller items*. Whenever feasible, the total RAC sample should be pulverized and a homogeneous 2.5 to 100 gram sample taken to assure uniformity. Processing the entire sample may not always be feasible. Guidelines are provided below to aid in preparing representative residue determination samples from bulky, large unit and many small item RAC samples. In addition to the guidelines below, **Table 1** offers examples of current processing practices of several commodities by IR-4/ARS facilities.

Bulky Samples: For more bulky samples [i.e., Alfalfa (green and dry), Barley, Field Corn (silage, stover), Sweet Corn (forage, husks), Clover Grass, Mint (hay), Oats (forage, fodder, or straw), Rice (plants), Rye, Sorghum (plants), Soybean (plants), Sugar Cane (green and/or dry) Tobacco (green, cured), and Wheat (forage, fodder, or straw)], acquiring the relatively small laboratory sample usually consists of two steps. First, the crop is chopped into smaller size fractions using either a chopping knife or scissors or through use of a large capacity chopper/mixer/grinder such as a spinning bowl or vertical chopper (ie: Hobart HCM-450, 84142, 84145, 84146, VCM-25, or equivalent). The chopped sample is then frozen to a brittle consistency using either liquid nitrogen (LN_2) or dry ice. This frozen material is then processed to a fine consistency using a sample grinder (ie: Hobart 4822 or equivalent). Alternatively the samples may be first broken or chopped or into smaller size fractions as described above and then thoroughly processed with a cryogen (LN_2 or dry ice) in a spinning bowl chopper/mixer, spinning blade food processor (ie: Robot-Coupe. RSI-6V or 10B) or other food grinder/chopper

Sub-sampling: Typically, sub-sampling of bulky or heavy units is performed in the field as directed by the Protocol. However, when there are physical limitations for the laboratory processing of the whole sample due to mass or sample size, sub-sampling of the component parts must be done in a manner that assures the residue distribution is representative of the whole vegetative part. Laboratory sub-sampling should only be performed by GLP trained staff **and in consultation with the Study Director and or Registration Manager.** If absolutely necessary, this practice must be limited to special circumstances and be conducted by properly trained staff that understands the importance of maintaining a fully representative sub-sample and the risks of possible residue/cross contamination and/or deterioration of the crop matrix. Some examples of representative sub-sampling in the laboratory include:

- Taking a well mixed portion of a large sample of very small items (berries, nuts, grain, and immature vegetables). This may be necessary due to sample capacity of processing/milling/grinding equipment (i.e., small Hobart/Robot-Coupe choppers, Tekmar Analytical Mills and other similar chopping/grinding devices). For example, a well-mixed 1 kg sub-sample from the 5 kg composited RAC sample bag of coffee beans can be pulverized by the Tekmar Analytical Mill to produce a representative sample.
- For larger items when ca.12 units may comprise the entire composited RAC (melons, pineapples, squash, see CODEX, reference 3 and PAM section 120c), ¼ of each unit can be separated and composited to produce a representative sample for processing.
- In preparing a homogeneous tree fruit sample, where 6 fruits from each of 4 trees is recommended (CODEX, reference 3), ½ of each unit can be separated and composited to produce a representative sample for processing.
- When the processing or chopping of samples results in rapid degradation or loss of residues during storage, a representative sub-sample shall be processed just prior to analysis. The crop unit number, crop unit size, and the number of analyses will determine the amount of sample to process with dry ice for each analysis.

If there is too much sample bulk to add the entire sample all at once and sub-sampling is not an option, process a portion of the sample, add add'l. sample and cryogen (if using), process and repeat until the chopper is full. Bulk bag and repeat processing until the entire sample is chopped. Combine all chopped matrix in the bulk bag, mix well and remove sample for analysis/storage.

Table 1.

Crop Group (Subgroup) Number and Name	Representative Commodities	Pre-Processing Preparation [1]	Processing [2]	Commodities
1. ROOT AND TUBER VEGETABLES	Carrot, potato, radish, and sugar beet.	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine. If greater than 10 pounds cut each unit in half, returning opposite half to sample bag. Continue until all can fit in chopper. If tops are included, cut with an electric knife. A heavy knife and hammer are useful if sample is too hard.	Robot Coupe, Grinder or Hobart with cryogen	Arracacha; arrowroot; artichoke, Chinese; artichoke, Jerusalem; beet, garden; beet, sugar; burdock, edible; canna, edible; carrot; cassava, bitter and sweet; celeriac; chayote (root); chervil, turnip-rooted; chicory; chufa; dasheen (taro); ginger; ginseng; horseradish; leren; parsley, turnip-rooted; parsnip; potato; radish; radish, oriental; rutabaga; salsify; salsify, black; salsify, Spanish; skirret; sweet potato; tanier; turmeric; turnip; yam bean; yam, true.
1A. Root vegetables subgroup	Carrot, radish, and sugar beet	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine. If tops are included, cut with an electric knife. A heavy knife and hammer are useful if sample is too hard.	Robot Coupe, Grinder or Hobart with cryogen	Beet, garden; beet, sugar, burdock, edible; carrot; celeriac; chervil, turnip-rooted; chicory; ginseng; horseradish; parsley, turnip-rooted; parsnip; radish; radish, oriental; rutabaga; salsify; salsify, black; salsify, Spanish; skirret; turnip
1B. Root vegetables (except sugar beet) subgroup	Carrot and radish	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine. If tops are included, cut with an electric knife. A heavy knife and hammer are useful if sample is too hard.	Robot Coupe, Grinder or Hobart with cryogen	Beet, garden; burdock, edible; carrot; celeriac; chervil, turnip-rooted; chicory; ginseng; horseradish; parsley, turnip-rooted; parsnip; radish; radish, oriental; rutabaga; salsify; salsify, black; salsify, Spanish; skirret; turnip.
1C. Tuberous and corm vegetables subgroup	Potato	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine	Robot Coupe, Grinder or Hobart with cryogen	Arracacha; arrowroot; artichoke, Chinese; artichoke, Jerusalem; canna, edible; cassava, bitter and sweet; chayote (root); chufa; dasheen (taro); ginger; leren; potato; sweet potato; tanier; turmeric; yam bean; yam, true
1D. Tuberous and corm vegetables (except potato) subgroup	Sweet potato	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine	Robot Coupe, Grinder or Hobart with cryogen	Arracacha; arrowroot; artichoke, Chinese; artichoke, Jerusalem; canna, edible; cassava, bitter and sweet; chayote (root); chufa; dasheen (taro); ginger; leren; sweet potato; tanier; turmeric; yam bean; yam, true
2. LEAVES OF ROOT AND TUBER VEGETABLES (HUMAN FOOD OR ANIMAL FEED)	Turnip and garden beet or sugar beet	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch or smaller pieces or cut with electric knife and then thoroughly mix to combine.	Robot Coupe, Grinder or Hobart with cryogen. If too much sample bulk to add all at once, process in batches until chopper is full as described in footnote 2.	Beet, garden; beet, sugar; burdock, edible; carrot; cassava, bitter and sweet; celeriac; chervil, turnip-rooted; chicory; dasheen (taro); parsnip; radish; radish, oriental (daikon); rutabaga; salsify, black; sweet potato; tanier; turnip; yam, true
3. BULB VEGETABLES	Onion, green; and onion, dry bulb	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine or cut in ~ 1in pieces	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice)	Garlic; garlic, great-headed; leek; onion, dry bulb and green; onion, Welsh; shallot

[1] and [2] – see footnotes at bottom of page 11

Table 1, cont.

Crop Group (Subgroup) Number and Name	Representative Commodities	Pre-Processing Preparation [1]	Processing [2]	Commodities
4. LEAFY VEGETABLES (EXCEPT BRASSICA VEGETABLES)	Celery, head lettuce, leaf lettuce, and spinach	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch or smaller pieces or cut with electric knife and then thoroughly mix to combine.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice). If too much sample bulk to add all at once, process in batches until chopper is full as described in footnote 2.	Amaranth (Chinese spinach); arugula (roquette); cardoon; celery; celery, Chinese; celtuce; chervil; chrysanthemum, edible-leaved; chrysanthemum, garland; corn salad; cress, garden; cress, upland; dandelion; dock (sorrel); endive (escarole); fennel, Florence; lettuce, head and leaf; orach; parsley; purslane, garden; purslane, winter; radicchio (red chicory); rhubarb; spinach; spinach, New Zealand; spinach, vine; Swiss chard
4A. Leafy greens subgroup	Head lettuce and leaf lettuce, and spinach	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch or smaller pieces or cut with electric knife and then thoroughly mix to combine.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Amaranth; arugula; chervil; chrysanthemum, edible-leaved; chrysanthemum, garland; corn salad; cress, garden; cress, upland; dandelion; dock; endive; lettuce; orach; parsley; purslane, garden; purslane, winter; radicchio; spinach; spinach, New Zealand; spinach, vine
4B. Leaf petioles subgroup	Celery	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Cardoon; celery; celery, Chinese; celtuce; fennel, Florence; rhubarb; Swiss chard
5. BRASSICA (COLE) LEAFY VEGETABLES	Broccoli or cauliflower; cabbage; and mustard greens.	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch or smaller pieces or cut with electric knife and then thoroughly mix to combine. May need to quarter lengthwise, using opposite pieces prior to mixing to reduce bulk.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice). If too much sample bulk to add all at once, process in batches until chopper is full as described in footnote 2.	Broccoli; broccoli, Chinese (gai lon); broccoli raab (rapini); Brussels sprouts; cabbage; cabbage, Chinese (bok choy); cabbage, Chinese (napa); cabbage, Chinese mustard(gai choy); cauliflower; cavalo broccolo; collards; kale; kohlrabi; mizuna; mustard greens; mustard spinach; rape greens
5A.Head & Stem Brassica subgroup	Broccoli or cauliflower and cabbage	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine. May need to quarter lengthwise, using opposite pieces prior to mixing to reduce bulk.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Broccoli; broccoli, Chinese; brussels sprouts; cabbage; cabbage, Chinese (napa); cabbage, Chinese mustard; cauliflower; cavalo broccolo; kohlrabi
5B.Leafy Brassica greens subgroup	Mustard greens	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine or cut with an electric knife.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Broccoli raab; cabbage, Chinese (bok choy); collards; kale; mizuna; mustard greens; mustard spinach; rape greens

Table 1, cont.

Crop Group (Subgroup) Number and Name	Representative Commodities	Pre-Processing Preparation [1]	Processing [2]	Commodities
6. LEGUME VEGETABLES (SUCCULENT OR DRIED)	Bean (Phaseolus),(succulent & dried),pea (Pisum) (succulent & dried) and soybean	Pre-processing not required.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice) For dried peas/beans - grinder type processor, coffee grinder or Robot Coupe	Bean (Lupinus) (includes grain lupin, sweet lupin, white lupin, and white sweet lupin); bean (Phaseolus) (includes field bean, kidney bean, lima bean, navy bean, pinto bean, runner bean, snap bean, tepary bean, wax bean); bean (Vigna) (includes adzuki bean, asparagus bean, blackeyed pea, catjang, Chinese longbean, cowpea, crowder pea, moth bean, mung bean, rice bean, southern pea, urd bean, yardlong bean); broad bean (fava); chickpea (garbanzo); guar; jackbean; lablab bean; lentil; pea (Pisum) (includes dwarf pea, edible-podded pea, English pea, field pea, garden pea, green pea, snowpea, sugar snap pea); pigeon pea; soybean; soybean (immature seed); sword bean
6A.Edible-podded legume vegetables subgroup	Any one succulent cultivar of edible-podded bean (Phaseolus) and any one succulent cultivar of edible-podded pea (Pisum)	Pre-processing not required	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice). For dried peas/beans - grinder type processor, coffee grinder or Robot Coupe	Bean (Phaseolus) (includes runner bean, snap bean, wax bean); bean (Vigna) (includes asparagus bean, Chinese longbean, moth bean, yardlong bean); jackbean; pea (Pisum) (includes dwarf pea, edible-podded pea, snow pea, sugar snap pea); pigeon pea; soybean (immature seed); sword bean
6B.Succulent shelled pea and bean subgroup	Any succulent shelled cultivar of bean (Phaseolus) and garden pea (Pisum)	Pre-processing not required	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice)	Bean (Phaseolus) (includes lima bean, green; broad bean, succulent); bean (Vigna) (includes blackeyed pea, cowpea, southern pea); pea (Pisum) (includes English pea, garden pea, green pea); pigeon pea
6C.Dried shelled pea and bean (except soybean) subgroup	Any one dried cultivar of bean (Phaseolus) and any one dried cultivar of pea (Pisum)	Pre-processing not required	Grinder type processor, coffee grinder or Robot Coupe with cryogen (LN2 or dry ice)	Dried cultivars of bean (Lupinus); bean (Phaseolus) (includes field bean, kidney bean, lima bean (dry), navy bean, pinto bean, tepary bean); bean (Vigna) (includes adzuki bean, blackeyed pea, catjang, cowpea, crowder pea, moth bean, mung bean, rice bean, southern pea, urd bean); broad bean (dry); chickpea; guar; lablab bean; lentil; pea (Pisum) (includes field pea); pigeon pea
7. FOLIAGE OF LEGUME VEGETABLES	Any cultivar of bean (Phaseolus), field pea (Pisum) and soybean	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch or smaller pieces or cut with electric knife and then thoroughly mix to combine.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice)	Plant parts of any legume vegetable included in the legume vegetables that will be used as animal feed.
7A.Foliage of legume vegetables (except soybeans) subgroup	Any cultivar of bean (Phaseolus) and field pea (Pisum)	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch or smaller pieces or cut with electric knife and then thoroughly mix to combine.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice)	Plant parts of any legume vegetable (except soybeans) included in the legume vegetables group that will be used as animal feed.

Table 1, cont.

Crop Group (Subgroup) Number and Name	Representative Commodities	Pre-Processing Preparation [1]	Processing [2]	Commodities
8. FRUITING VEGETABLES (EXCEPT CUCURBITS)	Tomato, bell pepper, and one cultivar of non-bell pepper	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine or chop with a knife.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Eggplant; groundcherry (Physalis spp); pepino; pepper (includes bell pepper, chili pepper, cooking pepper, pimento, sweet pepper); tomatillo; tomato
9. CUCURBIT VEGETABLES	Cucumber, muskmelon, and summer squash	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine. May need to quarter lengthwise, using opposite pieces prior to mixing to reduce bulk. Chop entire fruit including seeds and rind.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Chayote (fruit); Chinese waxgourd (Chinese preserving melon); citron melon; cucumber; gherkin; gourd, edible (includes hyotan, cucuzza, hechima, Chinese okra); Momordica spp (includes balsam apple, balsam pear, bittermelon, Chinese cucumber); muskmelon (includes cantaloupe); pumpkin; squash, summer; squash, winter (includes butternut squash, calabaza, hubbard squash, acorn squash, spaghetti squash); watermelon
9A.Melon subgroup	Cantaloupe	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine. May need to quarter lengthwise, using opposite pieces prior to mixing to reduce bulk. Chop entire fruit including seeds and rind.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Citron melon; muskmelon; watermelon
9B. Squash/Cucumber subgroup	One cultivar of summer squash and cucumber	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine. May need to quarter lengthwise, using opposite pieces prior to mixing to reduce bulk. Chop entire fruit including seeds and rind.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Chayote (fruit); Chinese waxgourd; cucumber; gherkin; gourd, edible; Momordica spp; pumpkin; squash, summer;squash, winter
10. CITRUS FRUITS	Sweet orange, lemon and grapefruit	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Calamondin; citrus citron; citrus hybrids (includes chironja, tangelo, tangor); grapefruit; kumquat; lemon; lime; mandarin (tangerine); orange, sour; orange, sweet; pummelo; Satsuma mandarin
11. POME FRUITS	Apple and pear	While inside IR4 bag and frozen break up with a mallet into approx. 1 to 2 inch pieces and mix to combine. May need to quarter lengthwise, using opposite pieces prior to mixing to reduce bulk. Chop entire fruit including seeds and peel.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Apple; crabapple; loquat; mayhaw; pear; pear, oriental; quince

Table 1, cont.

Crop Group (Subgroup) Number and Name	Representative Commodities	Pre-Processing Preparation [1]	Processing [2]	Commodities
12. STONE FRUITS	Sweet or tart cherry, peach, and plum or fresh prune	Pre-processing not required. May need to be pitted or cut into smaller pieces.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice)	Apricot; cherry, sweet; cherry, tart; nectarine; peach; plum; plum, Chickasaw; plum, Damson; plum, Japanese; plumcot; prune (fresh)
13. BERRIES	Any one blackberry or any one raspberry; and blueberry	Pre-processing typically not required. If larger than 1 to 2 in cut into smaller pieces.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice). A coffee grinder can be used for small sample sizes.	Blackberry (including bingleberry, boysenberry; dewberry; lowberry, marionberry, olallieberry, youngberry); blueberry; currant; elderberry; gooseberry; huckleberry; loganberry; raspberry, black and red
13A.Caneberry (blackberry and raspberry) subgroup	Any one blackberry or any one raspberry	Pre-processing not required	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Blackberry; loganberry; red and black raspberry; cultivars and/or hybrids of these
13B. Bushberry subgroup	Blueberry, highbush	Pre-processing not required	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Blueberry, highbush and lowbush; currant; elderberry; gooseberry; huckleberry
14.TREE NUTS	Almond and pecan	Pre-processing typically not required. Nut meat may need to be separated.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice). A coffee grinder can be used for small sample sizes.	Almond; beech nut; Brazil nut; butternut; cashew; chestnut; chinquapin; filbert (hazelnut); hickory nut; macadamia nut; pecan; walnut, black and English
15. CEREAL GRAINS	Corn (sweet and field), rice, sorghum, and wheat	Pre-processing not required	Wiley mill, coffee grinder or Robot Coupe or with cryogen (LN2 or dry ice).	Barley; buckwheat; corn; millet, pearl; millet, proso; oats; popcorn; rice; rye; sorghum (milo); teosinte; triticale; wheat; wild rice
16.FORAGE, FODDER AND STRAW OF CEREAL GRAINS	Corn, wheat, and any other cereal grain crop	Pre-processing typically not required. Use an electric knife if needed.	Robot Coupe, Grinder or smaller Hobart with cryogen (LN2 or dry ice)	Forage, fodder, and straw of all commodities included in the cereal grains group
17.GRASS FORAGE, FODDER, AND HAY GROUP	Bermuda grass; bluegrass; and bromegrass or fescue	Pre-processing typically not required. Use an electric knife if needed.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice). If too much sample bulk to add all at once, process in batches until chopper is full as described in footnote 2.	Any grass, Gramineae family (either green or cured) except sugarcane and those included in the cereal grains group, that will be fed to or grazed by livestock, all pasture and range grasses and grasses grown for hay or silage

Table 1, cont.

Crop Group (Subgroup) Number and Name	Representative Commodities	Pre-Processing Preparation [1]	Processing [2]	Commodities
18.NONGRASS ANIMAL FEEDS (FORAGE, FODDER, STRAW AND HAY)	Alfalfa and clover (Trifolium)	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Alfalfa; bean, velvet; clover (Trifolium, Melilotus); kudzu; lespedeza; lupin; sainfoin; trefoil; vetch; vetch, crown; vetch, milk
19.HERBS AND SPICES	Basil (fresh & dried); black pepper; chive; hop cones; and celery seed or dill seed	Pre-processing typically not required. Use an electric knife if needed.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice). For hops keep dry ice to a minimum and do not leave hops in chopper too long.	Allspice; angelica; anise; anise, star; annatto (seed); balm; basil; borage; burnet; camomile; caper buds; caraway; caraway, black; cardamom; cassia bark; cassia buds; catnip; celery seed; chervil (dried); chive; chive, Chinese; cinnamon; clary; clove buds; coriander leaf (cilantro or Chinese parsley); coriander seed (cilantro); costmary; culantro (leaf); culantro (seed); cumin; curry (leaf); dill (dillweed); dill (seed); fennel (common); fennel, Florence (seed); fenugreek; grains of paradise, hop cones; horehound; hyssop; juniper berry; lavender; lemongrass; lovage (leaf); lovage (seed); mace; marigold, marjoram; mustard (seed); nasturtium; nutmeg; parsley (dried); pennyroyal; pepper, black; pepper, white; poppy (seed); rosemary; rue; saffron; sage; savory, summer and winter; sweet bay; tansy; tarragon; thyme; vanilla; wintergreen; woodruff; wormwood
19A.Herb subgroup	Basil (fresh & dried) and chive	Pre-processing typically not required. Use an electric knife if needed.	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice). A coffee grinder can be used for small sample sizes.	Angelica; balm; basil; borage; burnet; camomile; catnip; chervil (dried); chive; chive, Chinese; clary; coriander (leaf); costmary; culantro (leaf); curry (leaf); dillweed; horehound; hyssop; lavender; lemongrass; lovage (leaf); marigold; marjoram; nasturtium; parsley (dried); pennyroyal; rosemary; rue; sage; savory, summer and winter; sweet bay; tansy; tarragon; thyme; wintergreen; woodruff; and wormwood
19B.Spice subgroup	Black pepper; and celery seed or dill seed	Pre-processing not required	Wiley mill, coffee grinder or Robot Coupe or with cryogen (LN2 or dry ice).	Allspice; anise (seed); anise, star; annatto (seed); caper (buds); caraway; caraway, black; cardamom; cassia (bark); cassia (buds);celery (seed); cinnamon; clove (buds); coriander (seed); culantro (seed); cumin; dill (seed); fennel, common; fennel, Florence (seed); fenugreek; grains of paradise; juniper (berry); lovage (seed); mace; mustard (seed); nutmeg; pepper, black; pepper, white; poppy (seed); saffron; and vanilla
TROPICAL FRUIT CROPS Grapefruit	grapefruit, punimelo, and their citrus hybrids (including Uniq(Ugli) fruit)	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Corresponds to Codex Citrus Fruits Definitions

Table 1, cont.

Crop Group (Subgroup) Number and Name	Representative Commodities	Pre-Processing Preparation [1]	Processing [2]	Commodities
Sugar Apple	sugar apple, cherimoya, atemoya, custard apple ilama, soursop, biriba	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	All crops in the Annonaceae; similar gross morphology; inedible peel
Lychee	lychee, longan, Spanish lime, rambutan, pulasan	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	All crops in the Sapindaceae; inedible peel
Papaya	papaya, star apple, black sapote, mango, sapodilla, canistel, mamey sapote	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice). Make sure seeds are chopped.	All crops have inedible peel; corresponds to Codex classification
Avocado	avocado, papaya, star apple, black sapote, mango, sapodilla, canistel, mamey sapote	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	All crops have inedible peel; corresponds to Codex classification
Guava	guava, feijoa, jaboticaba, wax jambu, starfruit, passionfruit, acerola	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	Primarily edible peel; note/peel rarely contaminates *Passiflora spp.* during juicing
Citrus Fruits	add White sapote (Casimiroa), and other cultivars and/or hybrids of these	While inside IR4 bag and frozen break up with a mallet into approx. 2 inch pieces and mix to combine	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice).	White sapote is in the Rutaceae (citrus)

1. Typical pre-processing tools include, but are not limited to: mallet, hammer, hatchet, cleaver, heavy knife, ginzu type knife, scissors, electric knife, and paper cutter. Caution must be taken when attempting to break samples with mallets while in the IR-4 bags. The sample bag may break. A secondary bag may be used to contain the pieces. Be aware that there may be a possibility of sample contamination with slivers of the bag/plastic lining. Alternatively, break-up of difficult frozen items using a heavy bladed knife, cleaver or heavy hammer/ mallets (2.5-4lb) may be done on a chopping board lined with butcher paper with the edges folded up to contain sample pieces. Care must be exercised when using metal knives, choppers or hammers that pieces do not cause personal injury in the event of breakage.
2. Use of serrated S-blades will improve chopping efficiency of Robot Coupe Systems when processing fibrous and hard sample matrices including green coffee bean, roasted coffee beans, and lychee whole fruit (with seed). Use of the Pulse or High speed (~3600 rpm) option for variable speed models is recommended for these difficult frozen matrices. A coffee grinder is useful for dry seeded samples. If there is too much sample bulk to add the entire sample all at once and sub-sampling is not an option, process a portion of the sample, add add'l. sample and cryogen (if using), process and repeat until chopper is full. Bulk bag and repeat processing until entire sample is chopped. Combine all chopped matrix in bulk bag, mix well and remove sample for analysis/storage.

Appendix 1: From Pesticide Assessment Manual (PAM) Volume 1, 3rd Edition

Table 102-a: Portion of Raw Agricultural Commodity to be Analyzed for Pesticide Residues

Root and tuber vegetables group[1]	Where separate tolerances are established for root or tuber, analyze whole commodity after removing adhering soil by lightly rinsing in running water. Where a tolerance is established on a root vegetable including tops and/or with tops, and tops and roots are marketed together, analyze tops and roots separately. Neither the pesticide residue on the roots nor the pesticide residue on the tops shall exceed the tolerance level. For carrots, parsnips, and rutabagas, remove and discard tops.
Bulb vegetables (green or dry) group	Whole commodity after removing and discarding roots. Remove adhering soil by lightly rinsing in running water. In the case of dry bulb onions and garlic, remove and discard stems and outer sheaths (husk or parchment skin) that are easily removed.
Leafy vegetables (except Brassica vegetables) group	Whole commodity after removing and discarding obviously decomposed or withered leaves. In the case of rhubarb, analyze only the stem without leaves. Remove adhering soil from celery by lightly rinsing in running water.
Brassica (cole) leafy vegetables group	Whole commodity after removing and discarding obviously decomposed or withered leaves, except remove and discard all leaves from cauliflower and headed broccoli and use sprouts only from brussels sprouts.
Legume vegetables (succulent or dried) group	Whole commodity, including pods for succulent and without pods for dry.
Fruiting vegetables (except cucurbits) group	Whole commodity after removing and discarding stems and husks.
Cucurbit vegetables group	Whole commodity after removing and discarding stems.
Citrus fruits group	Whole commodity.
Pome fruits group	Whole commodity after removing and discarding stems.
Stone fruits group	Whole commodity after removing and discarding stems and stones.
Small fruits and berries group	Whole commodity after removing and discarding caps and stems, except for currants, where the stems are to be included.

[1] Members of food groups are listed in 40 CFR 180.34 (f) (9).

Appendix 1 (con't)

Peanuts	Whole peanut meat (kernel) after removing hulls.
Peanut hulls	Whole commodity after removing peanut meat.
Dates and olives	Whole commodity after removing and discarding stems and stones or pits.
Pineapples	Whole commodity after removing and discarding crowns (leaves at top of fruit).
Avocados and mangoes	Whole commodity after removing and discarding stones.
Bananas	Whole commodity including peel after removing and discarding crown tissue and stalk.
Miscellaneous raw fruits and vegetables not previously included	Whole commodity after removing and discarding obviously decomposed or withered leaves, stems, stones or pits, shells or husks; if commodity has adhering amounts of soil, remove by lightly rinsing in running water.
Almond hulls	Whole commodity after removing shell and nutmeat.
Cereal grains group	Whole commodity (grain) except for fresh corn (including sweet corn). Include kernels plus cob after removing and discarding husk.
Eggs	Whole commodity after removing and discarding shells.
Fish	Edible portion of the commodity after removing and discarding heads, tails, scales, fins, viscera, bones (if inedible), and skin (if inedible).
Crab (hard shell)	Edible portion of commodity after removing and discarding shells, gills, and viscera.
Crab (soft shell)	Edible portion of commodity after removing and discarding gills.
Shrimp and crayfish	Edible portion of commodity after removing and discarding heads, shells, and inedible tails of shrimp.
Lobster	Edible portion of commodity including tomalley (liver) after removing and discarding shells and stomachs (hard sac near head).
Oyster, clam, and other shellfish	Edible portion of commodity including the liquor, after removing and discarding shells.
Rabbits and other game	Edible portion of commodity after removing and discarding bones.

References Cited:

1. 40 CFR 180.1 —Tolerances And Exemptions From Tolerances For Pesticide Chemicals In Food. Subpart A(j) —Definitions and Interpretative Regulations

2. Codex ‘‘Guidelines on Minimum Sample Sizes for Agricultural Commodities from Supervised Field Trials for Residue Analysis’’, ALINORM 87/24A (1987)

3. Codex Alimentarius Volume 2 Pesticides Residues In Food Section 2 Codex Classification Of Foods And Animal Feedstuffs. FAO, Rome 1993

4. Pesticide Assessment Manual (PAM) Volume 1, 3rd Edition, Section 102 and Section 203.

5. Residue Chemistry Test Guidelines OPPTS 860.1500 Crop Field Trials

Attachment 2

Sample Analytical Summary Report

ANALYTICAL SUMMARY REPORT

Flonicamid: Magnitude of the Residue on Mint

PR# 09358

Authors

Susan Erhardt, Eina Abouzied and Lester Geissel

Laboratory Research Director

Susan Erhardt

Testing Facility

IR-4 North Central Region Laboratory
Michigan State University
3815 Technology Boulevard, Suite 1031B
Lansing, MI 48910-8396

Laboratory Study Identification Number

09358.11-MIR05

Study Director

Kenneth S. Samoil

Sponsor

IR-4 Project Headquarters
500 College Road East, Suite 201W
Princeton, New Jersey 08540

Field Study Identification Number(s)

09358.11-WI17 09358.11-WI18 09358.11-WA*18

09358.11-ID12 09358.11-WA17

Study Timetable

Study Initiation Date: 26-Jan-2011
Experimental Termination Date: 8-Aug-2012

Report Date

19-Sep-2012

GLP COMPLIANCE STATEMENT

PR#: 09358 Lab ID#: 09358.11-MIR05

The study reported herein for the residues of Flonicamid and its metabolites, TFNA-AM, TFNG and TFNA on Mint was conducted and reported in compliance with the Good Laboratory Practices (GLP) Regulations Title 40, Part 160 of the Code of Federal Regulations of the United States of America.

Signature/Date
Susan Erhardt
Laboratory Research Director

IR-4 North Central Region Laboratory
Michigan State University
3900 Collins Road, Suite 1031B
Lansing, MI 48910-8396
Tel. No.: (517) 336-4653

Signature/Date
Eina Abouzied
Analyst

IR-4 North Central Region Laboratory
Michigan State University
3900 Collins Road, Suite 1031B
Lansing, MI 48910-8396
Tel. No.: (517) 336-4648

Signature/Date
Lester Geissel
Analyst

IR-4 North Central Region Laboratory
Michigan State University
3900 Collins Road, Suite 1031B
Lansing, MI 48910-8396
Tel. No.: (517) 336-4655

QUALITY ASSURANCE STATEMENT

LABORATORY PERSONNEL

Name	Designation
Robert Hollingworth	IR-4 North Central Region Director
Susan Erhardt	Laboratory Research Director
Robert T. Kon	Sample Control Officer, Archivist (Retired 7 May 2012)
Eina Abouzied	Principal analyst
Lester D. Geissel	Analyst
Mathew J. Witmer	Student aide (sample grinding, glassware cleaning)
Royal G. Fader	Laboratory aide (sample grinding, glassware cleaning)

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ARCHIVES
(Location of Raw Data)

Original raw data, a certified copy of the signed protocol, amendments, correspondence, a true copy of the analytical summary report and relevant facility related information for the study titled: "Flonicamid: Magnitude of the Residue on Mint PR# 09358" have been placed in the archives of the testing facility. The original of this Analytical Summary Report (ASR) has been forwarded to the sponsor.

Portions of the field samples will be retained at the testing facility in freezer at less than -20 °C for at least 12 months after submission of this Analytical Summary Report (ASR). The long term storage stability samples will be stored for at least 5 years at < -20 °C. The Study Director (SD) will be consulted before the field samples or storage stability samples are discarded.

Laboratory Research Director (LRD) and Laboratory Archivist	Susan Erhardt
Testing Facility:	IR-4 North Central Region Laboratory Michigan State University 3815 Technology Boulevard, Suite 1031B Lansing, MI 48910-8396

IR-4 NATIONAL PESTICIDE CLEARANCE RESEARCH PROGRAM ANALYTICAL SUMMARY REPORT PR# 09358: Flonicamid/Mint

I. Objective/Introduction

At the request of IR-4 Headquarters, the North Central Region Leader Laboratory at Michigan State University (MSU) has assayed Mint for residues of Flonicamid (EPA Reg. No. 71512-10-279, CAS# 158062-67-0) to provide data to support the establishment of a pesticide tolerance on mint tops (leaves & Stems) and mint oil matrices. The working method used for this study was an adaptation of two FMC Corporation Reports. Tops: based on the reference method "Analytical Methodology for IKI-220 (F1785) and its Major Metabolites in/on Peach, Potato Tuber, and Wheat Straw", was written by Audrey W. Chen, Ph. D., Report Number P-3561M, FMC Corporation, Agricultural Products Group, Princeton, NJ on August 28, 2002. For mint oil the method was based on " Magnitude of the Residues of IKI-220 on Cotton-USA in 2001", was written by Karen D.Dow, Report Number P-3567, FMC Corporation, Agricultural Products Group, Princeton, NJ, 08543, USA on December 18, 2002.

The working method (Working Outline WO 9.166 v.1) diverges from the reference methods as shown in Section V and Attachment D of this report. The study followed IR-4 National Pesticide Clearance Laboratory Phase Protocol PR# 09358 as amended. The validated method sensitivity (Lowest Level of Method Validation, LLMV) was 0.02 µg/gm of Flonicamid (IKI-220), its metabolites TFNA-AM, TFNG and TFNA on mint tops (leaves & stems) and mint oil matrices.

II. Sample Inventory and History

Receiving: Upon arrival at the laboratory, samples were inspected and checked against the enclosed shipping forms.

Unique laboratory sample numbers: Unique laboratory sample numbers were assigned as listed in Table I. Sample Inventory.

Grinding: Samples were stored frozen once received from the field. The raw agricultural commodity (RAC) samples (mint tops) were ground using a Robot Coupe RSI 10 B with dry ice. Untreated control samples were chopped first followed by treated samples. The entire sample was chopped and homogenized. Chopped samples were stored in labeled glass jars and placed in a freezer. The mint oil samples received no further processing and were analyzed as received.

Storage condition: All field samples were stored frozen (generally <-20 °C) until analyzed.

Table I. Sample Inventory

Field Trial (FRD)	Crop Fraction	Field Rep No.	Unique Lab Sample No.	Date		
				Sampled	Lab Receipt	Subsampled
11-WI17 (Dr. Scott Chapman)	Tops (Leaves &Stems)	A	18472	09-Aug-2011	31-Aug-2011	21-Mar-2012
		B	18473			
		C	18474			23-Mar-2012
		D	18475			
	Oil	G	18476			29-Mar-2012
		H	18477			
11-WI18 (Dr. Scott Chapman)	Tops (Leaves &Stems)	A	18478	16-Aug-2011	31-Aug-2011	22-Mar-2012
		B	18479			
		C	18480			23-Mar-2012
		D	18481			
11-WA*18 (John Harvey)	Tops (Leaves &Stems)	A	18466	28-Jul-2011	31-Aug-2011	21-Mar-2012
		B	18467			
		C	18468			26-Mar-2012
		D	18469			
	Oil	G	18470			08-Sep-2011
		H	18471			29-Mar-2012
11-ID12 (Will Meeks)	Tops (Leaves &Stems)	A	18352	18-Jul-2011	28-Jul-2011	04-Aug-2011
		B	18353			05-Aug-2011
		C	18354			
		D	18355			
11-WA17 (Dan Groenendale)	Tops (Leaves &Stems)	A	18462	28-Jul-2011	31-Aug-2011	20-Mar-2012
		B	18463			21-Mar-2012
		C	18464			26-Mar-2012
		D	18465			

III. Preparation of Storage Stability Samples

Storage Stability (SS) samples were prepared by taking a known amount of processed control sample and adding a known amount of Flonicamid and metabolites TFNA-AM, TFNG and TFNA as an analytical standard solution. The SS samples were stored under the same storage conditions as field samples (generally < -20°C). The preparation of the storage stability samples is provided in the following Table II.

Table II. Preparation of Storage Stability Samples for Mint Tops (Leaves & Steams) and Mint Oil

Field Trial	Field Rep No.	Crop Part	No. Pre-pared	Sample Size (g)	Standard Used			Fortification		Date Prepared
					Std #	Conc. (µg/mL)	Vol added (mL)	Amount (µg)	Level (ppm)	
11-ID12	A	Tops (leaves & Stems)	9	5.00	F307G-2	~1.00 each	1.0	~1.0 each	~0.2	10-Aug-11
11-WA*18	G	Oil	9	5.00	F307G-2	~1.00 each	1.0	~1.0 each	~0.2	08-Sep-11

IV. Preparation of Standards

Reference standards were received from ISK Biosciences Corporation. Specific purity and additional information regarding the reference standards are shown in Table III. These were prepared as a stock standard solution from the pure reference material Flonicamid (IKI-220) and metabolites (TFNA-AM, TFNG, and TFNA). Two separate lines of working standard solutions were diluted from the stock solution to produce "One Stock Two Lines". One line was for spiking or fortification, marked as "F" and the other used as calibration solutions for the quantification of the residues, marked as "A".

Table III. Reference Standard Information

Analyte:	Flonicamid (IKI-220)	TFNA-AM	TFNG	TFNA
ARS #: (Assigned by IR-4)	307	308	309	310
CAS no.:	158062-67-0	158062-71-6 158062-21-6	---	158063-66-2
Source:	Midwest Research Institute (MRI)		Harlan Laboratories	Midwest Research Institute (MRI)
Lot #:	9803	0006	0006-1	0006
Purity:	99.9± 0.1%	99.87% 99.7% (recertified)	92.4%	100.00%
Receipt Date:	Mar 17, 2010	Mar 17, 2010	Mar 17, 2010	Mar 17, 2010
Expiration Date:	May 15, 2014	Jan 06, 2011 Jan 03, 2016 (recertified)	Dec 2013	Jan 05, 2011 Jan 03, 2016 (recertified)
Storage:	IR-4 Freezer No. 2 (Typically <-10°C)			

Primary Stock Solutions:
A known amount of the standard was accurately weighed into a volumetric flask, dissolved and diluted to a known volume with acetonitrile (Table IV).

Table IV. Primary Stock Solutions

Compounds (ARS ID #)	Standard Weight (g)	Corrected Weight* (g)	Solvent Volume (mL)	Concentration µg/mL	Preparation Date	Solution ID
Flonicamid, (307)	0.01007	0.01006	100	100.6	16-Jul-2010	S307-1
TFNA-AM (308)	0.01048	0.01047	100	104.7	16-Jul-2010	S308-1
TFNG (309)	0.01079	0.00997	100	99.7	19-Jul-2010	S309-1A
TFNA (310)	0.01011	0.01011	100	101.1	16-Jul-2010	S310-1
Flonicamid, (307)	0.01011	0.01010	100	101.0	18-Jan-12	S307-4
TFNA-AM (308)	0.01014	0.01012	100	101.2	9-Aug-11	S308-4
TFNG (309)	0.01034	0.00955	100	95.5	18-Jan-12	S309-4
TFNA (310)	0.01001	0.01001	100	100.1	9-Aug-11	S310-4

*Correction factor based on % purity of the standard.

Fortification Standard Solutions:
Aliquots of primary stock solution were diluted or serially diluted with acetonitrile in volumetric flasks. All fortification standards were labeled with an F for identification for use for fortifying method validation or QC samples. Equivalent sample concentration as shown in Table V below is based on adding a 1 mL aliquot of fortification stock solution to 5 gms of sample matrix.

Table V. Preparation of Fortification Solutions

Source Solution				Fortification Standards			
Solution ID	**Concen-tration (µg/mL)**	**Aliquot Volume (mL)**	**Final Volume**	**Concen-tration (µg/mL)**	**Equivalent* Sample Concen-tration (ppm)**	**Solution ID**	**Preparation Date**
S307-1 S308-1 S309-1A S310-1	100.6 104.7 99.7 101.1	1 each	100	1.006 1.047 0.997 1.011	-	F307G-1	19-Jul-10
S307-4 S308-4 S309-4 S310-4	101.0 101.2 95.5 100.1	5 each	100	5.050 5.060 4.775 5.005	-	F307G-4	19-Jan-12
F307G-4	5.050 5.060 4.775 5.005	10	100	0.5050 0.5060 0.4775 0.5005	-	F307G-7	25-Apr-12
F307G-7	0.5050 0.5060 0.4775 0.5005	10	50	0.1010 0.1012 0.0955 0.1001	0.02	F307G-8	25-Apr-12
S307-4 S308-4 S309-4 S310-4	101.0 101.2 95.5 100.1	5 each	50	10.10 10.12 9.55 10.01	2.0	F307G-9	18-May-12
F307G-9	10.10 10.12 9.55 10.01	10	100	1.010 1.012 0.955 1.001	0.2	F307G-10	18-May-12

*assumes 1mL fortification solution to 5 g sample matrix.

HPLC Calibration Solutions

The working solution was prepared by pipetting an accurate amount of the stock solution into an appropriate volumetric flask and then diluting to the mark with acetonitrile. Subsequent working standards were prepared by pipetting a known amount of a working solutions and diluting with 50% acetonitrile in (HPLC) water. These working solutions, marked as “A”, were for calibration use only (Table VI).

Table VI. Preparation of Calibration Solutions

Source Solution				Calibration Standards		
Solution ID	**Concen-tration (µg/mL)**	**Aliquot Vol-ume (mL)**	**Final Vol-ume (mL)**	**Concen-tration (µg/mL)**	**Solution ID**	**Preparation Date**
Flonicamid (S307-4) TFNA-AM (S308-4) TFNG (S309-4) TFNA (S310-4)	101.0 101.2 95.50 100.1	1 each	100	1.010 1.012 0.9550 1.001	A307G-11	Jan-19-12
A307G-11	1.010 1.012 0.9550 1.001	1	10	0.1010 0.1012 0.0955 0.1001	A307G-20	May-22-12
A307G-11	1.010 1.012 0.9550 1.001	1	100	0.01010 0.01012 0.00955 0.01001	A307G-21	May-22-12
A307G-20	0.1010 0.1012 0.0955 0.1001	2.5	50	0.00505 0.00506 0.004775 0.005005	A307G-22	May-22-12
A307G-21	0.01010 0.01012 0.00955 0.01001	10	50	0.002020 0.002024 0.001910 0.002002	A307G-23	May-22-12
A307G-21	0.01010 0.01012 0.00955 0.01001	5	50	0.001010 0.001012 0.000955 0.001001	A307G-24	May-22-12
A307G-21	0.01010 0.01012 0.00955 0.01001	2.5	50	0.0005050 0.0005060 0.0004775 0.0005005	A307G-25	May-22-12
A307G-21	0.01010 0.01012 0.00955 0.01001	1	50	0.0002020 0.0002024 0.0001910 0.0002002	A307G-26	May-22-12
A307G-21	0.01010 0.01012 0.00955 0.01001	0.5	50	0.0001010 0.0001012 0.0000955 0.0001001	A307G-27	May-22-12

Storage Conditions of Standards:
When not in use, standards and standard solutions were stored in lab freezer units #2 and #7. During the period of this study, the average daily temperature was generally <-10 °C.

V. Analytical Procedure

For mint tops, five (5.0) g of sample was extracted using an extraction solvent (acetonitrile: water, 50:50, v/v). The sample was shaken, decanted into a flat bottom boiling flask. For mint oil, 2.5 g of sample was partitioned twice against hexane and the extraction solvent (acetonitrile:water, 50:50, v/v). The extraction solvent from the mint oil was combined in a flat bottom boiling flask. For both tops and oil the extraction solvent was evaporated to its aqueous remainder, filtered, acidified and made up to 50 mL. A portion of the extract (5 out of 50 mL) was taken through the rest of procedure that includes partitioning with ethyl acetate, evaporation of the ethyl acetate to dryness using N-EVAP Evaporator. The residues were dissolved in actonitrile:water, 50:50, v/v and then analyzed by HPLC/MS/MS.

Method

This working outline is an adaptation of two reference methods. The referenced methods used for development of the working method were "Analytical Methodology for IKI-220 (F1785) and its Major Metabolites in/on Peach, Potato Tuber, and Wheat Straw", was written by Audrey W. Chen on August 28, 2002, Princeton NJ, 08543, USA and "Magnitude of the Residues of IKI-220 on Cotton- USA in 2001" P-3567, Karen Dow, RMC Corporation, 2002.

Points where the working methods (Working Outline WO 9.166, V.1) diverge from the reference methods are noted in WO 9.166 V.1 (Attachment D) and below.

Modifications to Cited Method

A rotovap was used to concentrate the samples with the water bath, 45 °C) instead of a TurboVap (step 8) due available equipment in the lab. To facilitate this, the extract was decanted into 250 mL flat bottom boiling flask instead of TurboVap vessel as in steps 5 and 7. Also, 0.6 mL of concentrated HCI was used instead of 0.5 mL to improve recoveries.

A smaller size of Whatman No. 1 filter was used, 7 cm instead of 11 cm (Step 13) to accommodate existing laboratory equipment.

In step 14, 5 mL of sample extract was used instead of 2 mL to increase the final concentration of residues in the extract due to differences in sensitivity between instrumentation. Because of the increased amount of sample extract, three partitions (4 mL, 4 mL and 2 mL) of ethyl acetate instead of two partitions with 2 mL ethyl acetate were conducted in order to increase efficiency of extraction.

The temperature of the water bath was reduced in Step 16 from 45 °C to 25 °C to increase % recoveries.

The sample final volume was prepared in 50% acetonitrile instead of 30% acetonitrile due to optimization of the chromatography for the existing system.

For the UPLC mobile phase, 0.3% acetic acid in HPLC water and acetonitrile was used instead of 0.2% acetic acid in acetonitrile, 0.2% acetic acid and methanol in the original method. This original separation required the use of three channels. The LC pump in use only has two channels.

Calibration standards were prepared in 50% acetonitrile for compatibility with the optimized chromatography system.

VI. Quantitation

Calculations for residues used the peak area data collected from the instrument. Steps of calculating residues and spike recovery percentages are given in this section. A linear standard calibration curve is used:

$$y = \mathrm{m}\,x + \mathrm{b} \qquad \text{(Eq. 1)}$$

where y is peak area, x is residue concentration, m is slope and b is y-intercept. The residue concentration (x) can be calculated after Eq. 1 is rearranged to Eq. 2.

$$x = \frac{y - \mathrm{b}}{\mathrm{m}} \qquad \text{(Eq. 2)}$$

Spike recovery can be calculated by Eq. 3:

$$\text{Spike recovery}\,(\%) = \frac{x}{s} \cdot 100 \qquad \text{(Eq. 3)}$$

where x is determined residue concentration and s is spike concentration.

An example of calculating residue and spike recovery is given as follows.

Field trial ID:	11-WA*18
Crop fraction:	Mint Tops (Leaves&Stems)
Analysis for:	Flonicamid
ASR page No.:	91
Lab sample ID number:	18467A-QC-2.0-1
Extraction date:	26-Jul-2012
Analysis date:	30-Jul-2012

Analysis results:

Peak area of sample	y = 3026
Calibration coefficient	r^2 = 0.9960
Slope	m = 1780961
Intercept	b = -128.36

Thus, concentration (x') in final extract is,

$$x' = \left(\frac{3026 - (-128.36)}{1780961}\right) = 0.0017712 \text{ (µg/mL in the final extract)}$$

To calculate sample concentration, *x*, initial sample weight and final extract volume are taken into account (initial sample weight, w = 5.0 g, final extract volume, Vol = 5000 mL). It is calculated as follows:

$$x = x' \cdot \frac{\text{Final volume (Vol)}}{\text{Sample weight (w)}} = 0.0017712 \text{ µg/mL} \times \frac{5000\,\text{mL}}{5.0\,\text{g}} = 1.771 \text{ µg/g (in sample)}$$

Spike concentration, *s* = 10.10µg ÷ 5.0 g = 2.020 µg/g. The spike recovery is calculated as follows:

$$\text{Spike recovery (\%)} = \frac{\text{Determined residue } (x)}{\text{Spike concentration } (s)} \times 100$$

$$= \frac{1.771\,\mu\text{g/g}}{2.020\,\mu\text{g/g}} \times 100$$

$$= 87.68\%$$

\\

For some treated samples, the chromatograms of metabolite TFNG have been manually integrated due to a co elution interference and poor peak selection by the chromatography package. Manually integrated chromatograms have been identified on each of the chromatography pages.

VII. Results and Discussion

Residue results are reported as *ppm* (parts per million or µg/g) in this study. The results are summarized in the following tables and below.

Table VII.	Summary of Flonicamid Spike Recoveries (MV, CR, SSCR) for Mint Tops (Leaves & Stems)
Table VIII.	Summary of TFNA-AM Spike Recoveries (MV, CR, SSCR) for Mint Tops (Leaves & Stems)
Table IX.	Summary of TFNG Spike Recoveries (MV, CR, SSCR) for Mint Tops (Leaves & Stems)
Table X.	Summary of TFNA Spike Recoveries (MV, CR, SSCR) for Mint Tops (Leaves & Stems)
Table XI.	Summary of Flonicamid Spike Recoveries (MV, CR, SSCR) for Mint Oil
Table XII.	Summary of TFNA-AM Spike Recoveries (MV, CR, SSCR) for Mint Oil
Table XIII.	Summary of TFNG Spike Recoveries (MV, CR, SSCR) for Mint Oil
Table XIV.	Summary of TFNA Spike Recoveries (MV, CR, SSCR) for Mint Oil
Table XV.	Storage Stability (SS) of Flonicamid and its Metabolites on Mint Tops (Leaves & Stems)

Table XVI. Storage Stability (SS) of Flonicamid and its Metabolites on Mint Oil

Table XVII. Summary of Residue Data for Mint Tops (Leaves & Stems) and Mint Oil Samples for Flonicamid (IKI-220) and Metabolites TFNA-AM, TFNG and TFNA

Detailed calculations and related calibration data as well as LOQ and LOD calculations are given in Attachment B.

The working method was validated at three concentrations, 0.02, 0.2 and 2.0 µg/g or ppm for Flonicamid and its metabolites TFNA-AM, TFNA, and TFNG on Mint Tops (Leaves & Stems). Recoveries are shown in Tables VII, through X for each compound on mint tops. Average recoveries for the parent Flonicamid on mint tops ranged from a minimum of 67 ± 6 % at 0.02 ppm to a maximum of 80 ± 7% at 2.0 ppm (Table VII). Average recoveries for TFNA-AM ranged from 75 ± 5% at 0.02 ppm to 86 ± 4% at 0.2 ppm (Table VIII). For TFNG average recoveries ranged from 91 ± 5% at 2 ppm to a maximum of 111 ± 3% at 0.2 ppm (Table IX). Finally, recoveries for TFNA ranged from 98± 6% at 2 ppm to a maximum of 105 ± 3 at 0.2 ppm (Table X). Calculated LODs for Flonicamid, and its metabolites TFNA-AM, TFNG and TFNA were 0.004, 0.003, 0.002 and 0.004 µg/g respectively. While calculated LOQs for Flonicamid, and its metabolites TFNA-AM, TFNG and TFNA were 0.013, 0.010, 0.006 and 0.013µg/g, respectively. Calculations were based on the recoeveries at the LLMV using a one-tailed‘t’ statistic as described in The Handbook of Environmental Analysis, 4th Edition, by Roy-Keith Smith, Genium Publishing Corporation, 1999. These calculated values support the lowest level of quantification used as a part of this study as they are less than the LLMV of 0.02 ppm. The calculation spreadsheets used for generating calculated LOD and LOQs are shown in Attachment B.

The working method also was validated on mint oil at 0.02, 0.2 and 2.0 µg/gm. Recoveries are shown in Tables XI through XIV. Average recoveries for Flonicamid ranged from 87 ± 4 at 2 ppm to 96 ±7 at 0.02 ppm (Table XI). Average recoveries for TFNA-AM ranged from 87 ± 6% at 0.2 ppm to 96 ± 8 at 0.02 ppm (Table XII). For TFNG, average recoveries ranged from 88 ± 7% at 0.02 ppm to a maximum of 94 at both 0.2 and 2 ppm (Table XIII). Finally, recoveries for TFNA ranged from 89± 6% at 2 ppm to a maximum of 93 ± 4 at 0.2 ppm (Table XIV). Calculated LODs for Flonicamid, and its metabolites TFNA-AM, TFNG and TFNA on mint oil were 0.005, 0.006, 0.004 and 0.004 µg/g respectively. While calculated LOQs for Flonicamid, and its metabolites TFNA-AM, TFNG and TFNA were 0.014, 0.017, 0.013 and 0.012µg/g, respectively. Calculations were based on the LLMV using a one-tailed‘t’ statistic as described in The Handbook of Environmental Analysis, 4th Edition, by Roy-Keith Smith, Genium Publishing Corporation, 1999. These calculated values support the lowest level of quantification used as a part of this study as they are less than the LLMV of 0.02 ppm. The calculation spreadsheets used for generating calculated LOD and LOQs are shown in Attachment B.

Mint tops and mint oil storage stability samples were spiked with Flonicamid, and its metabolites TFNA-AM, TFNA and TFNG at 0.20 ppm for each compound. For mint tops, samples were stored for 372 days prior to analysis. Recoveries were 85 ± 3, 79 ± 4, 81 ± 2, and 73 ± 7 for Flonicamid, TFNA-AM, TFNG and TFNA, respectively. These results demonstrate that Flonicamid and its metabolites are stable under the storage conditions used for the treated samples (Table XV).

For mint oil storage stability, samples were stored 368 days prior to analysis. Samples were prepared using 5 g of mint oil rather than the 2.5 g used in the working method. The concurrent recovery used in the storage stability test was prepared with 5 g of oil to compensate for the difference. In addition, volumes were adjusted relative to the increased sample size throughout the methodology on the entire set of storage stability mint oil samples. Recoveries were 44 ± 2, 50 ± 4, 44 ± 2, and 45 ± 73for Flonicamid, TFNA-AM, TFNG and TFNA, respectively. While it appears that the low recoveries were due to an inaccurate spiking was the samples, there is no data from the storage stability preparation that would support this hypothesis. Concurrent recoveries for both sets were within 70 to 120% as designated by protocol.

The untreated control samples of both the mint tops and oil had no detectable residues of Flonicamid, or its metabolites TFNA-AM, TFNG and TFNA at greater than the LLMV (i.e., < 0.02 ppm) (Table XVII). On mint tops, residues of Flonicamid varied on treated samples from a minimum of 0.500 ppm on replicate C of trial 11-WI17 to a maximum of 2.41 ppm on replicate D from field trial 11-ID12. Residues for TFNA-AM on mint tops ranged from 0.0723 ppm on replicate C from trial 11-WI17 to a maximum of 0.254 ppm on replicate D from trial 11-WA17. For TFNG, residues ranged from 0.219 ppm replicate D from trial 11-WI17to a maximum of 0.461 ppm for sample D from trial 11-WA17. Residues of TFNA ranged from 0.107 ppm replicate C from trial 11-WI17on treated samples from to a maximum of 0.235 ppm on replicate D of trial 11-WA17.

Results from the mint oil analyses show that residues of Flonicamid and its metabolites TFNA-AM, TFNG and TFNA were less than the LLMV of 0.02ppm.

Table VII. Summary of Flonicamid Spike Recoveries (MV, CR, SSCR) for Mint Tops (Leaves & Stems)

Spike level (ppm)	Lab sample ID	Type of recovery	Determined value (ppm)	Average ± s.d. (ppm)	Spike recovery (%)	Average recovery (%)
Flonicamid (IKI-220)						
0.0202	18472A-MV-0.02-1	MV	0.0149	0.0135 ± 0.0012	74	67 ± 6
	18472A-MV-0.02-2	MV	0.0136		67	
	18472A-MV-0.02-3	MV	0.0143		71	
	18473A-QC-0.02-1	CR	0.0119		59	
	18353A-QC-0.02-1	CR	0.0144*		71	
	18463A-QC-0.02-1	CR	0.0121		60	
0.202	18472A-MV-0.2-1	MV	0.155	0.160 ± 0.0049	77	79 ± 2
	18472A-MV-0.2-2	MV	0.166		82	
	18472A-MV-0.2-3	MV	0.158		78	
	18352A-QC-0.2-1	SSCR	0.163		81	
2.02	18472A -MV-2-1	MV	1.48	1.62 ± 0.13	73	80 ± 7
	18472A -MV-2-2	MV	1.53		76	
	18472A -MV-2-3	MV	1.71		84	
	18467A-QC-2-1	CR	1.75		86	
3.0	18479A-QC-3-1	CR	2.42	---	80	---

*Average of four injections

Notes:

s.d. = standard deviation

MV = Method Validation

CR = Concurrent spike recovery

All concurrent recovery (CR) samples were analyzed twice (double injection) and the values are averages of multiple analysis.

Table VIII. Summary of TFNA-AM Spike Recoveries (MV, CR, SSCR) for Mint Tops (Leaves & Stems)

Spike level (ppm)	Lab sample ID	Type of recovery	Determined value (ppm)	Average ± s.d. (ppm)	Spike recovery (%)	Average recovery (%)
TFNA-AM						
0.02024	18472A-MV-0.02-1	MV	0.0149	0.0151 ± 0.0092	74	75 ± 5
	18472A-MV-0.02-2	MV	0.0145		72	
	18472A-MV-0.02-3	MV	0.0141		70	
	18473A-QC-0.02-1	CR	0.0146		72	
	18353A-QC-0.02-1	CR	0.0163*		81	
	18463A-QC-0.02-1	CR	0.0162		80	
0.2024	18472A-MV-0.2-1	MV	0.182	0.173 ± 0.0075	90	86 ± 4
	18472A-MV-0.2-2	MV	0.172		85	
	18472A-MV-0.2-3	MV	0.175		86	
	18352A-QC-0.2-1	SSCR	0.164		81	
2.024	18472A -MV-2-1	MV	1.49	1.66 ± 0.12	73	82 ± 7
	18472A -MV-2-2	MV	1.69		83	
	18472A -MV-2-3	MV	1.77		87	
	18467A-QC-2-1	CR	1.71		84	
3.0	18479A-QC-3-1	CR	2.44	---	80	---

*average of 4 injections

Notes:

s.d. = standard deviation

MV = Method Validation

CR = Concurrent spike recovery

All concurrent recovery (CR) samples were analyzed twice (double injection) and the values are averages of multiple analysis.

Table IX. Summary of TFNG Spike Recoveries (MV, CR, SSCR) for Mint Tops (Leaves & Stems)

Spike level (ppm)	Lab sample ID	Type of recovery	Determined value (ppm)	Average ± s.d. (ppm)	Spike recovery (%)	Average recovery (%)
TFNG						
0.0191	18472A-MV-0.02-1	MV	0.0209	0.0212 ± 0.0006	110	111 ± 3
	18472A-MV-0.02-2	MV	0.0205		107	
	18472A-MV-0.02-3	MV	0.0215		113	
	18473A-QC-0.02-1	CR	0.0221		115	
	18353A-QC-0.02-1	CR	0.0207*		108	
	18463A-QC-0.02-1	CR	0.0213		112	
0.191	18472A-MV-0.2-1	MV	0.186	0.179 ± 0.0096	97	94 ± 6
	18472A-MV-0.2-2	MV	0.183		96	
	18472A-MV-0.2-3	MV	0.183		96	
	18352A-QC-0.2-1	SSCR	0.165		86	
1.91	18472A -MV-2-1	MV	1.63	1.74 ± 0.089	85	91 ± 5
	18472A -MV-2-2	MV	1.73		90	
	18472A -MV-2-3	MV	1.84		96	
	18467A-QC-2-1	CR	1.76		92	
3.0	18479A-QC-3-1	CR	2.67	---	93	---

*average of 4 injections

Notes:

s.d. = standard deviation

MV = Method Validation

CR = Concurrent spike recovery

All concurrent recovery (CR) samples were analyzed twice (double injection) and the values are averages of multiple analysis.

Table X. Summary of TFNA Spike Recoveries (MV, CR, SSCR) for Mint Tops (Leaves & Stems)

Spike level (ppm)	Lab sample ID	Type of recovery	Determined value (ppm)	Average ± s.d. (ppm)	Spike recovery (%)	Average recovery (%)
TFNA						
0.02002	18472A-MV-0.02-1	MV	0.0205	0.0206 ± 0.0013	103	103 ± 7
	18472A-MV-0.02-2	MV	0.0209		104	
	18472A-MV-0.02-3	MV	0.0215		107	
	18473A-QC-0.02-1	CR	0.0185		93	
	18353A-QC-0.02-1	CR	0.0223*		111	
	18463A-QC-0.02-1	CR	0.0197		98	
0.2002	18472A-MV-0.2-1	MV	0.208	0.210 ± 0.0062	104	105 ± 3
	18472A-MV-0.2-2	MV	0.220		110	
	18472A-MV-0.2-3	MV	0.207		103	
	18352A-QC-0.2-1	SSCR	0.207		104	
2.002	18472A -MV-2-1	MV	1.83	1.96 ± 0.12	91	98 ± 6
	18472A -MV-2-2	MV	1.89		95	
	18472A -MV-2-3	MV	2.11		105	
	18467A-QC-2-1	CR	1.99		100	
3.0	18479A-QC-3-1	CR	2.93	---	98	---

* average of 4 injections

Notes:
s.d. = standard deviation
MV = Method Validation
CR = Concurrent spike recovery
All concurrent recovery (CR) samples were analyzed twice (double injection) and the values are averages of multiple analysis.

Table XI. Summary of Flonicamid Spike Recoveries (MV, CR, SSCR) for Mint Oil

Spike level (ppm)	Lab sample ID	Type of recovery	Determined value (ppm)	Average ± s.d. (ppm)	Spike recovery (%)	Average recovery (%)
Flonicamid (IKI-220)						
0.0202	18476A-MV-0.02-1	MV	0.0208	0.0194 ± 0.0014	103	96 ± 7
	18476A-MV-0.02-2	MV	0.0176		87	
	18476A-MV-0.02-3	MV	0.0205		102	
	18476A-QC-0.02-1	CR	0.0179		89	
	18470A-QC-0.02-1	CR	0.0198		98	
	18470A-QC-0.02-2	CR	0.0199		98	
0.202	18476A-MV-0.2-1	MV	0.189	0.188 ± 0.0083	93	93 ± 4
	18476A-MV-0.2-2	MV	0.178		88	
	18476A-MV-0.2-3	MV	0.199		98	
	18476A-QC-0.2-1	CR	0.181		89	
	18470A-QC-0.2-1	SSCR	0.192		95	
2.02	18476A-MV-2-1	MV	1.83	1.75 ± 0.074	91	87 ± 4
	18476A-MV-2-2	MV	1.75		87	
	18476A-MV-2-3	MV	1.68		83	

Notes:
s.d. = standard deviation
MV = Method Validation
CR = Concurrent spike recovery
All concurrent recovery (CR) samples were analyzed twice (double injection) and the values are averages of multiple analysis.

Table XII. Summary of TFNA-AM Spike Recoveries (MV, CR, SSCR) for Mint Oil

Spike level (ppm)	Lab sample ID	Type of recovery	Determined value (ppm)	Average ± s.d. (ppm)	Spike recovery (%)	Average recovery (%)
TFNA-AM						
0.02024	18476A-MV-0.02-1	MV	0.0188	0.0194 ± 0.0016	93	96 ± 8
	18476A-MV-0.02-2	MV	0.0197		97	
	18476A-MV-0.02-3	MV	0.0206		102	
	18476A-QC-0.02-1	CR	0.0165		81	
	18470A-QC-0.02-1	CR	0.0210		104	
	18470A-QC-0.02-2	CR	0.0202		100	
0.2024	18476A-MV-0.2-1	MV	0.191	0.176 ± 0.013	94	87 ± 6
	18476A-MV-0.2-2	MV	0.179		88	
	18476A-MV-0.2-3	MV	0.186		92	
	18476A-QC-0.2-1	CR	0.163		80	
	18470A-QC-0.2-1	SSCR	0.164		81	
2.024	18476A-MV-2-1	MV	1.78	1.78 ± 0.052	88	88 ± 3
	18476A-MV-2-2	MV	1.83		91	
	18476A-MV-2-3	MV	1.73		85	

Notes:
s.d. = standard deviation
MV = Method Validation
CR = Concurrent spike recovery
All concurrent recovery (CR) samples were analyzed twice (double injection) and the values are averages of multiple analysis.

Table XIII. Summary of TFNG Spike Recoveries (MV, CR, SSCR) for Mint Oil

Spike level (ppm)	Lab sample ID	Type of recovery	Determined value (ppm)	Average ± s.d. (ppm)	Spike recovery (%)	Average recovery (%)
TFNG						
0.0191	18476A-MV-0.02-1	MV	0.0178	0.0169 ± 0.0013	93	88 ± 7
	18476A-MV-0.02-2	MV	0.0167		87	
	18476A-MV-0.02-3	MV	0.0178		93	
	18476A-QC-0.02-1	CR	0.0151		79	
	18470A-QC-0.02-1	CR	0.0157		82	
	18470A-QC-0.02-2	CR	0.0181		95	
0.191	18476A-MV-0.2-1	MV	0.181	0.179 ± 0.0094	95	94 ± 6
	18476A-MV-0.2-2	MV	0.184		96	
	18476A-MV-0.2-3	MV	0.191		100	
	18476A-QC-0.2-1	CR	0.170		89	
	18470A-QC-0.2-1	SSCR	0.169		89	
1.91	18476A-MV-2-1	MV	1.83	1.80 ± 0.041	96	94 ± 2
	18476A-MV-2-2	MV	1.83		96	
	18476A-MV-2-3	MV	1.76		92	

Notes:
s.d. = standard deviation
MV = Method Validation
CR = Concurrent spike recovery
All concurrent recovery (CR) samples were analyzed twice (double injection) and the values are averages of multiple analysis.

Table XIV. Summary of TFNA Spike Recoveries (MV, CR, SSCR) for Mint Oil

Spike level (ppm)	Lab sample ID	Type of recovery	Determined value (ppm)	Average ± s.d. (ppm)	Spike recovery (%)	Average recovery (%)
TFNA						
0.02002	18476A-MV-0.02-1	MV	0.0177	0.0183 ± 0.0011	88	91 ± 6
	18476A-MV-0.02-2	MV	0.0181		90	
	18476A-MV-0.02-3	MV	0.0185		93	
	18476A-QC-0.02-1	CR	0.0173		86	
	18470A-QC-0.02-1	CR	0.0177		89	
	18470A-QC-0.02-2	CR	0.0205		102	
0.2002	18476A-MV-0.2-1	MV	0.182	0.186 ± 0.0086	91	93 ± 4
	18476A-MV-0.2-2	MV	0.180		90	
	18476A-MV-0.2-3	MV	0.189		95	
	18476A-QC-0.2-1	CR	0.180		90	
	18470A-QC-0.2-1	SSCR	0.200		100	
2.002	18476A-MV-2-1	MV	1.91	1.78 ± 0.12	95	89 ± 6
	18476A-MV-2-2	MV	1.73		86	
	18476A-MV-2-3	MV	1.70		85	

Notes:
s.d. = standard deviation
MV = Method Validation
CR = Concurrent spike recovery
All concurrent recovery (CR) samples were analyzed twice (double injection) and the values are averages of multiple analysis.

Table XV. Storage Stability (SS) of Flonicamid and its Metabolites on Mint Tops (Leaves & Stems)

Crop part	Lab sample ID	Flonicamid (0.2012 ppm spike)		TFNA-AM (Metabolite) (0.2024 ppm spike)		TFNG (Metabolite) (0.1994 ppm spike)		TFNA (Metabolite) (0.2002 ppm spike)	
		Value (ppm)	Recovery (%)	Value (ppm)	Recovery (%)	Value (ppm)	Recovery (%)	Value (ppm)	Recovery (%)
Tops (leaves& stems)	18352A-0.2-SS1	0.1737	86	0.1628	80	0.1656	83	0.1513	76
	18352A-0.2-SS2	0.1646	82	0.1526	75	0.1574	79	0.1292	65
	18352A-0.2-SS3	0.1739	86	0.1666	82	0.1598	80	0.1556	78
	Average ± s.d.	0.17 ± 0.005	85 ± 3	0.16 ± 0.007	79 ± 4	0.16 ± 0.004	81 ± 2	0.15 ± 0.014	73 ± 7

Crop part	Lab sample ID	Date SS sample prepared	Date SS sample extracted	SS sample Stored (>90%) (days)	Maximum actual field sample storage	
					days	Trial No.
Tops (leaves& stems)	18352A-0.2-SS1	10-Aug-11	08-Aug-12	364	372	11-ID12
	18352A-0.2-SS2					
	18352A-0.2-SS3					

Table XVI. Storage Stability (SS) of Flonicamid and its Metabolites on Mint Oil

Crop part	Lab sample ID	Flonicamid (0.2012 ppm spike)		TFNA-AM (Metabolite) (0.2024 ppm spike)		TFNG (Metabolite) (0.1994 ppm spike)		TFNA (Metabolite) (0.2002 ppm spike)	
		Value (ppm)	Recovery (%)	Value (ppm)	Recovery (%)	Value (ppm)	Recovery (%)	Value (ppm)	Recovery (%)
Mint Oil	18470A-0.2-SS1	0.0926	46	0.1077	53	0.0840	42	0.0845	42
	18470A-0.2-SS2	0.0870	43	0.0923	46	0.0899	45	0.0974	49
	18470A-0.2-SS3	0.0874	43	0.1068	53	0.0903	45	0.0894	45
Average ± s.d.		0.089 ± 0.003	44 ± 2	0.10 ± 0.009	50 ± 4	0.088 ± 0.004	44 ± 2	0.09 ± 0.007	45 ± 3

Crop part	Lab sample ID	Date SS sample prepared	Date SS sample extracted	SS sample Stored (>90%) (days)	Maximum actual field sample storage	
					days	Trial No.
Mint oil	18470A-0.2-SS1	8-Sep-11	07-Aug-12	334	368	11-WA*18
	18470A-0.2-SS2					
	18470A-0.2-SS3					

Table XVII. Summary of Residue Data for Mint Tops (Leaves & Stems) and Mint Oil Samples for Flonicamid (IKI-220) and Metabolites TFNA-AM, TFNG and TFNA

Trial ID	Crop Part	Field rep No.	Unique lab sample ID	Date sampled	Date extracted	Date analyzed	Days Stored	Flonicamid Residue (ppm)	TFNA-AM Residue (ppm)	TFNG Residue (ppm)	TFNA Residue (ppm)
11-WI17	Tops (Leaves & Stems)	A	18472	09-Aug-2011	N/A	N/A	---	N/A	N/A	N/A	N/A
		B	18473		31-Jul-2012	02-Aug-2012	357	< 0.02	< 0.02	< 0.02	< 0.02
		C	18474					0.500	0.0723	0.219	0.107
		D	18475					0.504	0.0752	0.225	0.108
	Oil	G	18476		06-Aug-2012	06-Aug-2012	---	< 0.02	< 0.02	< 0.02	< 0.02
		H	18477				363	< 0.02	< 0.02	< 0.02	< 0.02
11-WI18	Tops (Leaves & Stems)	A	18478	16-Aug - 2011	N/A	N/A	---	N/A	N/A	N/A	N/A
		B	18479		03-Aug-2012	03-Aug -2012	353	< 0.02	< 0.02	< 0.02	< 0.02
		C	18480					1.90	0.167	0.367	0.211
		D	18481					1.93	0.164	0.356	0.204
11-WA*18	Tops (Leaves & Stems)	A	18466	28-Jul-2011	N/A	N/A	---	N/A	N/A	N/A	N/A
		B	18467		26-Jul-2012	30-Jul-2012	364	< 0.02	< 0.02	< 0.02	< 0.02
		C	18468					1.55	0.170	0.329	0.193
		D	18469					1.59	0.177	0.349	0.193
	Oil	G	18470		30-Jul-2012	30-Jul-2012	---	< 0.02	< 0.02	< 0.02	< 0.02
		H	18471				368	< 0.02	< 0.02	< 0.02	< 0.02
11-ID12	Tops (Leaves & Stems)	A	18352	18-Jul-2011	N/A	N/A	---	N/A	N/A	N/A	N/A
		B	18353		24-Jul-2012	25-Jul-2012	372	< 0.02	< 0.02	< 0.02	< 0.02
		C	18354					2.31	0.0856	0.376*	0.146*
		D	18355					2.41	0.125*	0.377*	0.133*
11-WA17	Tops (Leaves & Stems)	A	18462	28-Jul-2011	N/A	N/A	---	N/A	N/A	N/A	N/A
		B	18463		25-Jul-2012	25-Jul-2012	363	< 0.02	< 0.02	< 0.02	< 0.02
		C	18464					1.67	0.214	0.451	0.222
		D	18465					1.73	0.254	0.461	0.235

*average of 4 injections

(End of Table XVII)

Attachment A: Index to Representative Chromatograms

Each chromatogram represents a 10-µL injection. Chromatograms included are for demonstration purposes only and not all of the chromatograms obtained are included.

- WA*18-Tops (Sample D, 1.52 ppm of Flonicamid (IKI-220), 0.156 ppm of TFNA-AM, 316 ppm of TFNG & 0.168 ppm of TFNA)
- WA*18-Oil (Sample H, <0.02 ppm of Flonicamid (IKI-220), <0.02 ppm of TFNA-AM, <0.02(0.004) ppm of TFNG & <0.02ppm of TFNA)
- ID12-Tops (Sample D, 2.45ppm of Flonicamid (IKI-220), 0.150 ppm of TFNA-AM, 0.404 ppm of TFNG & 0.139ppm of TFNA)
- WA17-Tops (Sample C, 1.70 ppm of Flonicamid (IKI-220), 0.233ppm of TFNA-AM, 0.468 ppm of TFNG & 0.227 ppm of TFNA)

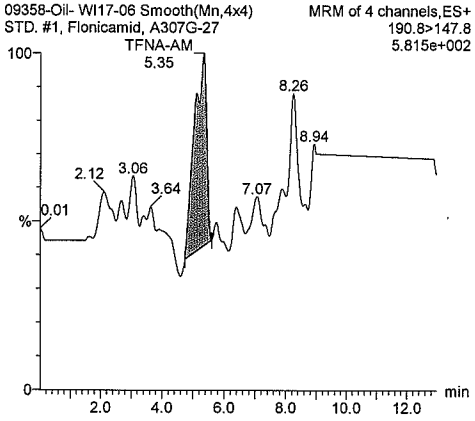
WI17-58 Aug-7-12 EG

Quantify Sample Report **MassLynx 4.1 SCN 714** Page 1 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358- Oil -WA17- 2012-8-6.qld

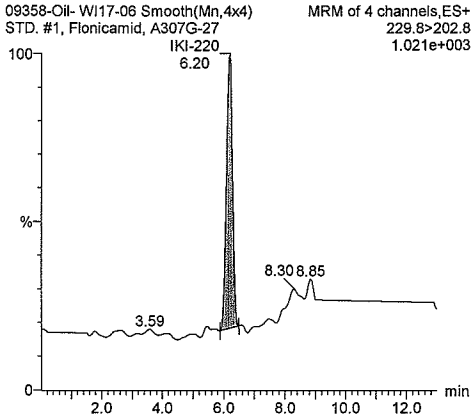
Method: C:\MassLynx\Data\09358.PRO\MethDB\Flonicamid .mdb 23 May 2012 12:28:34
Calibration: C:\MassLynx\Data\09358.PRO\CurveDB\Fludioxonil.cdb 28 Nov 2006 15:37:17

Name: 09358-Oil- WI17-06
Description: STD. #1, Flonicamid, A307G-27
Date: 06-Aug-2012
Time: 13:48:38
Vial: 1:A,2
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

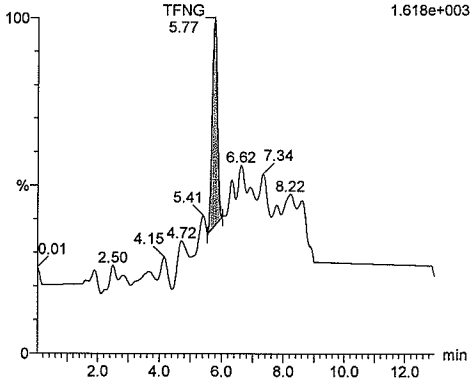
TFNA-AM



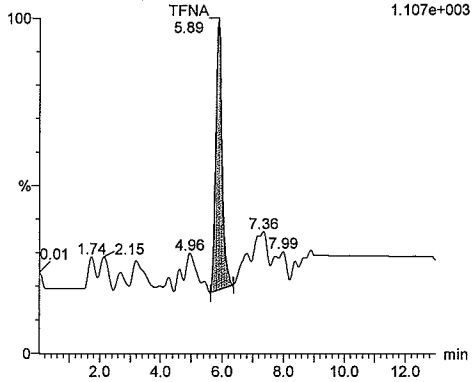
IKI-220



TFNG



TFNA



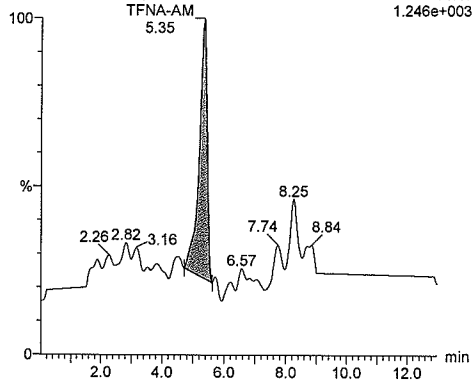
#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.35	155	bb	10
2	IKI-220	229.8>202.8	6.20	194	bb	118
3	TFNG	248.8>202.8	5.77	198	bb	17
4	TFNA	191.8>147.8	5.89	218	bb	25

WI17-59 Aug-7-12 EG

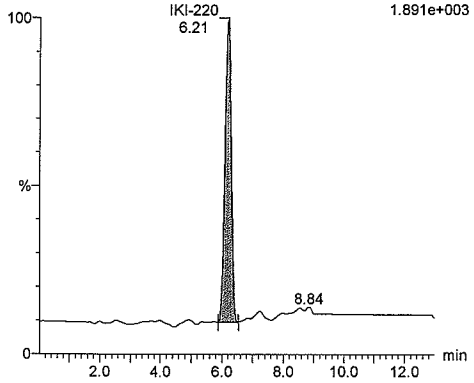
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 2 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358- Oil -WA17- 2012-8-6.qld

Name: 09358-Oil- WI17-07
Description: STD. #2, Flonicamid, A307G-26
Date: 06-Aug-2012
Time: 14:02:52
Vial: 1:A,3
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

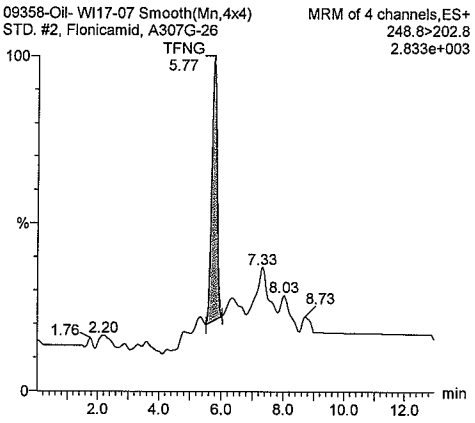
TFNA-AM



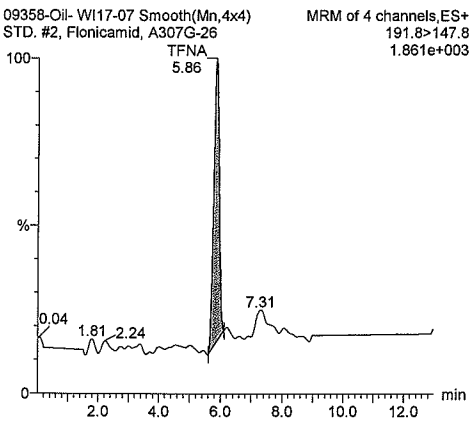
IKI-220



TFNG



TFNA



	# Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.35	288	bb	34
2	IKI-220	229.8>202.8	6.21	411	bb	93
3	TFNG	248.8>202.8	5.77	451	bb	91
4	TFNA	191.8>147.8	5.86	331	bb	117

WI17-60 Aug-7-12 EG

Quantify Sample Report **MassLynx 4.1 SCN 714** Page 3 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358- Oil -WA17- 2012-8-6.qld

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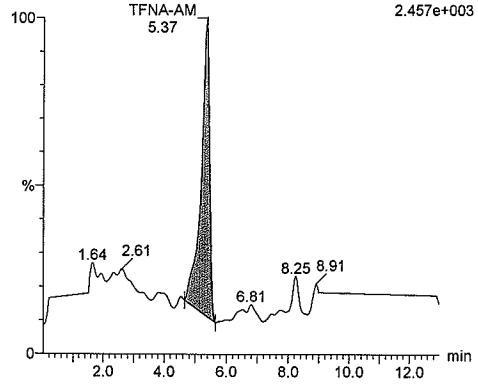
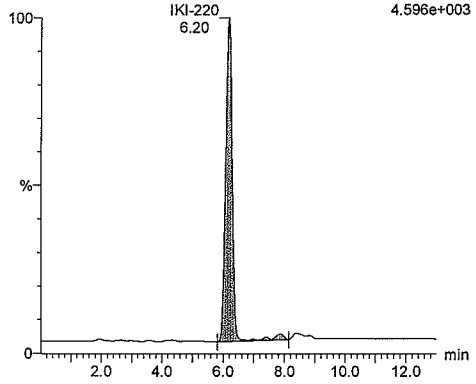
Date: 06-Aug-2012
Time: 14:20:36
Vial: 1:A,4
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

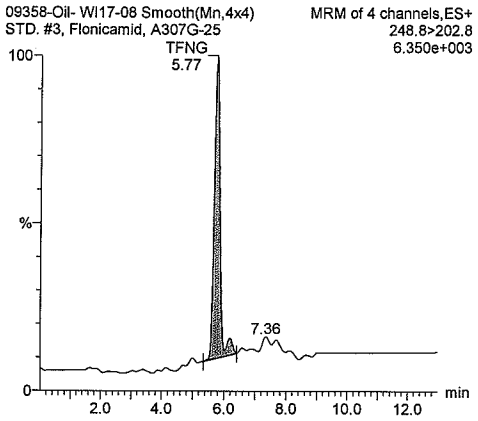
TFNA-AM



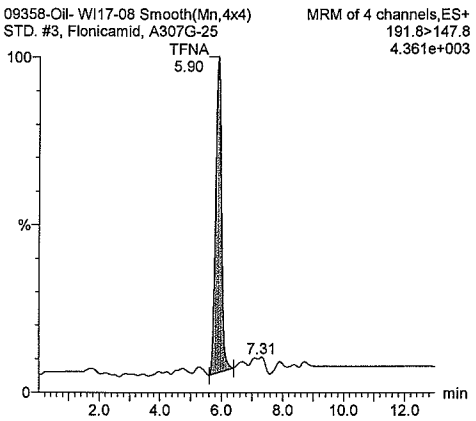
IKI-220



TFNG



TFNA



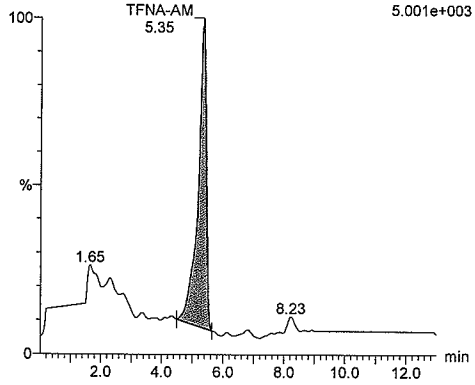
#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.37	694	bb	74
2	IKI-220	229.8>202.8	6.20	1139	bb	453
3	TFNG	248.8>202.8	5.77	1253	bb	115
4	TFNA	191.8>147.8	5.90	915	bb	196

WI17-61 Aug-7-12 EG

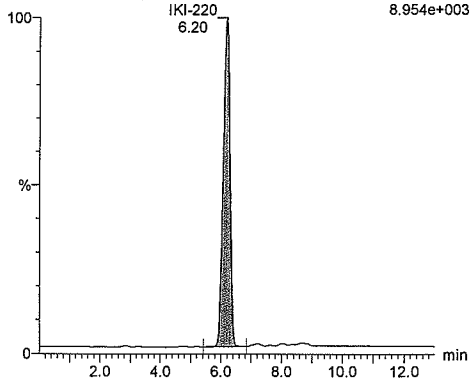
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 4 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358- Oil -WA17- 2012-8-6.qld

Name: 09358-Oil- WI17-09
Description: STD. #4, Flonicamid, A307G-24
Date: 06-Aug-2012
Time: 14:34:50
Vial: 1:A,5
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

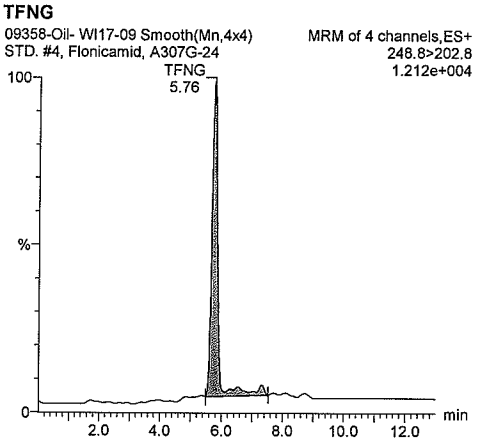
TFNA-AM



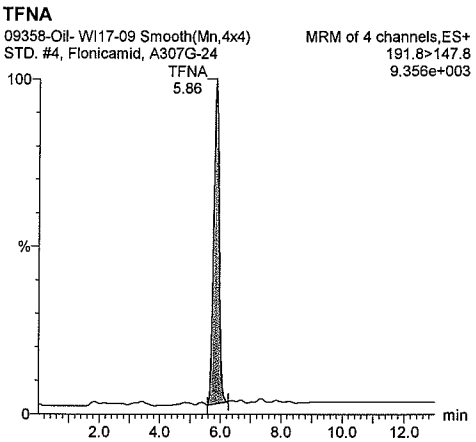
IKI-220



TFNG



TFNA



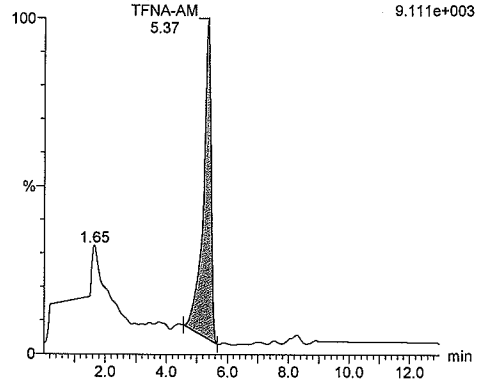
	# Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.35	1444	bb	133
2	IKI-220	229.8>202.8	6.20	2297	bb	540
3	TFNG	248.8>202.8	5.76	2679	bb	315
4	TFNA	191.8>147.8	5.86	1950	bb	512

WI17-62 Aug-7-12 EG

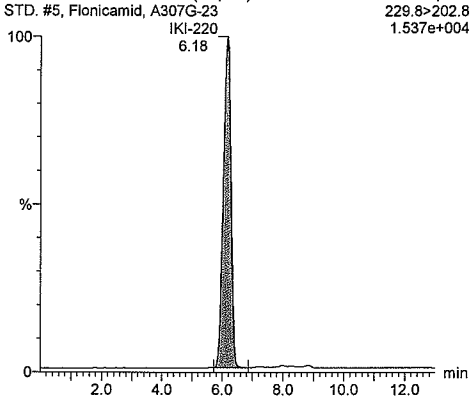
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 5 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358- Oil -WA17- 2012-8-6.qld

Name: 09358-Oil- WI17-10
Description: STD. #5, Flonicamid, A307G-23
Date: 06-Aug-2012
Time: 14:49:04
Vial: 1:A,6
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

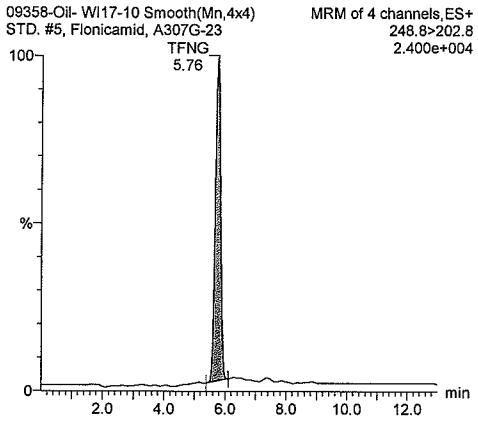
TFNA-AM



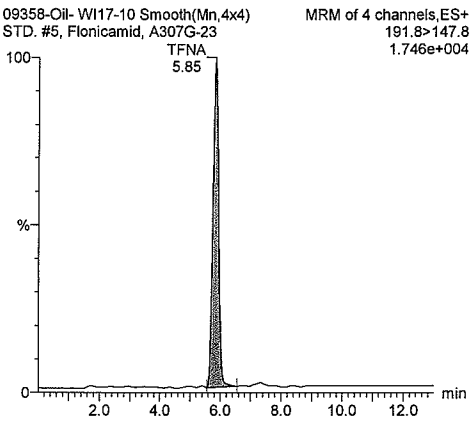
IKI-220



TFNG



TFNA



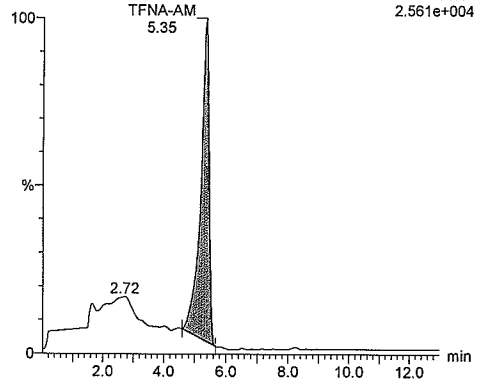
#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.37	2625	bb	190
2	IKI-220	229.8>202.8	6.18	4453	bb	1593
3	TFNG	248.8>202.8	5.76	4746	bb	542
4	TFNA	191.8>147.8	5.85	3791	bb	389

WI17-63 Aug-7-12 EG

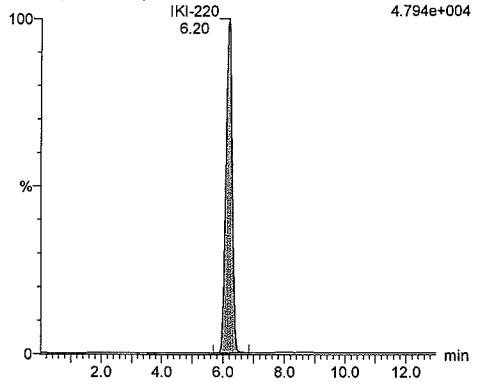
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 6 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358- Oil -WA17- 2012-8-6.qld

Name: 09358-Oil- WI17-11
Description: STD. #6, Flonicamid, A307G-22
Date: 06-Aug-2012
Time: 15:03:17
Vial: 1:A,7
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

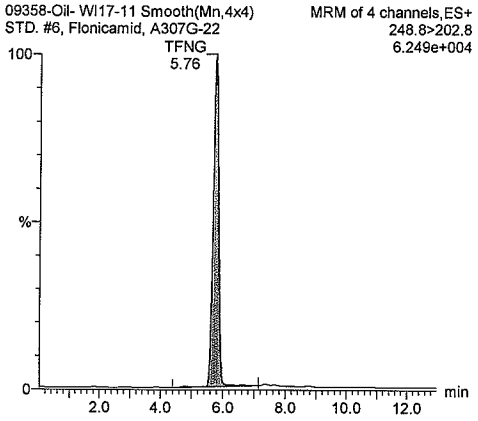
TFNA-AM



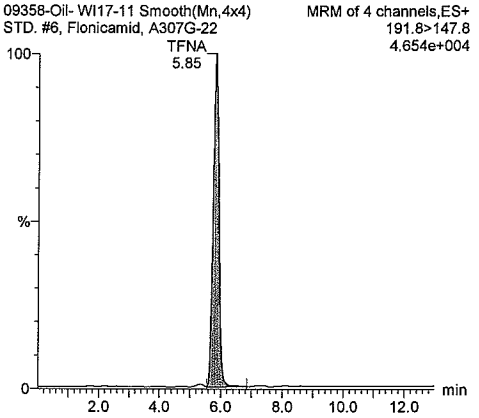
IKI-220



TFNG



TFNA



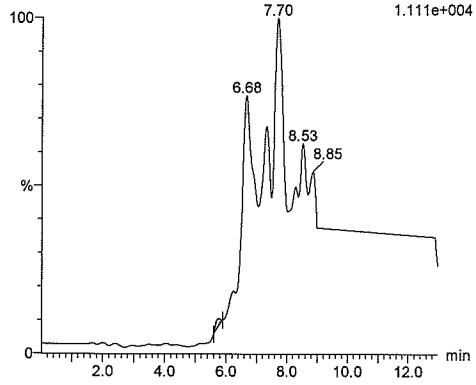
	# Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.35	7730	bb	310
2	IKI-220	229.8>202.8	6.20	11530	bb	4513
3	TFNG	248.8>202.8	5.76	12865	bb	1724
4	TFNA	191.8>147.8	5.85	10191	bb	1362

WI17-30 3 Aug 12 EG

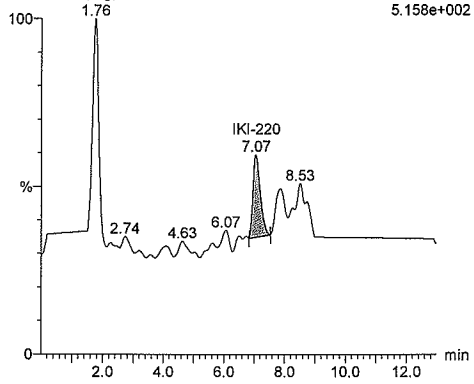
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 8 of 23
Dataset: C:\MassLynx\Data\09358.PRO\09358-Tops-WI17- 2012-8-2 .qld

Name: 09358-Tops-WI17-39
Description: 18473A-C-1, 5g, FV= 200 mL
Date: 02-Aug-2012
Time: 13:02:37
Vial: 1:B,1
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

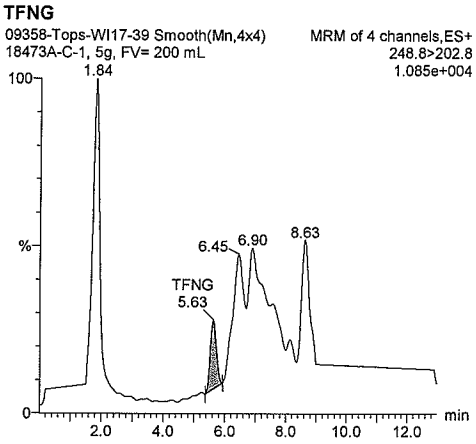
TFNA-AM



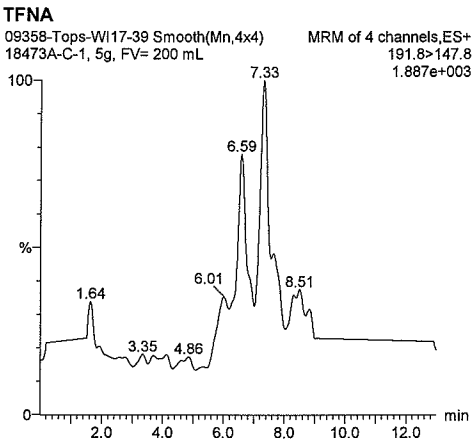
IKI-220



TFNG



TFNA



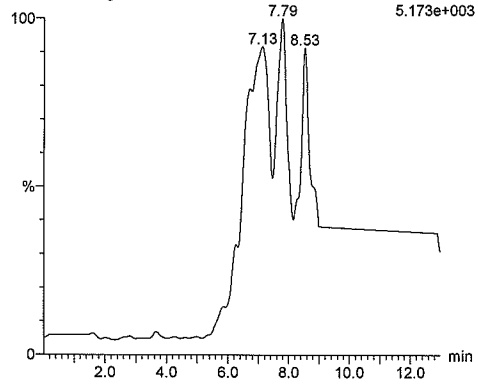
#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.77	48	bb	3
2	IKI-220	229.8>202.8	7.07	37	bb	11
3	TFNG	248.8>202.8	5.63	514	bb	29
4	TFNA	191.8>147.8				

WA*18-31 July 31-12 EG

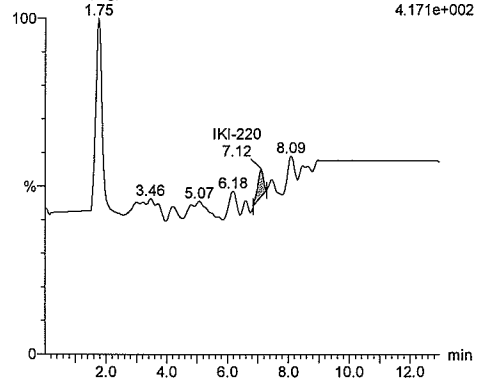
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 9 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358-Tops-WA18- 2012-7-30.qld

Name: 09358-Tops-WA*18-14
Description: 18467A-C-1, 5g, FV= 200 mL
Date: 30-Jul-2012
Time: 11:49:26
Vial: 1:B,1
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

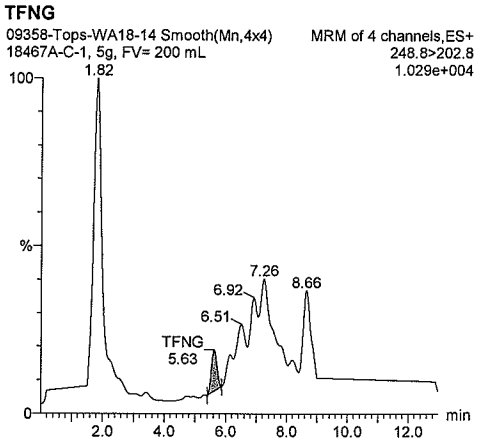
TFNA-AM



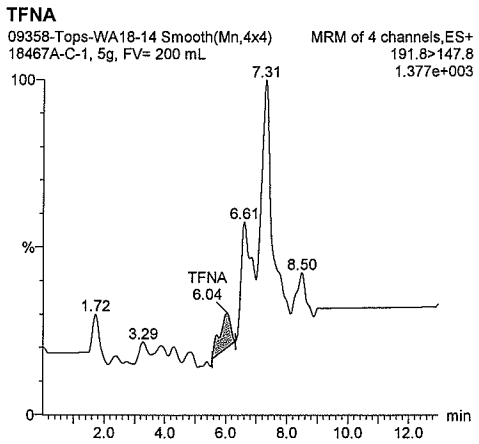
IKI-220



TFNG



TFNA



#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8				
2	IKI-220	229.8>202.8	7.12	8	bb	4
3	TFNG	248.8>202.8	5.63	269	bb	14
4	TFNA	191.8>147.8	6.04	62	bb	2

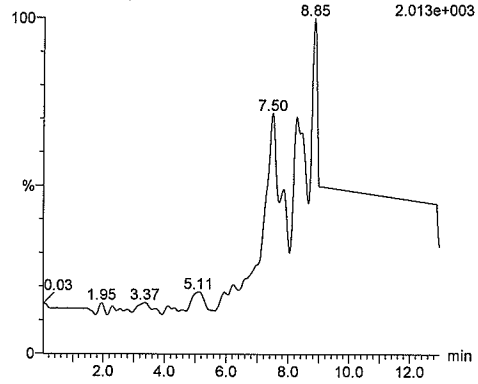
* EC=1, EG, 7-31-12

WI17-66 Aug-7-12 EG

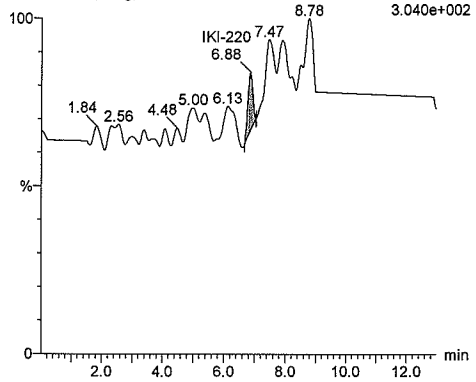
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 9 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358- Oil -WA17- 2012-8-6.qld

Name: 09358-Oil- WI17-14
Description: 184676A-C-4, 2.5g, FV= 100 mL
Date: 06-Aug-2012 EG, EC=4, 8-7-12
Time: 16:46:01
Vial: 1:B,1
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

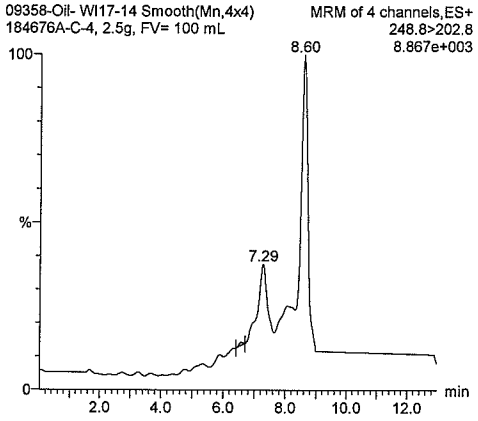
TFNA-AM



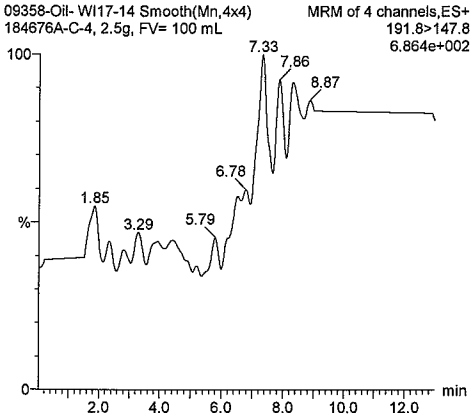
IKI-220



TFNG



TFNA



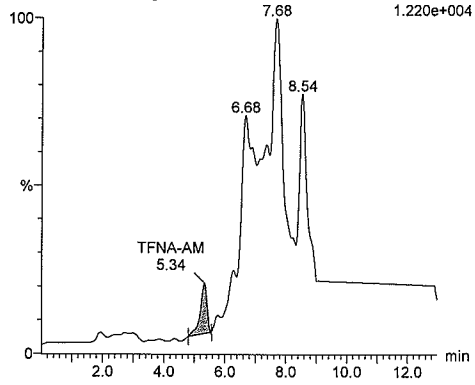
#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8				
2	IKI-220	229.8>202.8	6.88	10	bb	5
3	TFNG	248.8>202.8	6.59	13	bb	12
4	TFNA	191.8>147.8				

MV-95 July-20-12 26

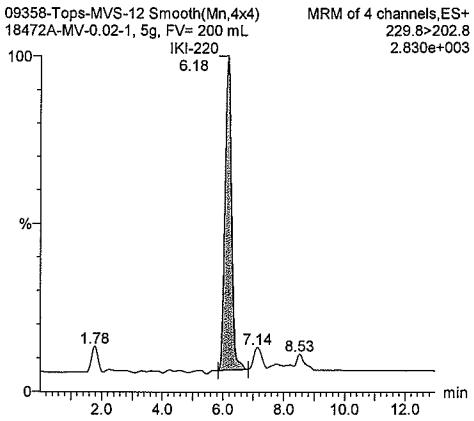
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 9 of 18
Dataset: C:\MassLynx\Data\09358.PRO\09358-Topsl- 0.02 MV- 2012-7-19.qld

Name: 09358-Tops-MVS-12
Description: 18472A-MV-0.02-1, 5g, FV= 200 mL
Date: 19-Jul-2012
Time: 15:48:56
Vial: 1:B,2
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

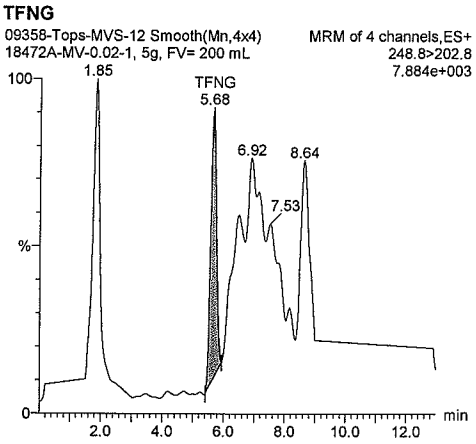
TFNA-AM



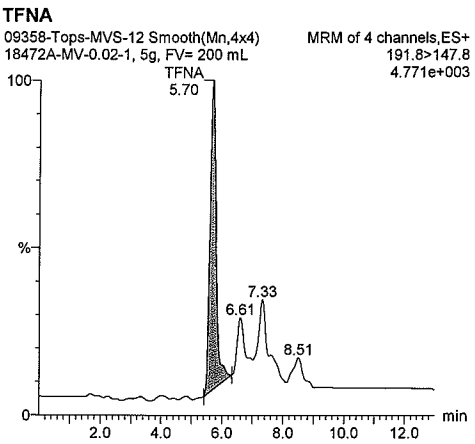
IKI-220



TFNG



TFNA



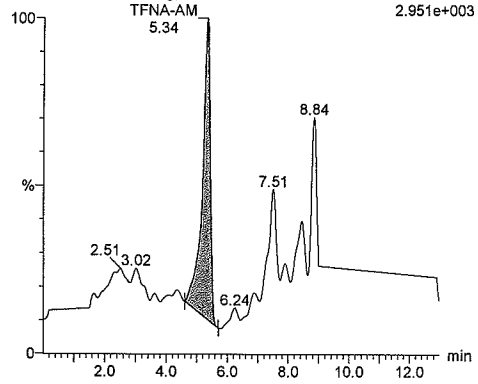
#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.34	469	bb	11
2	IKI-220	229.8>202.8	6.18	684	bb	342
3	TFNG	248.8>202.8	5.68	1414	bb	81
4	TFNA	191.8>147.8	5.70	1049	bb	337

MV-17 July-12-12 EG

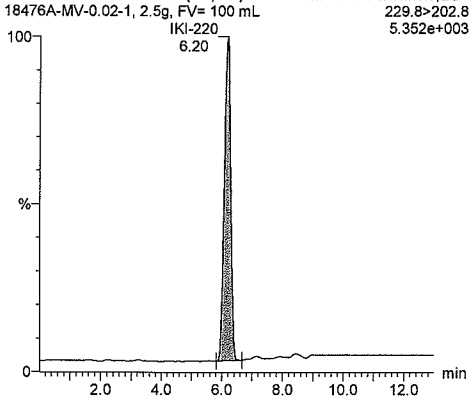
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 9 of 18
Dataset: C:\MassLynx\Data\09358.PRO\09358- Oil- 0.02 MV- 2012-7-11.qld

Name: 09358-Oil-MVS-12
Description: 18476A-MV-0.02-1, 2.5g, FV= 100 mL
Date: 11-Jul-2012
Time: 12:33:48
Vial: 1:B,2
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

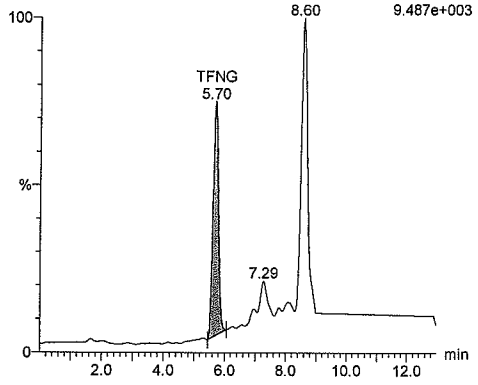
TFNA-AM



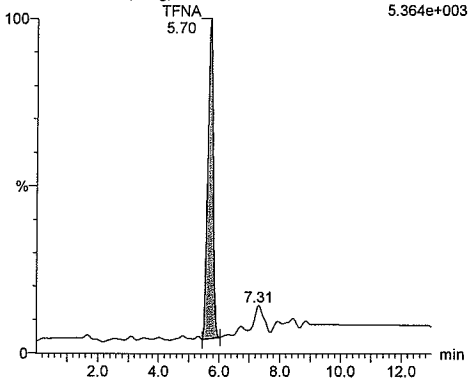
IKI-220



TFNG



TFNA



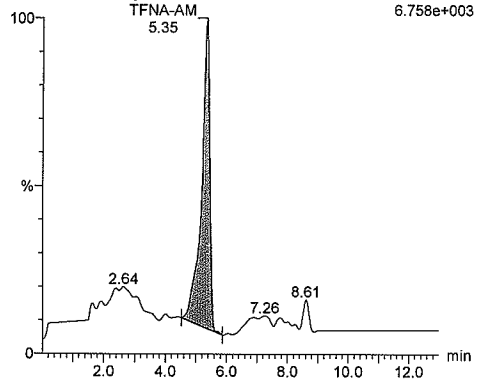
	# Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.34	801	bb	48
2	IKI-220	229.8>202.8	6.20	1275	bb	565
3	TFNG	248.8>202.8	5.70	1395	bb	212
4	TFNA	191.8>147.8	5.70	1068	bb	236

WA*18-33 July 31-12 EG

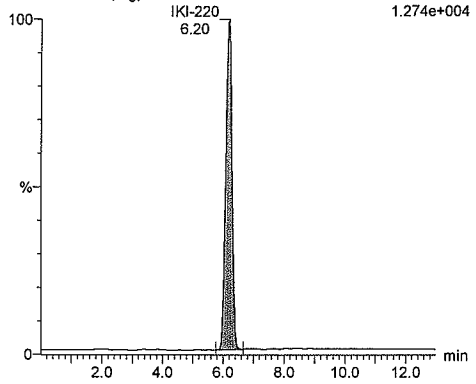
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 11 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358-Tops-WA18- 2012-7-30.qld

Name: 09358-Tops-WA18-16*
Description: 18467A-QC-2-1, 5g, FV= 5000 mL
Date: 30-Jul-2012
Time: 12:17:52
Vial: 1:B,2
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

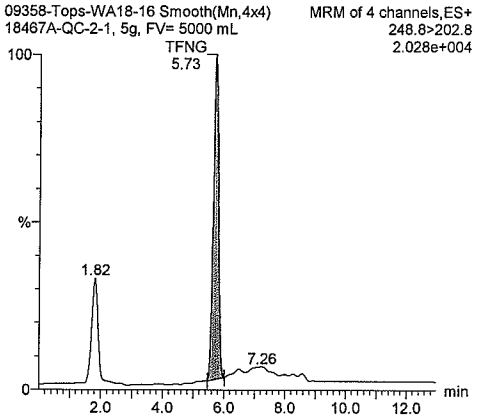
TFNA-AM



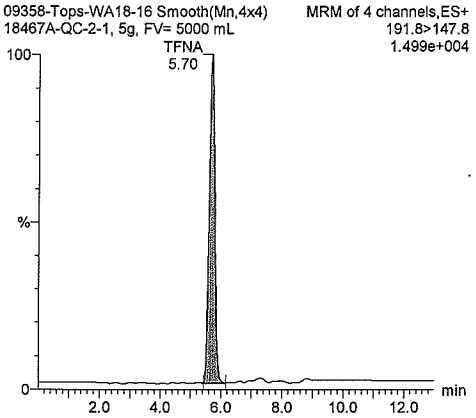
IKI-220



TFNG



TFNA



	# Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.35	1959	bb	136
2	IKI-220	229.8>202.8	6.20	3026	bb	651
3	TFNG	248.8>202.8	5.73	3995	bb	588
4	TFNA	191.8>147.8	5.70	3186	bb	580

* EC=1, EG, 7-31-12

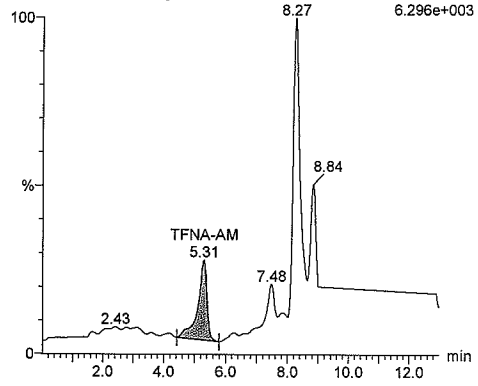
Attachment A: Representative Chromatograms

WA*18-65 July 31-12 EG

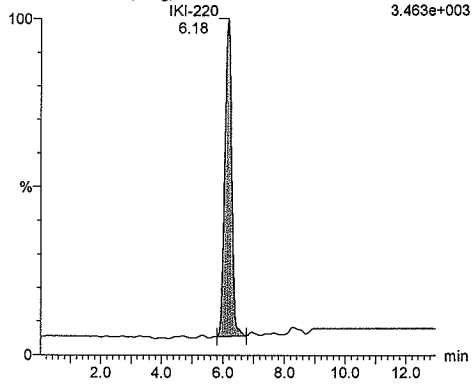
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 10 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358- Oil -WA18- 2012-7-30.qld

Name: 09358-Oil-WA*18-14
Description: 18470A-QC-0.02-1, 2.5g, FV= 100 mL
Date: 30-Jul-2012
Time: 18:20:31
Vial: 1:B,2
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

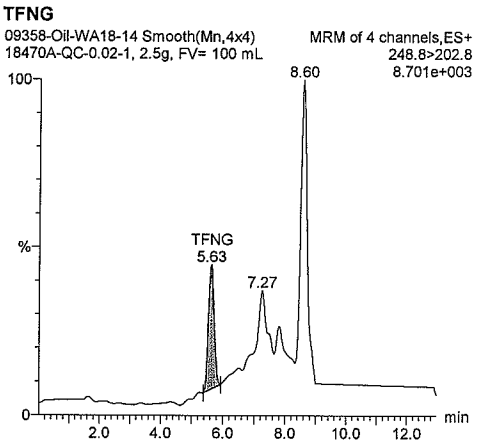
TFNA-AM



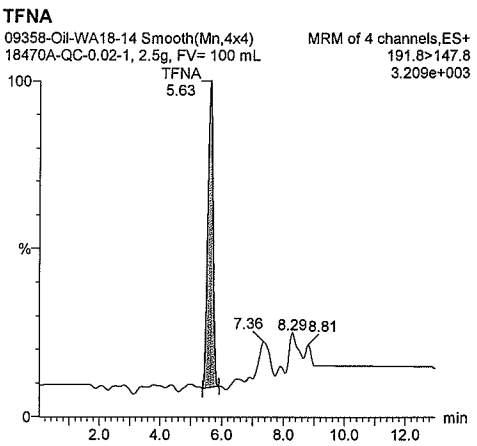
IKI-220



TFNG



TFNA



#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.31	530	bb	23
2	IKI-220	229.8>202.8	6.18	867	bb	235
3	TFNG	248.8>202.8	5.63	698	bb	158
4	TFNA	191.8>147.8	5.63	588	bb	122

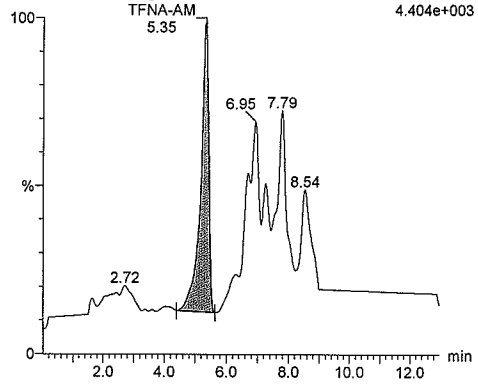
* EC=1, EG, 7-31-12

SS-48 Aug-9-12 EG

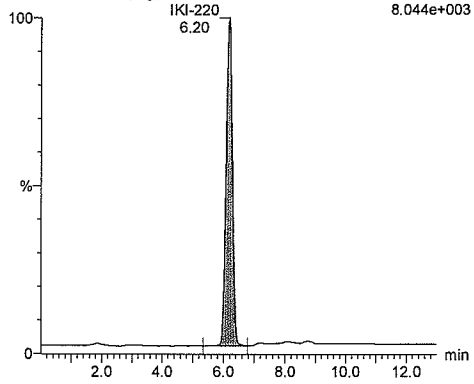
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 11 of 20
Dataset: C:\MassLynx\Data\09358.PRO\09358- Tops-SS-2012-8-8 .qld

Name: 09358-Tops - SS- 13
Description: 18352A- 0.2- SS 1, 5g, FV= 1000 mL
Date: 08-Aug-2012
Time: 16:42:21
Vial: 1:B,3
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

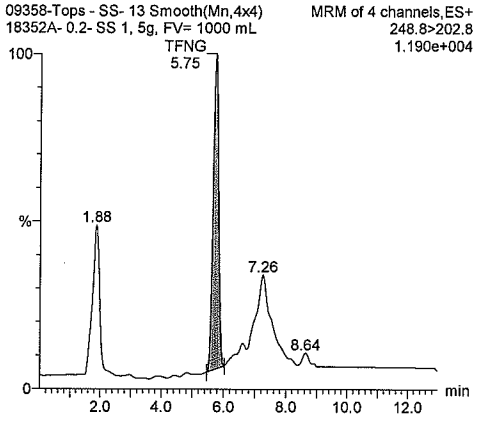
TFNA-AM



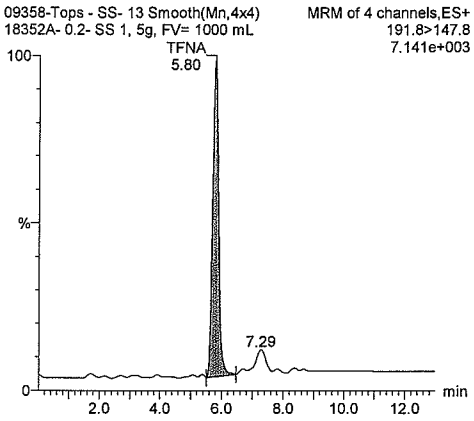
IKI-220



TFNG



TFNA



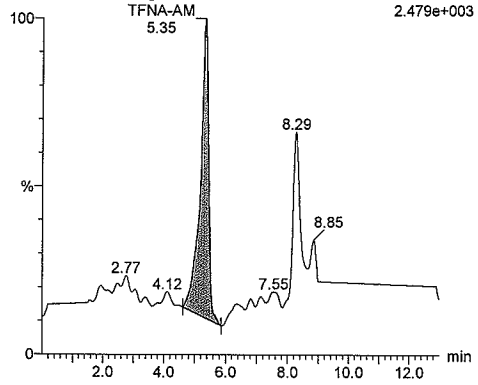
#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.35	1168	bb	77
2	IKI-220	229.8>202.8	6.20	1896	bb	1255
3	TFNG	248.8>202.8	5.75	2282	bb	136
4	TFNA	191.8>147.8	5.80	1535	bb	108

SS-19 Aug-8-12 EG

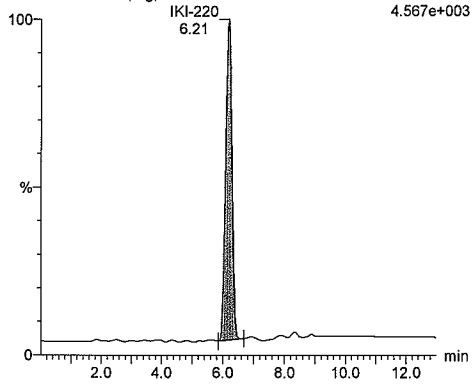
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 10 of 19
Dataset: C:\MassLynx\Data\09358.PRO\09358- Oil- SS - 2012-8-7 .qld

Name: 09358-Oil- SS- 13
Description: 18470A- 0.2- SS 1, 5g, FV= 1000 mL
Date: 07-Aug-2012
Time: 16:49:41
Vial: 1:B,3
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

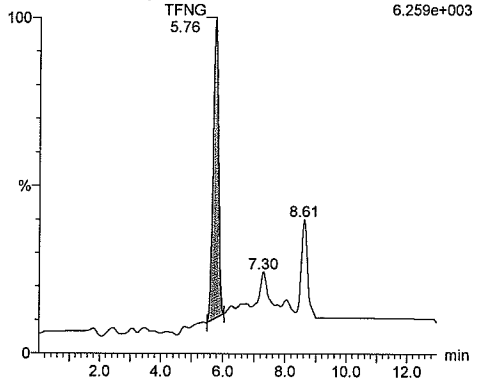
TFNA-AM



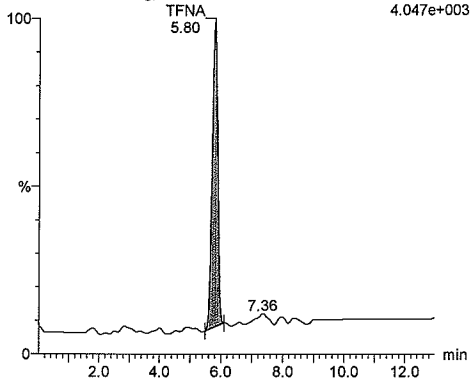
IKI-220



TFNG



TFNA



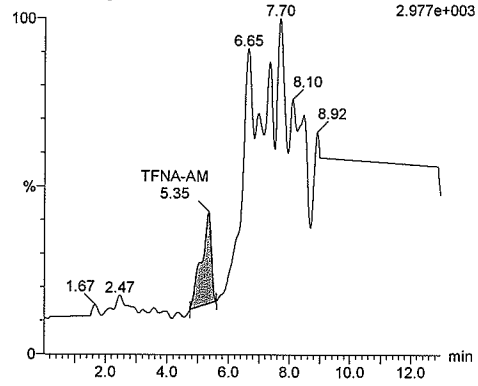
#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.35	696	bb	57
2	IKI-220	229.8>202.8	6.21	1050	bb	207
3	TFNG	248.8>202.8	5.76	1153	bb	68
4	TFNA	191.8>147.8	5.80	786	bb	211

WI17-37 3 Aug 12 EG

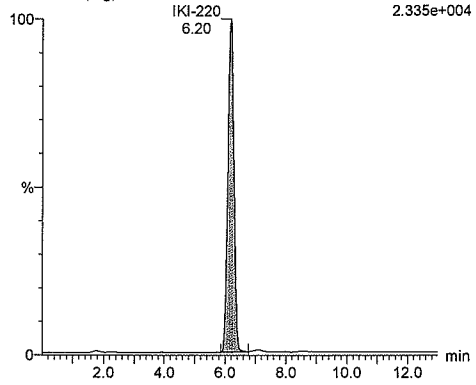
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 15 of 23
Dataset: C:\MassLynx\Data\09358.PRO\09358-Tops-WI17- 2012-8-2 .qld

Name: 09358-Tops-WI17-46
Description: 18475A-T-1, 5g, FV= 1000 mL
Date: 02-Aug-2012
Time: 14:42:07
Vial: 1:B,4
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

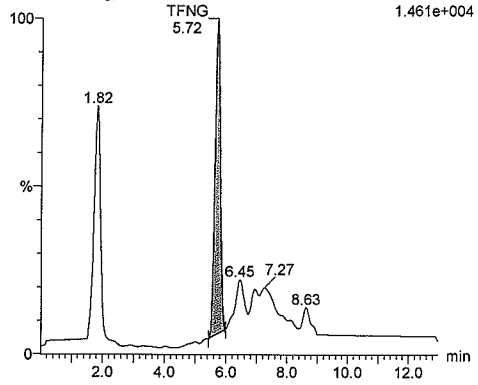
TFNA-AM



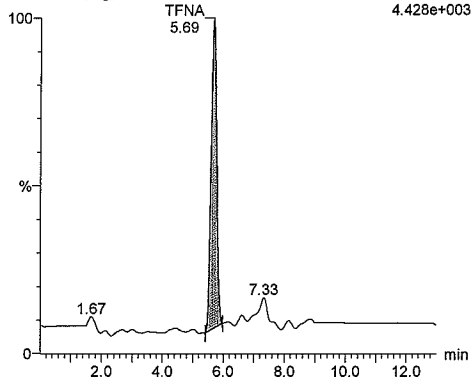
IKI-220



TFNG



TFNA



#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.35	304	bb	6
2	IKI-220	229.8>202.8	6.20	5520	bb	1550
3	TFNG	248.8>202.8	5.72	2857	bb	98
4	TFNA	191.8>147.8	5.69	890	bb	138

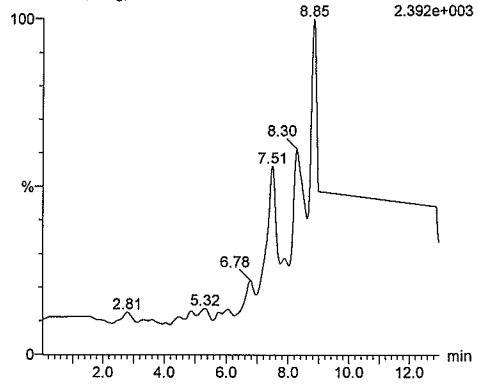
Attachment A: Representative Chromatograms

WI17-72 Aug-7-12 EG

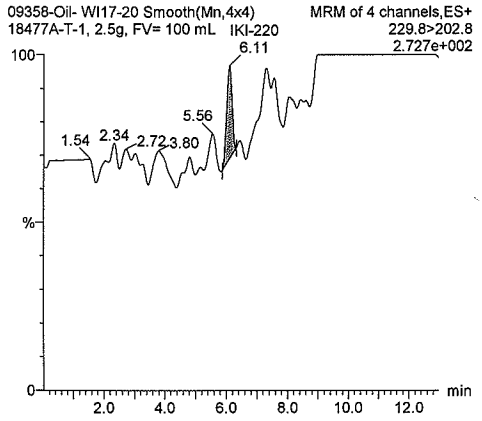
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 15 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358- Oil -WA17- 2012-8-6.qld

Name: 09358-Oil- WI17-20
Description: 18477A-T-1, 2.5g, FV= 100 mL
Date: 06-Aug-2012
Time: 18:11:16
Vial: 1:B,4
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

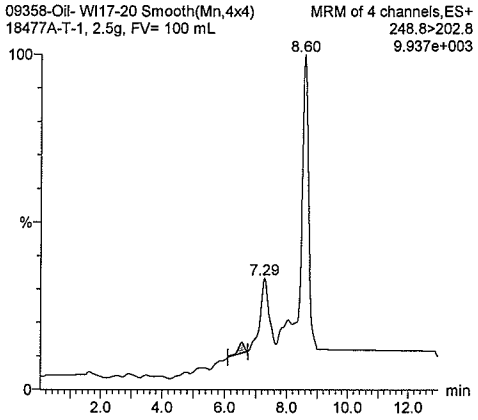
TFNA-AM



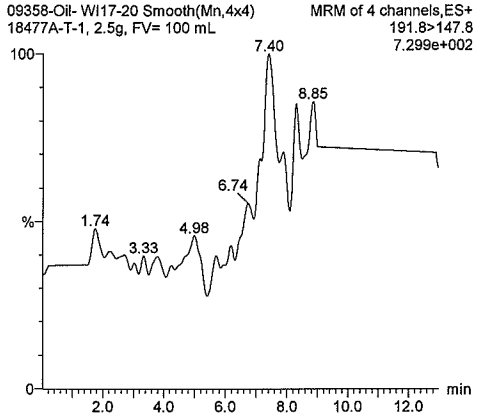
IKI-220



TFNG



TFNA



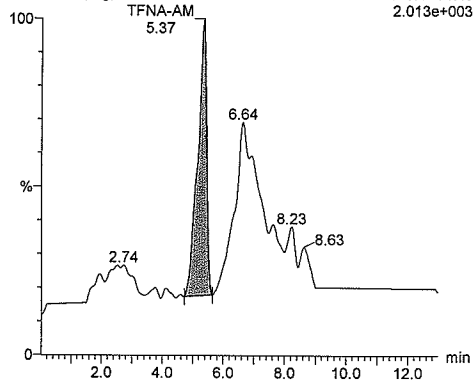
#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8				
2	IKI-220	229.8>202.8	6.11	16	bb	6
3	TFNG	248.8>202.8	6.57	76	bb	10
4	TFNA	191.8>147.8				

WI18-33 Aug-6-12 EG

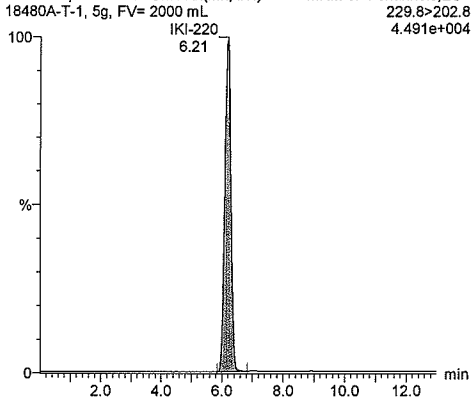
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 13 of 23
Dataset: C:\MassLynx\Data\09358.PRO\09358-Tops-WI18- 2012-8-3.qld

Name: 09358-Tops-WI18-18
Description: 18480A-T-1, 5g, FV= 2000 mL
Date: 03-Aug-2012
Time: 17:14:36
Vial: 1:B,3
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

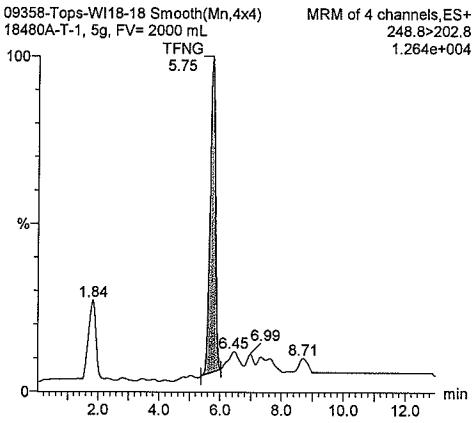
TFNA-AM



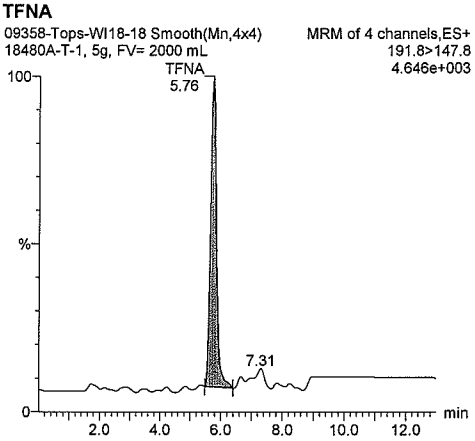
IKI-220



TFNG



TFNA



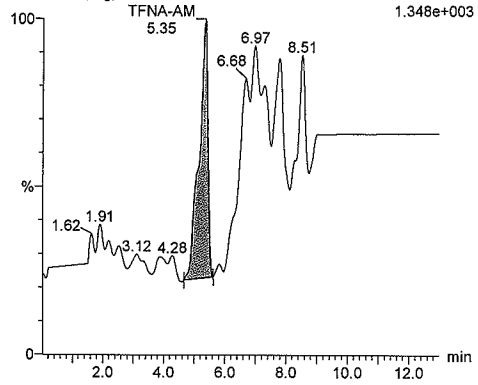
#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.37	570	bb	29
2	IKI-220	229.8>202.8	6.21	10570	bb	3284
3	TFNG	248.8>202.8	5.75	2461	bb	252
4	TFNA	191.8>147.8	5.76	981	bb	145

WA*18-36 Jug 31-12 EG

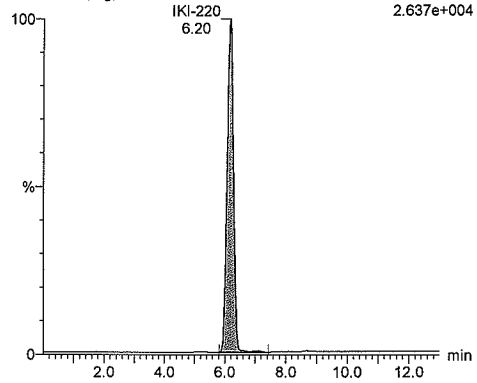
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 14 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358-Tops-WA18- 2012-7-30.qld

Name: 09358-Tops-WA*18-19
Description: 18469A-T-1, 5g, FV= 2000 mL
Date: 30-Jul-2012
Time: 13:00:32
Vial: 1:B,4
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

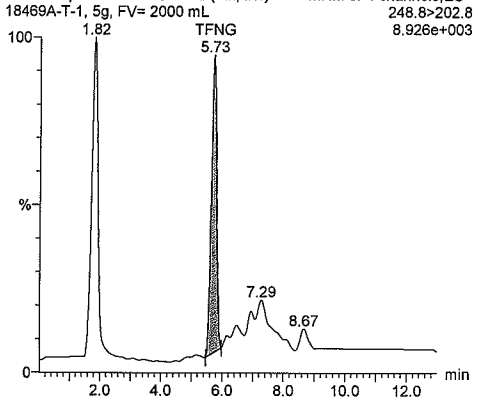
TFNA-AM



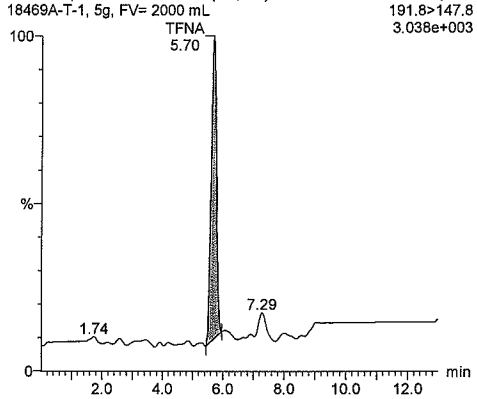
IKI-220



TFNG



TFNA



	# Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.35	351	bb	18
2	IKI-220	229.8>202.8	6.20	6633	bb	1745
3	TFNG	248.8>202.8	5.73	1618	bb	159
4	TFNA	191.8>147.8	5.70	569	bb	110

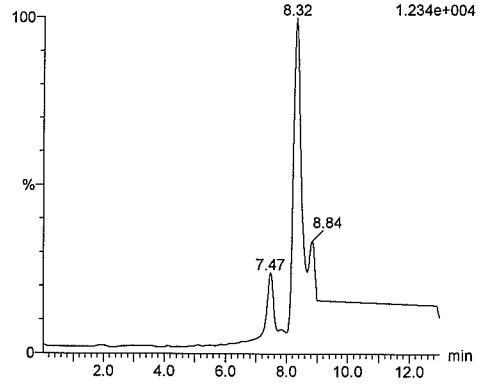
* EC=1, EG, 7-31-12

WA*18-70 July 31-12 EG

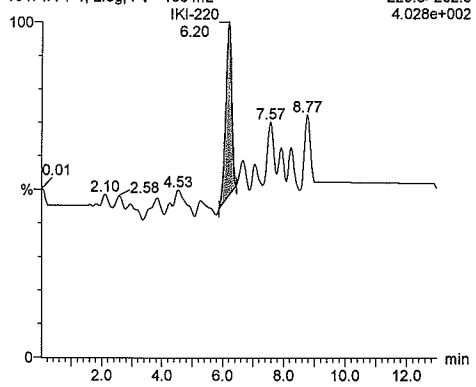
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 15 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358- Oil -WA18- 2012-7-30.qld

Name: 09358-Oil-WA*18-19
Description: 18471A-T-1, 2.5g, FV= 100 mL
Date: 30-Jul-2012
Time: 19:31:35
Vial: 1:B,4
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

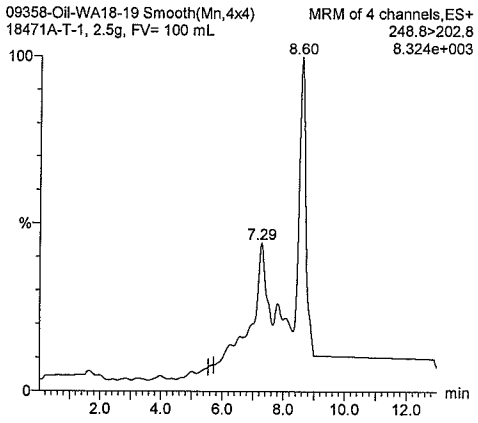
TFNA-AM



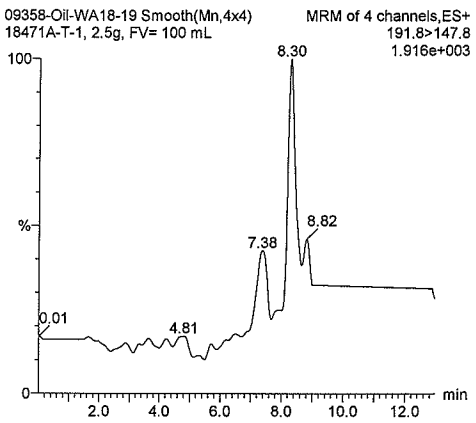
IKI-220



TFNG



TFNA



#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8				
2	IKI-220	229.8>202.8	6.20	48	bb	12
3	TFNG	248.8>202.8	5.66	2	bb	-2
4	TFNA	191.8>147.8				

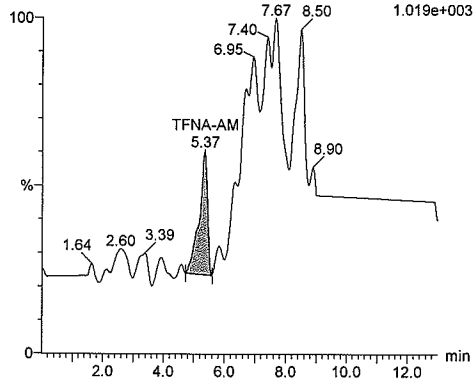
*EC=1, EG, 7-31-12

ID12-65 July 25-12 EG

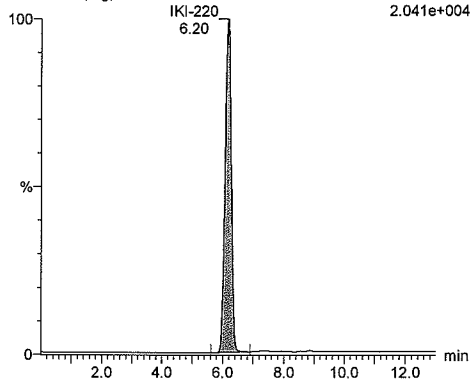
Quantify Sample Report **MassLynx 4.1 SCN 714** Page 15 of 22
Dataset: C:\MassLynx\Data\09358.PRO\09358-Tops-ID12- 2012-7-25.qld

Name: 09358-Tops-ID12-47
Description: 18355A-T-1, 5g, FV= 4000 mL
Date: 25-Jul-2012
Time: 12:13:49
Vial: 1:B,4
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

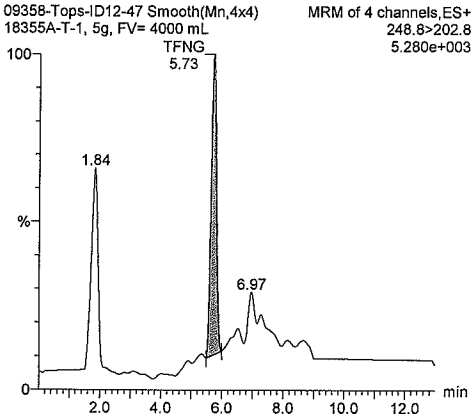
TFNA-AM



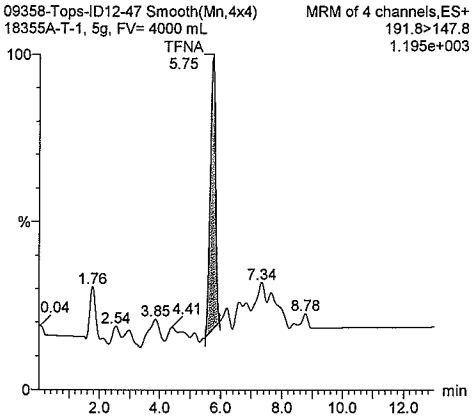
IKI-220



TFNG



TFNA



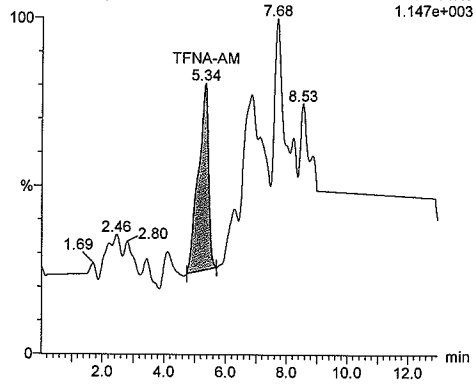
#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.37	121	bb	5
2	IKI-220	229.8>202.8	6.20	4829	bb	698
3	TFNG	248.8>202.8	5.73	946	bb	86
4	TFNA	191.8>147.8	5.75	202	bb	41

WA17-33 26 July 12 EG

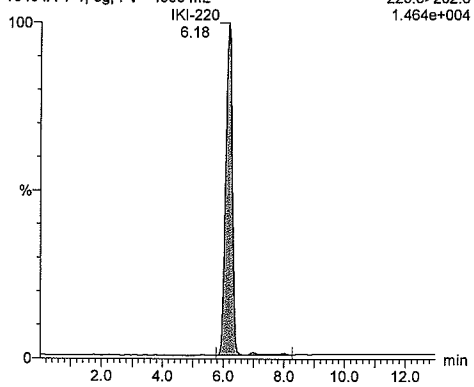
Quantify Sample Report **MassLynx 4.1 SCN 714**
Dataset: C:\MassLynx\Data\09358.PRO\09358-Tops- WA17- 2012-7-25.qld

Name: 09358-Tops-WA17-17
Description: 18464A-T-1, 5g, FV= 4000 mL
Date: 25-Jul-2012
Time: 17:56:04
Vial: 1:B,3
Inlet Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\LC-Flonicamid
MS Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MRM-Flonicamid.EXP
Tune Method Name: C:\MassLynx\Data\09358.PRO\ACQUDB\MSMS- Flonicamid.IPR
User: EG

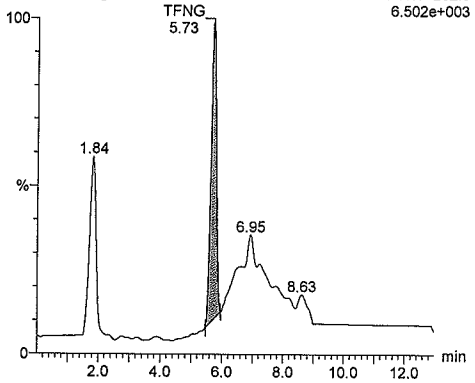
TFNA-AM



IKI-220



TFNG



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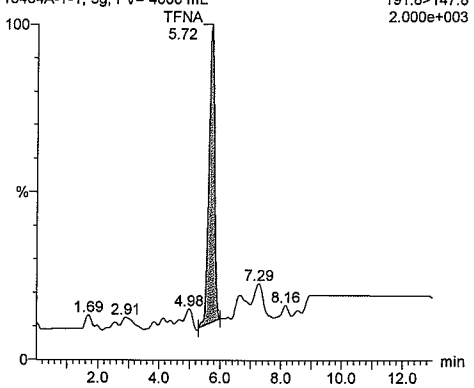


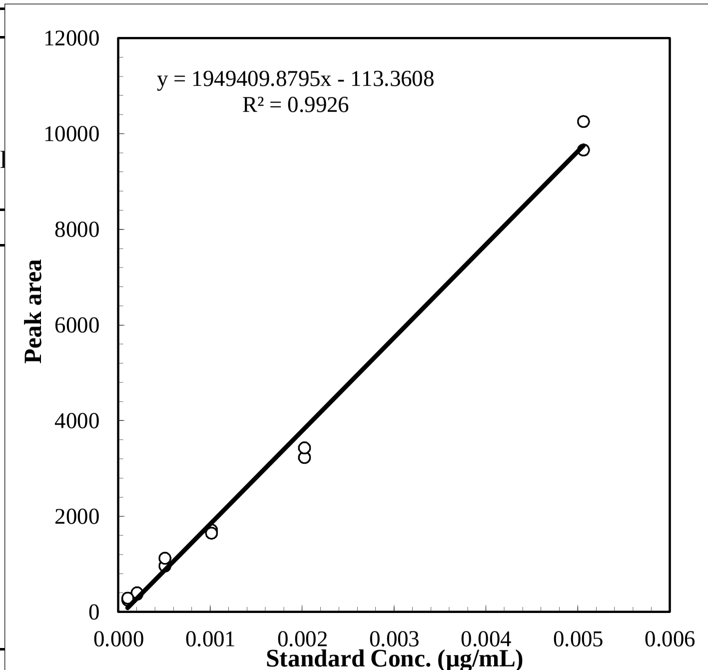
#	Name	Trace	RT	Area	Detection Flags	S/N
1	TFNA-AM	190.8>147.8	5.34	248	bb	15
2	IKI-220	229.8>202.8	6.18	3716	bb	1469
3	TFNG	248.8>202.8	5.73	1191	bb	187
4	TFNA	191.8>147.8	5.72	388	bb	61

Attachment A: Representative Chromatograms

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNA-AM	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS



CALIBRATION DATA

Date of calibration analysis: 11-Jul-12

RTSDB Page	Calibration standard ID (or name)	Calibration standard Conc.(µg/mL)	Peak Area
MV-09A	A307G-27	0.0001012	250
MV-10	A307G-26	0.0002024	375
MV-11	A307G-25	0.0005060	964
MV-12	A307G-24	0.001012	1715
MV-13	A307G-23	0.0020240	3232
MV-14	A307G-22	0.005060	9663
MV-21	A307G-22	0.005060	10258
MV-22	A307G-23	0.0020240	3434
MV-23A	A307G-24	0.001012	1650
MV-24	A307G-25	0.0005060	1126
MV-25	A307G-26	0.0002024	403
MV-26	A307G-27	0.0001012	291

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1949410
Intercept, b = -113.36
R^2 = 0.9926
LOQ (µg/g) = 0.02

ANALYTICAL DATA

Extraction date: 11-Jul-12 Analysis date: 11-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-16	18476A-C-1	C	NA	2.50	NA	0	100	< 0.02	NA
MV-17	18476A-MV-0.02-1	F	0.0506	2.50	0.02024	801	100	0.01876	92.70
MV-18	18476A-MV-0.02-2	F	0.0506	2.50	0.02024	846	100	0.01969	97.26
MV-19	18476A-MV-0.02-3	F	0.0506	2.50	0.02024	892	100	0.02063	101.92

NOTES

*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

Attachment B: Calculation Worksheets, Standard Curves and LOD/LOQ Calculations

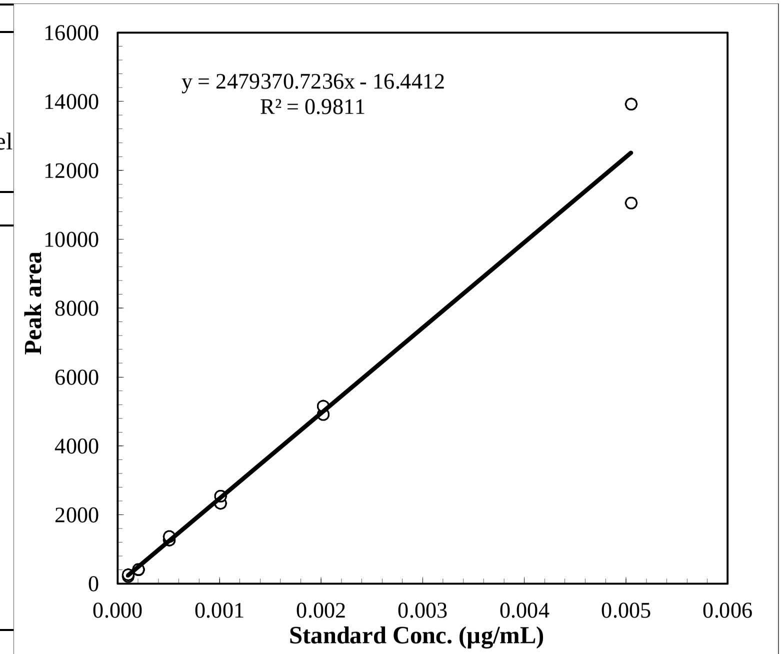
CALCULATION PAGE

PROJECT INFORMATION

R Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
nalysis for:	Flonicamid (IKI-220)	Field Research Director:	Dr. Scott Chapman
ommodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 11-Jul-12

RTSDB Page	Calibration standard ID (or name)	Calibration standard Conc.(µg/mL)	Peak Area	----	---- ----------------
MV-09	A307G-27	0.0001010	213	Type:	Linear regression
MV-10	A307G-26	0.0002020	407	Equation:	y = mx + b
MV-11	A307G-25	0.0005050	1270	Slope, m =	2479371
MV-12	A307G-24	0.001010	2337	Intercept, b =	-16.44
MV-13	A307G-23	0.0020200	4915	R^2 =	0.9811
MV-14	A307G-22	0.005050	11055	LOQ (µg/g) =	0.02
MV-21	A307G-22	0.005050	13926		
MV-22	A307G-23	0.0020200	5157		
MV-23	A307G-24	0.001010	2544		
MV-24	A307G-25	0.0005050	1371		
MV-25	A307G-26	0.0002020	417		
MV-26	A307G-27	0.0001010	264		



ANALYTICAL DATA Extraction date: 11-Jul-12 Analysis date: 11-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-16	18476A-C-1	C	NA	2.50	NA	8	100	0.000394	NA
MV-17	18476A-MV-0.02-1	F	0.0505	2.50	0.02020	1275	100	0.02083	103.14
MV-18	18476A-MV-0.02-2	F	0.0505	2.50	0.02020	1072	100	0.01756	86.93
MV-19	18476A-MV-0.02-3	F	0.0505	2.50	0.02020	1256	100	0.02053	101.63

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNG	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

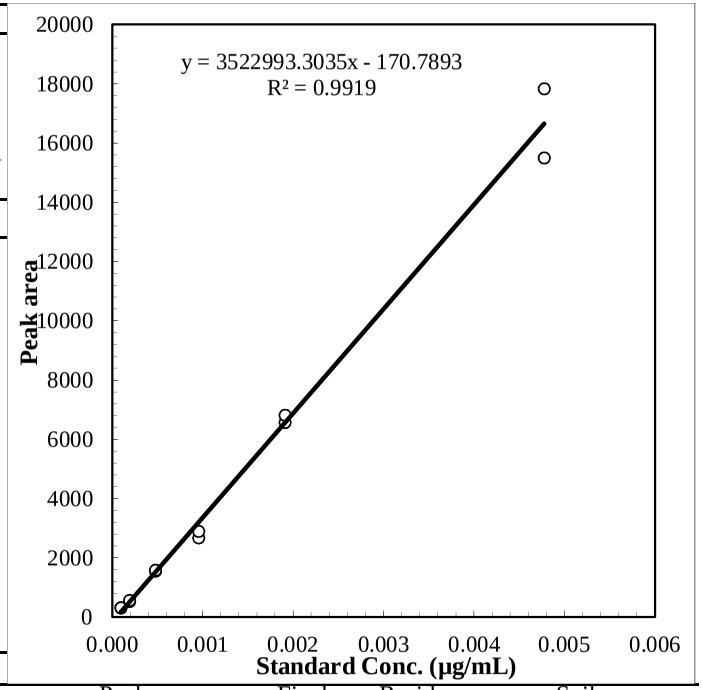
CALIBRATION DATA

Date of calibration analysis: 11-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL	Peak Area
MV-09	A307G-27	0.00009550	317
MV-10	A307G-26	0.0001910	528
MV-11	A307G-25	0.0004775	1554
MV-12	A307G-24	0.00095500	2676
MV-13	A307G-23	0.0019100	6572
MV-14	A307G-22	0.004775	15500
MV-21	A307G-22	0.004775	17835
MV-22	A307G-23	0.0019100	6818
MV-23	A307G-24	0.00095500	2897
MV-24	A307G-25	0.0004775	1586
MV-25	A307G-26	0.0001910	568
MV-26	A307G-27	0.00009550	314

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 3522993
Intercept, b = -170.79
R^2 = 0.9919
LOQ (µg/g) = 0.02



ANALYTICAL DATA

Extraction date: 11-Jul-12 Analysis date: 11-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-16	18476A-C-1	C	NA	2.50	NA	23	100	0.00220	NA
MV-17	18476A-MV-0.02-1	F	0.04775	2.50	0.019100	1395	100	0.01778	93.08
MV-18	18476A-MV-0.02-2	F	0.04775	2.50	0.019100	1298	100	0.01668	87.31
MV-19	18476A-MV-0.02-3	F	0.04775	2.50	0.019100	1398	100	0.01781	93.26

NOTES

*1: C=Control, F=Fortified, T=Treated

*2: Spike amount = (Spike added) ÷ (Sample weight)

*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)

*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100

"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

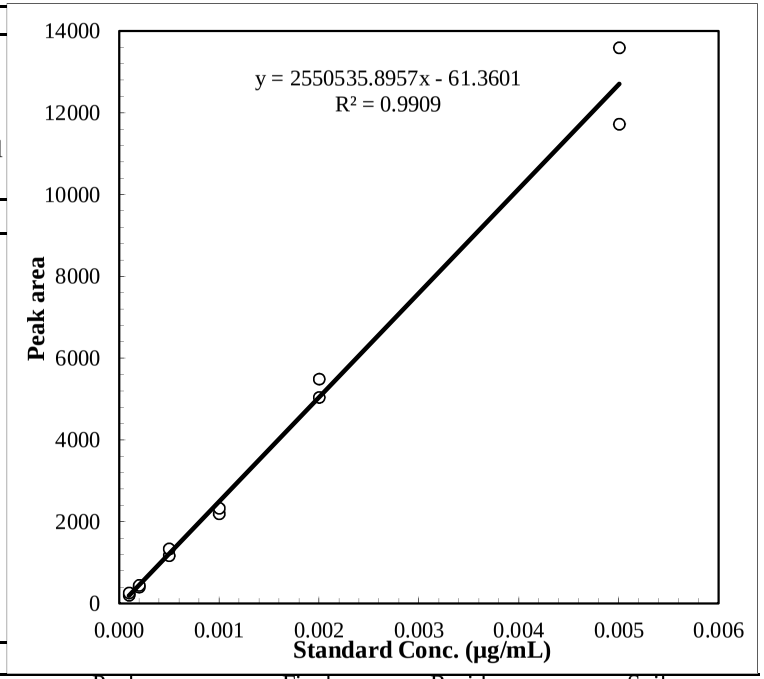
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNA	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 11-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-09	A307G-27	0.0001001	205
MV-10	A307G-26	0.0002002	402
MV-11	A307G-25	0.0005005	1172
MV-12	A307G-24	0.001001	2197
MV-13	A307G-23	0.0020020	5041
MV-14	A307G-22	0.005005	11723
MV-21	A307G-22	0.005005	13592
MV-22	A307G-23	0.0020020	5490
MV-23	A307G-24	0.001001	2332
MV-24	A307G-25	0.0005005	1338
MV-25	A307G-26	0.0002002	446
MV-26	A307G-27	0.0001001	260

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2550536
Intercept, b = -61.36
R^2 = 0.9909
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 11-Jul-12 Analysis date: 11-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-16	18476A-C-1	C	NA	2.50	NA	0	100	< 0.02	NA
MV-17	18476A-MV-0.02-1	F	0.0501	2.50	0.02002	1068	100	0.01771	88.47
MV-18	18476A-MV-0.02-2	F	0.0501	2.50	0.02002	1091	100	0.01807	90.27
MV-19	18476A-MV-0.02-3	F	0.0501	2.50	0.02002	1121	100	0.01854	92.62

NOTES

*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

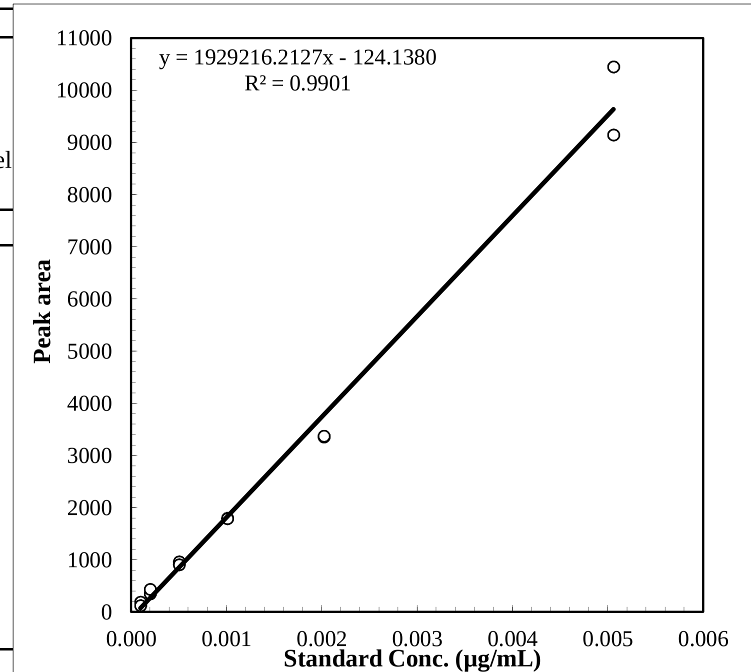
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNA-AM	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 12-Jul-12

RTSDB Page	Calibration standard ID (or name)	Calibration standard Conc.(μg/mL)	Peak Area
MV-35	A307G-27	0.0001012	190
MV-36	A307G-26	0.0002024	350
MV-37	A307G-25	0.0005060	961
MV-38	A307G-24	0.001012	1798
MV-39A	A307G-23	0.0020240	3356
MV-40	A307G-22	0.005060	9144
MV-47	A307G-22	0.005060	10449
MV-48	A307G-23	0.0020240	3371
MV-49	A307G-24	0.001012	1790
MV-50	A307G-25	0.0005060	906
MV-51	A307G-26	0.0002024	434
MV-52	A307G-27	0.0001012	123

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1929216
Intercept, b = -124.14
R^2 = 0.9901
LOQ (μg/g) = 0.02



ANALYTICAL DATA Extraction date: 12-Jul-12 Analysis date: 12-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (μg)	Sample Weight (g)	Spike Conc. (μg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (μg/g)*3	Spike Recovery (%)*4
MV-42	18476A-C-2	C	NA	2.50	NA	45	100	0.00351	NA
MV-43	18476A-MV-0.2-1	F	0.5060	2.50	0.2024	1718	500	0.1910	94.35
MV-44	18476A-MV-0.2-2	F	0.5060	2.50	0.2024	1598	500	0.1785	88.21
MV-45	18476A-MV-0.2-3	F	0.5060	2.50	0.2024	1674	500	0.1864	92.10

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

R Number: 09358 | Laboratory ID: 09358.11-MIR05
Chemical: Flonicamid | Field ID No. for MV: 09358.11-WI17
nalysis for: Flonicamid (IKI-220) | Field Research Director: Dr. Scott Chapman
ommodity: Mint | Analyst(s): Eina Abouzied&Lester Geissel
Crop part: Mint Oil | Instrument: UPLC/MS/MS

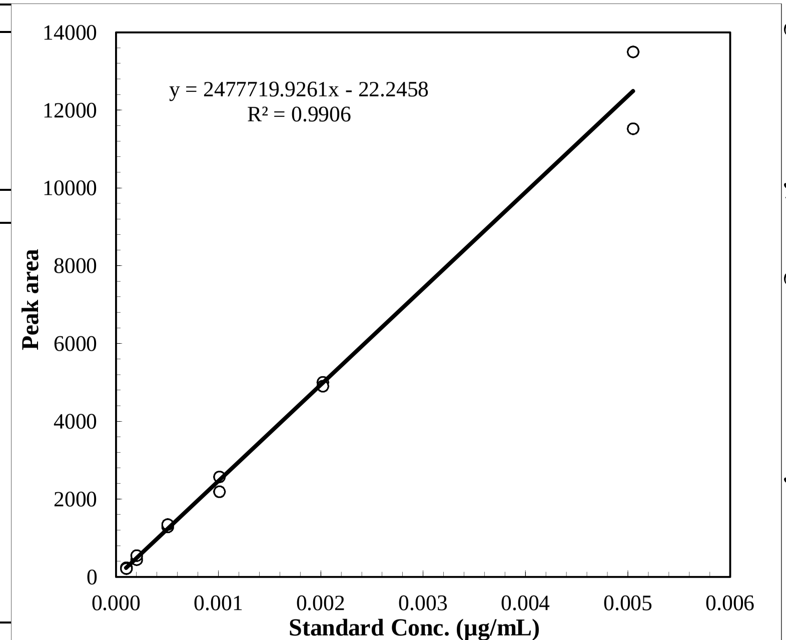
CALIBRATION DATA

Date of calibration analysis: 12-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-35	A307G-27	0.0001010	237
MV-36	A307G-26	0.0002020	444
MV-37	A307G-25	0.0005050	1288
MV-38	A307G-24	0.001010	2191
MV-39	A307G-23	0.0020200	5005
MV-40	A307G-22	0.005050	11524
MV-47	A307G-22	0.005050	13499
MV-48	A307G-23	0.0020200	4905
MV-49	A307G-24	0.001010	2572
MV-50	A307G-25	0.0005050	1348
MV-51	A307G-26	0.0002020	549
MV-52	A307G-27	0.0001010	215

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2477720
Intercept, b = -22.25
R^2 = 0.9906
LOQ (µg/g) = 0.02



ANALYTICAL DATA

Extraction date: 12-Jul-12 Analysis date: 12-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-42	18476A-C-2	C	NA	2.50	NA	0	100	< 0.02	NA
MV-43	18476A-MV-0.2-1	F	0.5050	2.50	0.2020	2316	500	0.1887	93.44
MV-44	18476A-MV-0.2-2	F	0.5050	2.50	0.2020	2188	500	0.1784	88.32
MV-45	18476A-MV-0.2-3	F	0.5050	2.50	0.2020	2440	500	0.1988	98.39

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

Attachment B: Calculation Worksheets, Standard Curves and LOD/LOQ Calculations

CALCULATION PAGE

PROJECT INFORMATION

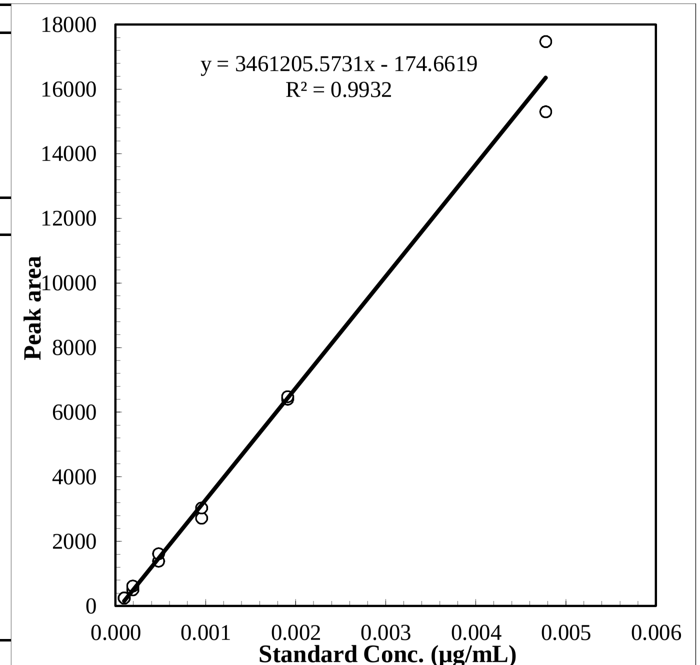
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WA*18
Analysis for:	TFNG	Field Research Director:	09358.11-WI18
Commodity:	Mint	Analyst(s):	Dr. Scott Chapman
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 12-Jul-12

RTSDB Page	Calibration standard ID (or name)	Calibration standard Conc.(µg/mL	Peak Area
MV-35	A307G-27	0.00009550	250
MV-36	A307G-26	0.0001910	506
MV-37	A307G-25	0.0004775	1392
MV-38	A307G-24	0.00095500	2725
MV-39	A307G-23	0.0019100	6405
MV-40	A307G-22	0.004775	15305
MV-47	A307G-22	0.004775	17477
MV-48	A307G-23	0.0019100	6484
MV-49	A307G-24	0.00095500	3040
MV-50	A307G-25	0.0004775	1623
MV-51	A307G-26	0.0001910	623
MV-52	A307G-27	0.00009550	250

---- ----

Type: Linear regression
Equation: y = mx + b
Slope, m = 3461206
Intercept, b = -174.66
R^2 = 0.9932
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 12-Jul-12 Analysis date: 12-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-42	18476A-C-2	C	NA	2.50	NA	0	100	< 0.02	NA
MV-43	18476A-MV-0.2-1	F	0.47750	2.50	0.1910	2961	500	0.1812	94.86
MV-44	18476A-MV-0.2-2	F	0.47750	2.50	0.1910	3014	500	0.1843	96.47
MV-45	18476A-MV-0.2-3	F	0.47750	2.50	0.1910	3135	500	0.1912	100.13

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

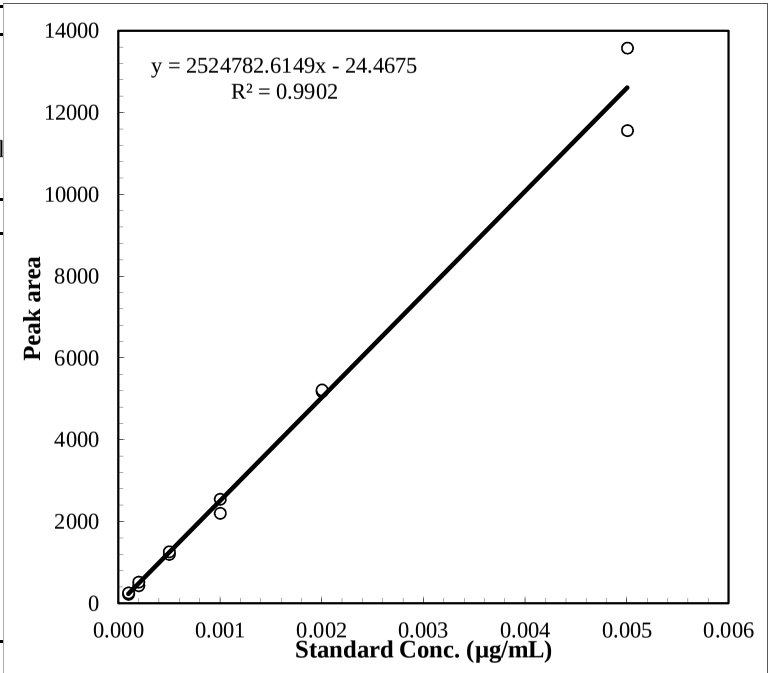
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNA	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 12-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-35	A307G-27	0.0001001	224
MV-36	A307G-26	0.0002002	432
MV-37	A307G-25	0.0005005	1199
MV-38	A307G-24	0.001001	2204
MV-39	A307G-23	0.0020020	5180
MV-40	A307G-22	0.005005	11562
MV-47	A307G-22	0.005005	13579
MV-48	A307G-23	0.0020020	5219
MV-49	A307G-24	0.001001	2548
MV-50	A307G-25	0.0005005	1262
MV-51	A307G-26	0.0002002	521
MV-52	A307G-27	0.0001001	257

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2524783
Intercept, b = -24.47
R^2 = 0.9902
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 12-Jul-12 Analysis date: 12-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-42	18476A-C-2	C	NA	2.50	NA	0	100	< 0.02	NA
MV-43	18476A-MV-0.2-1	F	0.5005	2.50	0.2002	2267	500	0.1815	90.67
MV-44	18476A-MV-0.2-2	F	0.5005	2.50	0.2002	2247	500	0.1799	89.88
MV-45	18476A-MV-0.2-3	F	0.5005	2.50	0.2002	2367	500	0.1894	94.63

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

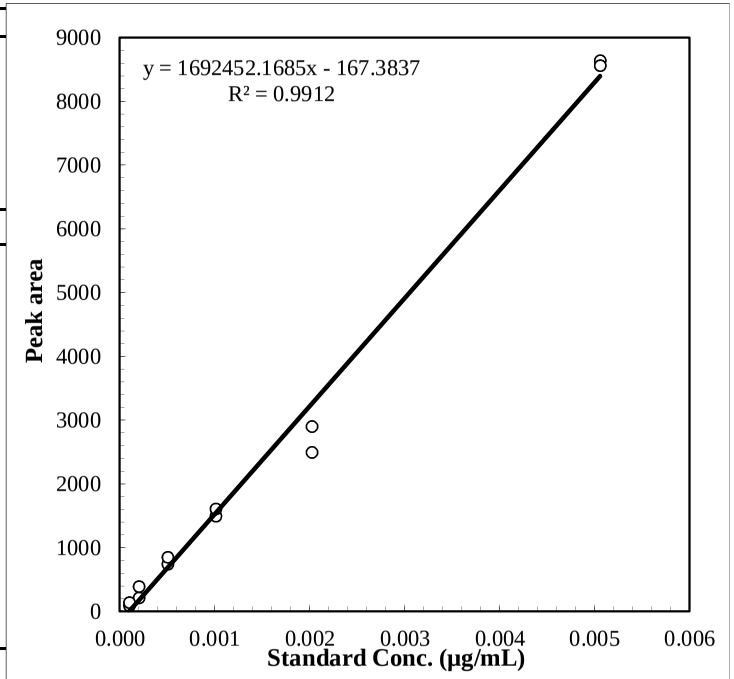
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNA-AM	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 19-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-61	A307G-27	0.0001012	91
MV-62	A307G-26	0.0002024	214
MV-63	A307G-25	0.0005060	743
MV-64	A307G-24	0.001012	1496
MV-65	A307G-23	0.0020240	2496
MV-66	A307G-22	0.005060	8639
MV-73	A307G-22	0.005060	8562
MV-74	A307G-23	0.0020240	2902
MV-75	A307G-24	0.001012	1609
MV-76	A307G-25	0.0005060	851
MV-77A	A307G-26	0.0002024	392
MV-78	A307G-27	0.0001012	141

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1692452
Intercept, b = -167.38
R^2 = 0.9912
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 16-Jul-12 Analysis date: 19-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-68	18476A-C-3	C	NA	2.50	NA	0	100	< 0.02	NA
MV-69	18476A-MV-2-1	F	5.060	2.50	2.024	2851	2500	1.783	88.11
MV-70	18476A-MV-2-2	F	5.060	2.50	2.024	2933	2500	1.832	90.51
MV-71	18476A-MV-2-3	F	5.060	2.50	2.024	2757	2500	1.728	85.37

NOTES *1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

R Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
nalysis for:	Flonicamid (IKI-220)	Field Research Director:	Dr. Scott Chapman
ommodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

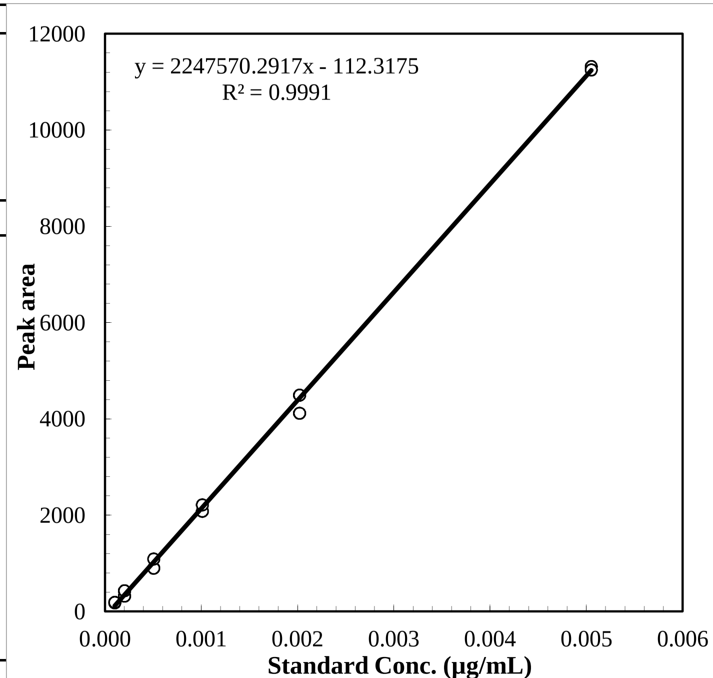
CALIBRATION DATA

Date of calibration analysis: 19-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-61	A307G-27	0.0001010	181
MV-62	A307G-26	0.0002020	321
MV-63	A307G-25	0.0005050	901
MV-64	A307G-24	0.001010	2083
MV-65	A307G-23	0.0020200	4120
MV-66	A307G-22	0.005050	11322
MV-73	A307G-22	0.005050	11253
MV-74	A307G-23	0.0020200	4495
MV-75	A307G-24	0.001010	2216
MV-76	A307G-25	0.0005050	1092
MV-77	A307G-26	0.0002020	431
MV-78	A307G-27	0.0001010	190

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2247570
Intercept, b = -112.32
R^2 = 0.9991
LOQ (µg/g) = 0.02



ANALYTICAL DATA

Extraction date: 16-Jul-12 Analysis date: 19-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-68	18476A-C-3	C	NA	2.50	NA	29	100	0.00252	NA
MV-69	18476A-MV-2-1	F	5.050	2.50	2.020	3998	2500	1.829	90.53
MV-70	18476A-MV-2-2	F	5.050	2.50	2.020	3827	2500	1.753	86.77
MV-71	18476A-MV-2-3	F	5.050	2.50	2.020	3666	2500	1.681	83.22

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

Attachment B: Calculation Worksheets, Standard Curves and LOD/LOQ Calculations

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNG	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

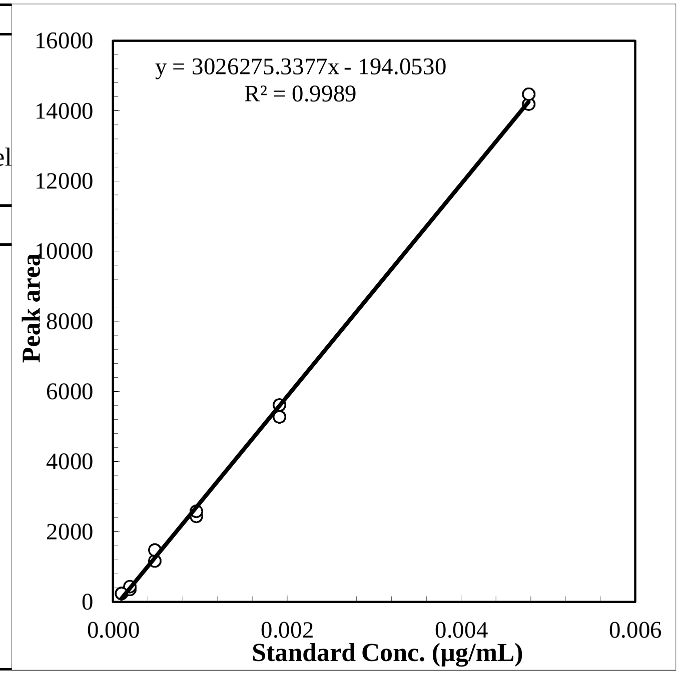
CALIBRATION DATA

Date of calibration analysis: 19-Jul-12

RTSDB Page	Calibration standard ID (or name)	Calibration standard Conc.(µg/mL	Peak Area
MV-61	A307G-27	0.00009550	244
MV-62	A307G-26	0.0001910	359
MV-63	A307G-25	0.0004775	1171
MV-64	A307G-24	0.00095500	2442
MV-65	A307G-23	0.0019100	5277
MV-66	A307G-22	0.004775	14195
MV-73	A307G-22	0.004775	14479
MV-74	A307G-23	0.0019100	5618
MV-75	A307G-24	0.00095500	2587
MV-76	A307G-25	0.0004775	1483
MV-77	A307G-26	0.0001910	439
MV-78	A307G-27	0.00009550	243

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 3026275
ntercept, b = -194.05
R^2 = 0.9989
OQ (µg/g) = 0.02



ANALYTICAL DATA

Extraction date: 16-Jul-12 Analysis date: 19-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-68	18476A-C-3	C	NA	2.50	NA	75	100	0.00356	NA
MV-69	18476A-MV-2-1	F	4.775	2.50	1.910	5339	2500	1.828	95.72
MV-70	18476A-MV-2-2	F	4.775	2.50	1.910	5335	2500	1.827	95.66
MV-71	18476A-MV-2-3	F	4.775	2.50	1.910	5122	2500	1.757	91.97

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

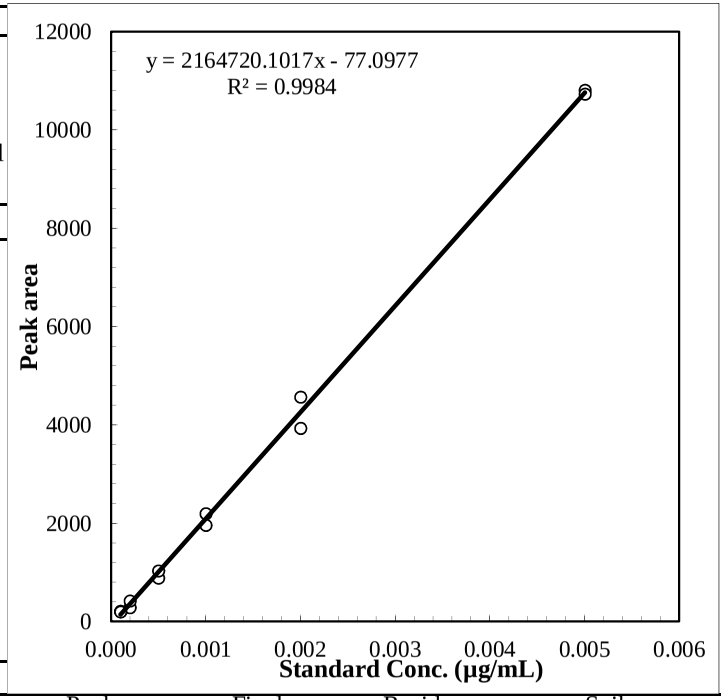
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNA	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 19-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-61	A307G-27	0.0001001	211
MV-62	A307G-26	0.0002002	285
MV-63	A307G-25	0.0005005	885
MV-64	A307G-24	0.001001	1959
MV-65	A307G-23	0.0020020	3931
MV-66	A307G-22	0.005005	10806
MV-73	A307G-22	0.005005	10728
MV-74	A307G-23	0.0020020	4566
MV-75	A307G-24	0.001001	2194
MV-76	A307G-25	0.0005005	1030
MV-77	A307G-26	0.0002002	420
MV-78	A307G-27	0.0001001	197

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2164720
Intercept, b = -77.10
R^2 = 0.9984
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 16-Jul-12 Analysis date: 19-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-68	18476A-C-3	C	NA	2.50	NA	20	100	0.00179	NA
MV-69	18476A-MV-2-1	F	5.005	2.50	2.002	4057	2500	1.910	95.39
MV-70	18476A-MV-2-2	F	5.005	2.50	2.002	3663	2500	1.728	86.30
MV-71	18476A-MV-2-3	F	5.005	2.50	2.002	3594	2500	1.696	84.71

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

PR Number: 09358
Chemical: Flonicamid
Analysis for: TFNA-AM
Commodity: Mint
Crop part: Mint Tops (leaves&stems)

Laboratory ID: 09358.11-MIR05
Field ID No. for MV: 09358.11-WI17
Field Research Director: Dr. Scott Chapman
Analyst(s): Eina Abouzied&Lester Geissel
Instrument: UPLC/MS/MS

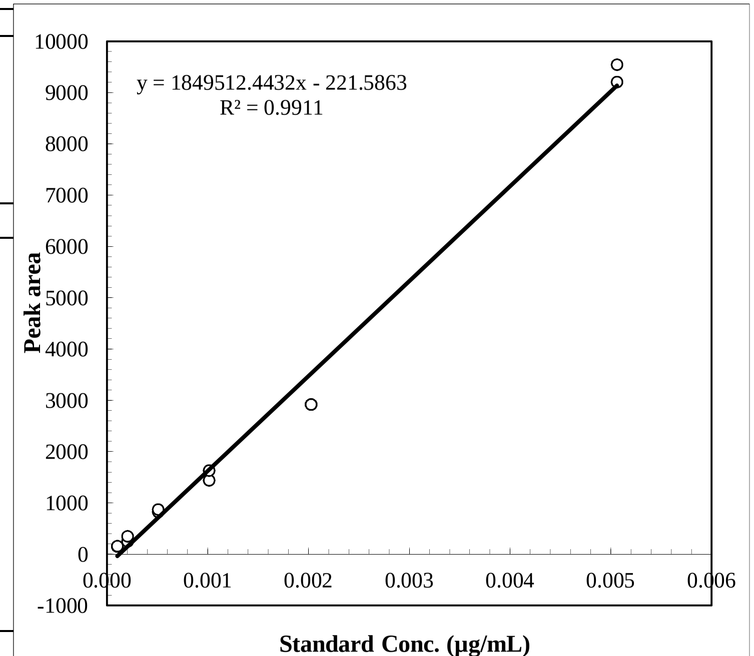
CALIBRATION DATA

Date of calibration analysis: 19-Jul-12

RTSDB Page	Calibration standard ID (or name)	Calibration standard Conc.(µg/mL)	Peak Area
MV-87	A307G-27	0.0001012	152
MV-88	A307G-26	0.0002024	251
MV-89	A307G-25	0.0005060	827
MV-90	A307G-24	0.001012	1442
MV-91	A307G-23	0.0020240	2921
MV-92	A307G-22	0.005060	9546
MV-99	A307G-22	0.005060	9207
MV-100A	A307G-23	0.0020240	2921
MV-101	A307G-24	0.001012	1633
MV-102	A307G-25	0.0005060	873
MV-103	A307G-26	0.0002024	351
MV-104	A307G-27	0.0001012	159

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1849512
Intercept, b = -221.59
R^2 = 0.9911
LOQ (µg/g) = 0.02



ANALYTICAL DATA

Extraction date: 17-Jul-12 Analysis date: 19-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-94	18472A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
MV-95	18472A-MV-0.02-1	F	0.1012	5.00	0.0202	469	200	0.01494	73.79
MV-96	18472A-MV-0.02-2	F	0.1012	5.00	0.0202	448	200	0.01448	71.55
MV-97	18472A-MV-0.02-3	F	0.1012	5.00	0.0202	431	200	0.01411	69.73

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	Flonicamid (IKI-220)	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

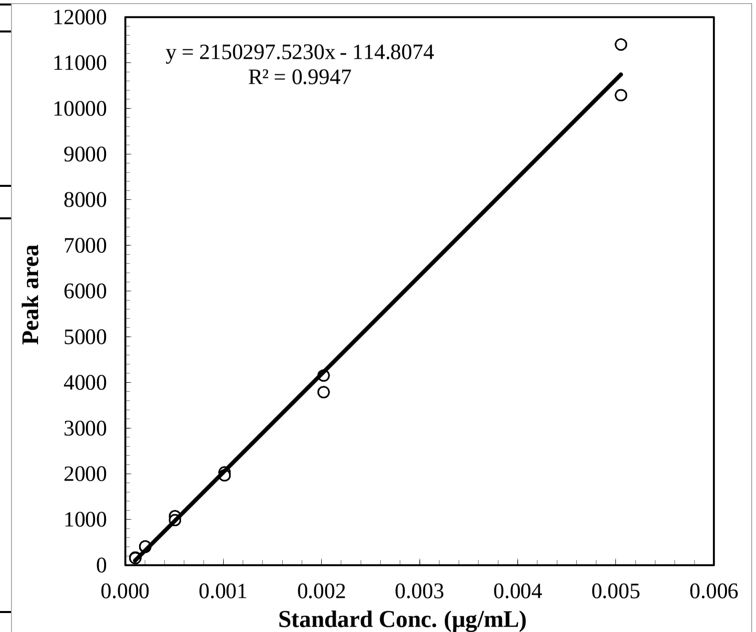
CALIBRATION DATA

Date of calibration analysis: 19-Jul-12

RTSDB Page	Calibration standard ID (or name)	Calibration standard Conc.(µg/mL)	Peak Area
MV-87	A307G-27	0.0001010	168
MV-88	A307G-26	0.0002020	403
MV-89	A307G-25	0.0005050	1075
MV-90	A307G-24	0.001010	2034
MV-91	A307G-23	0.0020200	4154
MV-92	A307G-22	0.005050	11402
MV-99	A307G-22	0.005050	10292
MV-100	A307G-23	0.0020200	3791
MV-101	A307G-24	0.001010	1969
MV-102	A307G-25	0.0005050	989
MV-103	A307G-26	0.0002020	412
MV-104	A307G-27	0.0001010	157

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2150298
Intercept, b = -114.81
R^2 = 0.9947
LOQ (µg/g) = 0.02



ANALYTICAL DATA

Extraction date: 17-Jul-12 Analysis date: 19-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-94	18472A-C-1	C	NA	5.00	NA	94	200	0.00388	NA
MV-95	18472A-MV-0.02-1	F	0.1010	5.00	0.0202	684	200	0.01486	73.56
MV-96	18472A-MV-0.02-2	F	0.1010	5.00	0.0202	615	200	0.01358	67.21
MV-97	18472A-MV-0.02-3	F	0.1010	5.00	0.0202	655	200	0.01432	70.89

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNG	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

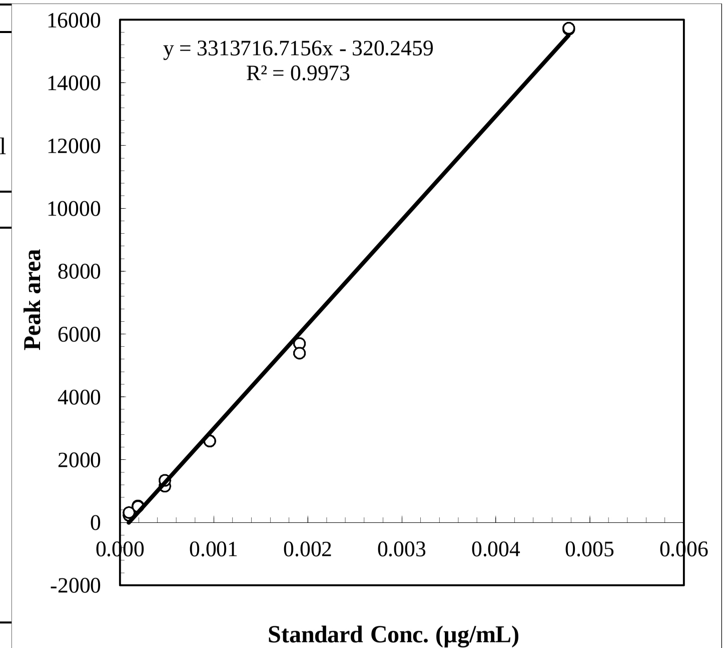
CALIBRATION DATA

Date of calibration analysis: 19-Jul-12

RTSDB Page	Calibration standard ID (or name)	Calibration standard Conc.(µg/mL)	Peak Area
MV-87	A307G-27	0.00009550	235
MV-88	A307G-26	0.0001910	534
MV-89	A307G-25	0.0004775	1162
MV-90	A307G-24	0.00095500	2600
MV-91	A307G-23	0.0019100	5700
MV-92	A307G-22	0.004775	15707
MV-99	A307G-22	0.004775	15739
MV-100	A307G-23	0.0019100	5395
MV-101	A307G-24	0.00095500	2599
MV-102	A307G-25	0.0004775	1346
MV-103	A307G-26	0.0001910	514
MV-104A	A307G-27	0.00009550	323

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 3313717
Intercept, b = -320.25
R^2 = 0.9973
LOQ (µg/g) = 0.02



ANALYTICAL DATA

Extraction date: 17-Jul-12 Analysis date: 19-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-94	18472A-C-1	C	NA	5.00	NA	455	200	0.00936	NA
MV-95	18472A-MV-0.02-1	F	0.0955	5.00	0.0191	1414	200	0.02093	109.60
MV-96	18472A-MV-0.02-2	F	0.0955	5.00	0.0191	1376	200	0.02048	107.20
MV-97	18472A-MV-0.02-3	F	0.0955	5.00	0.0191	1460	200	0.02149	112.51

NOTES

*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

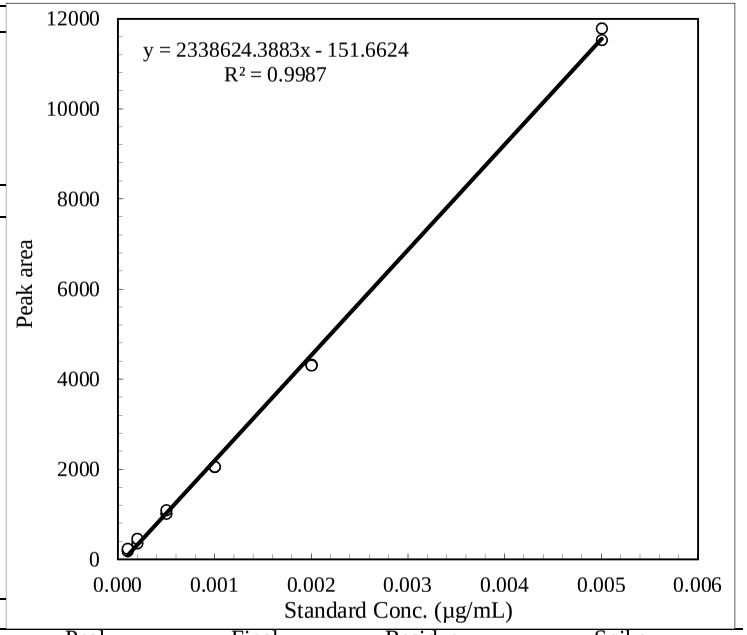
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNA	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 19-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-87	A307G-27	0.0001001	183
MV-88	A307G-26	0.0002002	357
MV-89	A307G-25	0.0005005	1012
MV-90	A307G-24	0.001001	2050
MV-91	A307G-23	0.0020020	4320
MV-92	A307G-22	0.005005	11787
MV-99	A307G-22	0.005005	11529
MV-100	A307G-23	0.0020020	4305
MV-101	A307G-24	0.001001	2057
MV-102	A307G-25	0.0005005	1092
MV-103	A307G-26	0.0002002	455
MV-104	A307G-27	0.0001001	234

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2338624
Intercept, b = -151.66
R^2 = 0.9987
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 17-Jul-12 Analysis date: 19-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-94	18472A-C-1	C	NA	5.00	NA	56	200	0.00355	NA
MV-95	18472A-MV-0.02-1	F	0.1001	5.00	0.02002	1049	200	0.02054	102.58
MV-96	18472A-MV-0.02-2	F	0.1001	5.00	0.02002	1068	200	0.02086	104.20
MV-97	18472A-MV-0.02-3	F	0.1001	5.00	0.02002	1103	200	0.02146	107.19

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

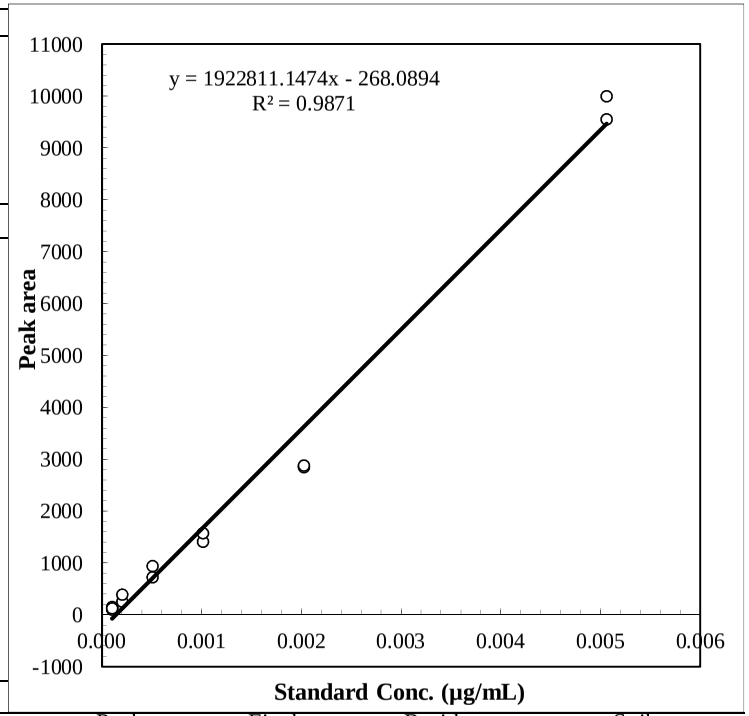
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNA-AM	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 20-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-118	A307G-27	0.0001012	152
MV-119	A307G-26	0.0002024	256
MV-120A	A307G-25	0.0005060	726
MV-121	A307G-24	0.001012	1411
MV-122	A307G-23	0.0020240	2848
MV-126	A307G-27	0.0001012	102
MV-137	A307G-22	0.005060	9553
MV-138	A307G-23	0.0020240	2879
MV-139	A307G-24	0.001012	1574
MV-140	A307G-25	0.0005060	941
MV-141	A307G-26	0.0002024	391
MV-142	A307G-27	0.0001012	126
MV-143	A307G-22	0.005060	9998

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1922811
Intercept, b = -268.09
R^2 = 0.9871
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 18-Jul-12 Analysis date: 20-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-127	18472A-C-2	C	NA	5.00	NA	0	200	< 0.02	NA
MV-128	18472A-MV-0.2-1	F	1.012	5.00	0.2024	1482	1000	0.1820	89.94
MV-129	18472A-MV-0.2-2	F	1.012	5.00	0.2024	1381	1000	0.1715	84.75
MV-130	18472A-MV-0.2-3	F	1.012	5.00	0.2024	1413	1000	0.1749	86.39

NOTES *1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

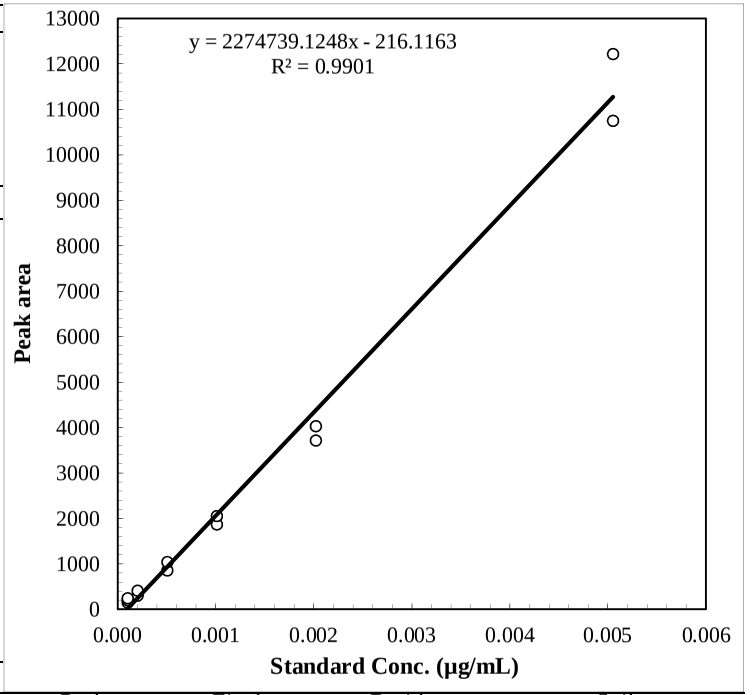
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	Flonicamid (IKI-220)	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 20-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-118	A307G-27	0.0001010	149
MV-119	A307G-26	0.0002020	304
MV-120	A307G-25	0.0005050	858
MV-121	A307G-24	0.001010	1869
MV-122	A307G-23	0.0020200	3717
MV-126	A307G-27	0.0001010	204
MV-137	A307G-22	0.005050	10752
MV-138	A307G-23	0.0020200	4030
MV-139	A307G-24	0.001010	2054
MV-140	A307G-25	0.0005050	1043
MV-141	A307G-26	0.0002020	411
MV-142	A307G-27	0.0001010	246
MV-143	A307G-22	0.005050	12219

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2274739
Intercept, b = -216.12
R^2 = 0.9901
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 18-Jul-12 Analysis date: 20-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-127	18472A-C-2	C	NA	5.00	NA	122	200	0.005946	NA
MV-128	18472A-MV-0.2-1	F	1.010	5.00	0.2020	1544	1000	0.1548	76.61
MV-129	18472A-MV-0.2-2	F	1.010	5.00	0.2020	1668	1000	0.1657	82.01
MV-130	18472A-MV-0.2-3	F	1.010	5.00	0.2020	1581	1000	0.1580	78.22

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

CALCULATION PAGE

PROJECT INFORMATION

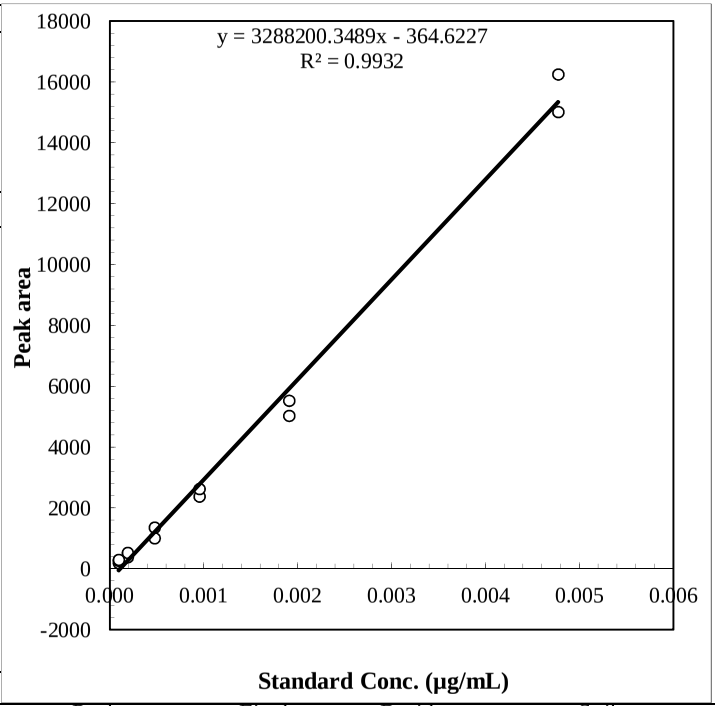
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNG	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 20-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-118	A307G-27	0.00009550	188
MV-119	A307G-26	0.0001910	383
MV-120	A307G-25	0.0004775	1006
MV-121	A307G-24	0.00095500	2377
MV-122	A307G-23	0.0019100	5029
MV-126	A307G-27	0.00009550	264
MV-137	A307G-22	0.004775	15015
MV-138	A307G-23	0.0019100	5526
MV-139	A307G-24	0.00095500	2626
MV-140	A307G-25	0.0004775	1356
MV-141	A307G-26	0.0001910	526
MV-142	A307G-27	0.00009550	296
MV-143	A307G-22	0.004775	16250

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 3288200
Intercept, b = -364.62
R^2 = 0.9932
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 18-Jul-12 Analysis date: 20-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-127	18472A-C-2	C	NA	5.00	NA	451	200	0.00992	NA
MV-128	18472A-MV-0.2-1	F	0.9550	5.00	0.1910	2687	1000	0.1856	97.18
MV-129	18472A-MV-0.2-2	F	0.9550	5.00	0.1910	2649	1000	0.1833	95.97
MV-130	18472A-MV-0.2-3	F	0.9550	5.00	0.1910	2647	1000	0.1832	95.90

NOTES

*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

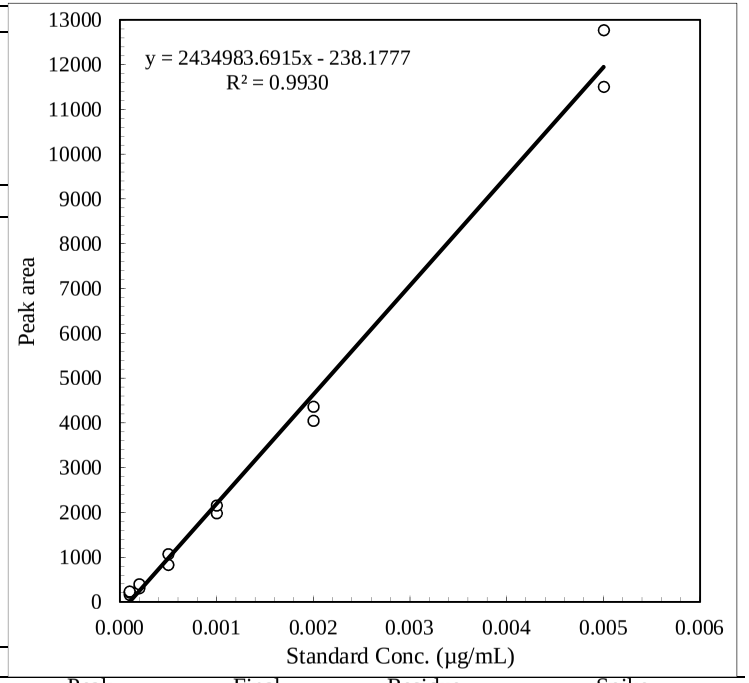
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNA	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 20-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-118	A307G-27	0.0001001	162
MV-119	A307G-26	0.0002002	308
MV-120	A307G-25	0.0005005	830
MV-121	A307G-24	0.001001	1983
MV-122	A307G-23	0.0020020	4048
MV-126	A307G-27	0.0001001	213
MV-137	A307G-22	0.005005	11508
MV-138	A307G-23	0.0020020	4362
MV-139	A307G-24	0.001001	2156
MV-140	A307G-25	0.0005005	1070
MV-141	A307G-26	0.0002002	398
MV-142	A307G-27	0.0001001	235
MV-143	A307G-22	0.005005	12773

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2434984
Intercept, b = -238.18
R^2 = 0.9930
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 18-Jul-12 Analysis date: 20-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-127	18472A-C-2	C	NA	5.00	NA	100	200	0.00556	NA
MV-128	18472A-MV-0.2-1	F	1.001	5.00	0.2002	2292	1000	0.2078	103.81
MV-129	18472A-MV-0.2-2	F	1.001	5.00	0.2002	2438	1000	0.2198	109.80
MV-130	18472A-MV-0.2-3	F	1.001	5.00	0.2002	2282	1000	0.2070	103.40

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

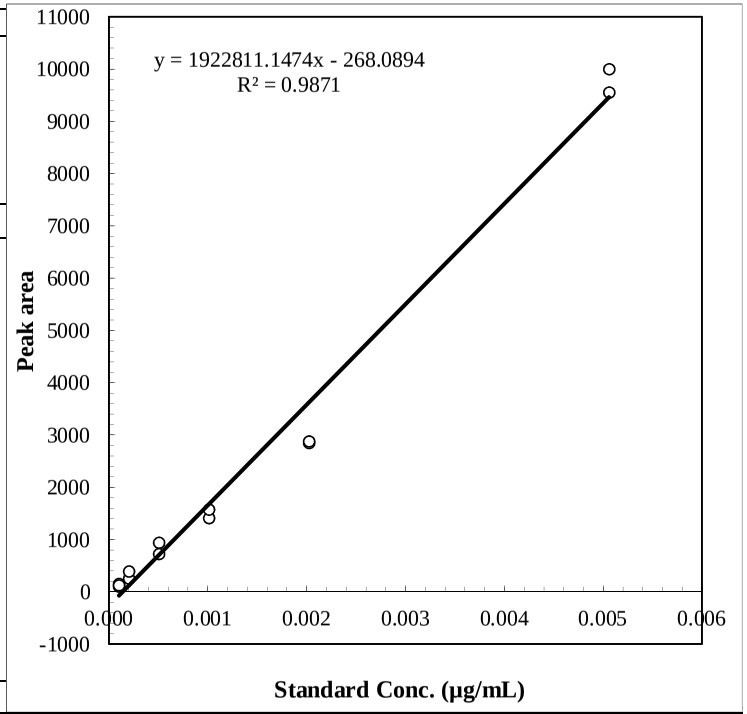
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNA-AM	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 20-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-118	A307G-27	0.0001012	152
MV-119	A307G-26	0.0002024	256
MV-120A	A307G-25	0.0005060	726
MV-121	A307G-24	0.001012	1411
MV-122	A307G-23	0.0020240	2848
MV-126	A307G-27	0.0001012	102
MV-137	A307G-22	0.005060	9553
MV-138	A307G-23	0.0020240	2879
MV-139	A307G-24	0.001012	1574
MV-140	A307G-25	0.0005060	941
MV-141	A307G-26	0.0002024	391
MV-142	A307G-27	0.0001012	126
MV-143	A307G-22	0.005060	9998

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1922811
Intercept, b = -268.09
R^2 = 0.9871
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 19-Jul-12 Analysis date: 20-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-132	18472A-C-3	C	NA	5.00	NA	0	200	< 0.02	NA
MV-133	18472A-MV-2-1	F	10.12	5.00	2.024	2587	5000	1.485	73.36
MV-134	18472A-MV-2-2	F	10.12	5.00	2.024	2975	5000	1.687	83.33
MV-135	18472A-MV-2-3	F	10.12	5.00	2.024	3132	5000	1.768	87.37

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

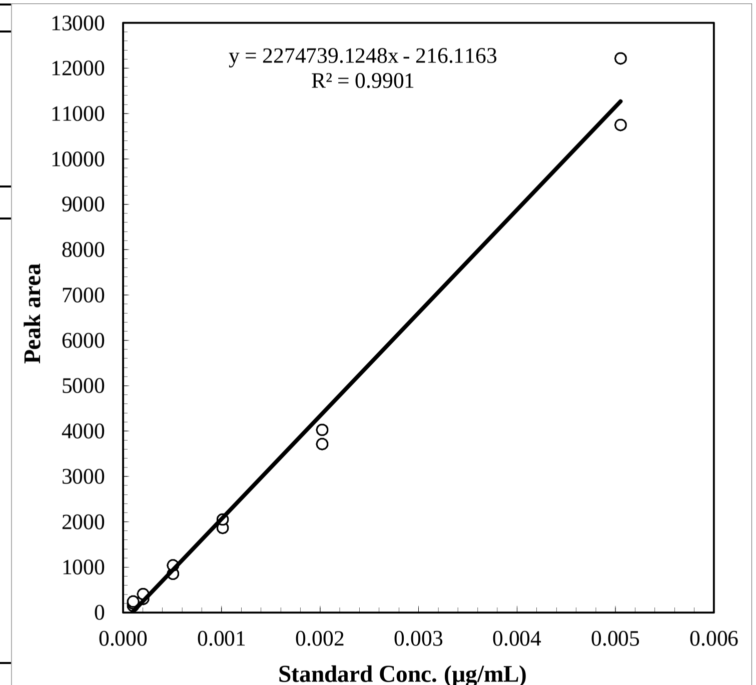
PR Number: 09358
Chemical: Flonicamid
Analysis for: Flonicamid (IKI-220)
Commodity: Mint
Crop part: Mint Tops (leaves&stems)
Laboratory ID: 09358.11-MIR05
Field ID No. for MD: 09358.11-WI17
Field Research Director: Dr. Scott Chapman
Analyst(s): Eina Abouzied&Lester Geissel
Instrument: UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 20-Jul-12

RTSDB Page	Calibration standard ID (or name)	Calibration standard Conc.(µg/mL)	Peak Area
MV-118	A307G-27	0.0001010	149
MV-119	A307G-26	0.0002020	304
MV-120	A307G-25	0.0005050	858
MV-121	A307G-24	0.001010	1869
MV-122	A307G-23	0.0020200	3717
MV-126	A307G-27	0.0001010	204
MV-137	A307G-22	0.005050	10752
MV-138	A307G-23	0.0020200	4030
MV-139	A307G-24	0.001010	2054
MV-140	A307G-25	0.0005050	1043
MV-141	A307G-26	0.0002020	411
MV-142	A307G-27	0.0001010	246
MV-143	A307G-22	0.005050	12219

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2274739
Intercept, b = -216.12
R^2 = 0.9901
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 19-Jul-12 Analysis date: 20-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-132	18472A-C-3	C	NA	5.00	NA	72	200	0.005066	NA
MV-133	18472A-MV-2-1	F	10.10	5.00	2.020	3142	5000	1.476	73.08
MV-134	18472A-MV-2-2	F	10.10	5.00	2.020	3267	5000	1.531	75.80
MV-135	18472A-MV-2-3	F	10.10	5.00	2.020	3666	5000	1.707	84.49

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNG	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

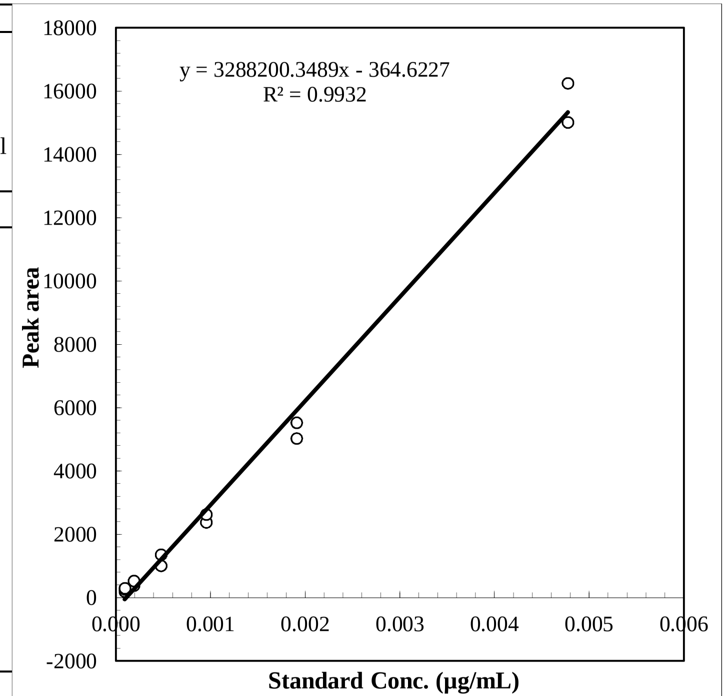
CALIBRATION DATA

Date of calibration analysis: 20-Jul-12

RTSDB Page	Calibration standard ID (or name)	Calibration standard Conc.(µg/mL)	Peak Area
MV-118	A307G-27	0.00009550	188
MV-119	A307G-26	0.0001910	383
MV-120	A307G-25	0.0004775	1006
MV-121	A307G-24	0.00095500	2377
MV-122	A307G-23	0.0019100	5029
MV-126	A307G-27	0.00009550	264
MV-137	A307G-22	0.004775	15015
MV-138	A307G-23	0.0019100	5526
MV-139	A307G-24	0.00095500	2626
MV-140	A307G-25	0.0004775	1356
MV-141	A307G-26	0.0001910	526
MV-142	A307G-27	0.00009550	296
MV-143	A307G-22	0.004775	16250

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 3288200
Intercept, b = -364.62
R^2 = 0.9932
LOQ (µg/g) = 0.02



ANALYTICAL DATA

Extraction date: 19-Jul-12 Analysis date: 20-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-132	18472A-C-3	C	NA	5.00	NA	465	200	0.01009	NA
MV-133	18472A-MV-2-1	F	9.550	5.00	1.910	4985	5000	1.627	85.18
MV-134A	18472A-MV-2-2	F	9.550	5.00	1.910	5315	5000	1.727	90.43
MV-135	18472A-MV-2-3	F	9.550	5.00	1.910	5693	5000	1.842	96.45

NOTES

*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

Attachment B: Calculation Worksheets, Standard Curves and LOD/LOQ Calculations

CALCULATION PAGE

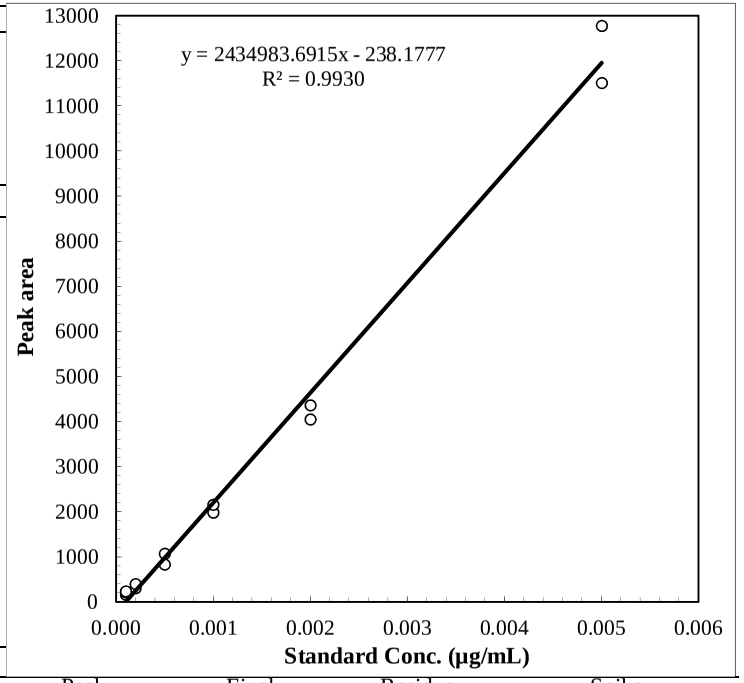
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No. for MV:	09358.11-WI17
Analysis for:	TFNA	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 20-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
MV-118	A307G-27	0.0001001	162
MV-119	A307G-26	0.0002002	308
MV-120	A307G-25	0.0005005	830
MV-121	A307G-24	0.001001	1983
MV-122	A307G-23	0.0020020	4048
MV-126	A307G-27	0.0001001	213
MV-137	A307G-22	0.005005	11508
MV-138	A307G-23	0.0020020	4362
MV-139	A307G-24	0.001001	2156
MV-140	A307G-25	0.0005005	1070
MV-141	A307G-26	0.0002002	398
MV-142	A307G-27	0.0001001	235
MV-143	A307G-22	0.005005	12773

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2434984
Intercept, b = -238.18
R^2 = 0.9930
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 19-Jul-12 Analysis date: 20-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
MV-132	18472A-C-3	C	NA	5.00	NA	99	200	0.00554	NA
MV-133	18472A-MV-2-1	F	10.01	5.00	2.002	4219	5000	1.830	91.43
MV-134	18472A-MV-2-2	F	10.01	5.00	2.002	4370	5000	1.892	94.53
MV-135	18472A-MV-2-3	F	10.01	5.00	2.002	4892	5000	2.107	105.24

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Method Validation

CALCULATION PAGE

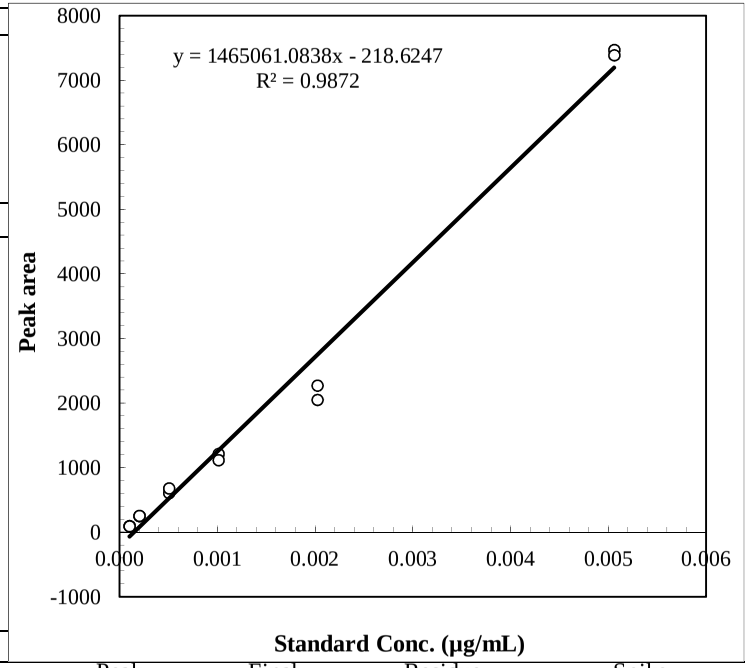
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WI17
Analysis for:	TFNA-AM	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 02-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WI17-23	A307G-27	0.0001012	95
WI17-24	A307G-26	0.0002024	248
WI17-25	A307G-25	0.0005060	605
WI17-26	A307G-24	0.001012	1212
WI17-27A	A307G-23	0.0020240	2049
WI17-28	A307G-22	0.005060	7468
WI17-40	A307G-22	0.005060	7387
WI17-41	A307G-23	0.0020240	2270
WI17-42	A307G-24	0.001012	1115
WI17-43	A307G-25	0.0005060	678
WI17-44	A307G-26	0.0002024	253
WI17-45	A307G-27	0.0001012	91

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1465061
Intercept, b = -218.62
R^2 = 0.9872
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 31-Jul-12 Analysis date: 02-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WI17-30	18473A-C-1	C	NA	5.00	NA	48	200	0.00728	NA
WI17-31	18473A-C-1	C	NA	5.00	NA	61	200	0.00763	NA
WI17-32	18473A-QC-0.02-1	F	0.1012	5.00	0.02024	324	200	0.01482	73.20
WI17-33	18473A-QC-0.02-1	F	0.1012	5.00	0.02024	310	200	0.01443	71.31
WI17-34	18474A-T-1	T		5.00		295	1000	0.07012	
WI17-35	18474A-T-1	T		5.00		327	1000	0.07448	
WI17-36	18475A-T-1	T		5.00		361	1000	0.07913	
WI17-37	18475A-T-1	T		5.00		304	1000	0.07135	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WI17
Analysis for:	Flonicamid (IKI-220)	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 02-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WI17-23	A307G-27	0.0001010	219
WI17-24	A307G-26	0.0002020	385
WI17-25	A307G-25	0.0005050	1028
WI17-26	A307G-24	0.001010	2121
WI17-27	A307G-23	0.0020200	4110
WI17-28	A307G-22	0.005050	11033
WI17-40	A307G-22	0.005050	10590
WI17-41	A307G-23	0.0020200	4415
WI17-42	A307G-24	0.001010	2078
WI17-43	A307G-25	0.0005050	1136
WI17-44	A307G-26	0.0002020	433
WI17-45	A307G-27	0.0001010	263

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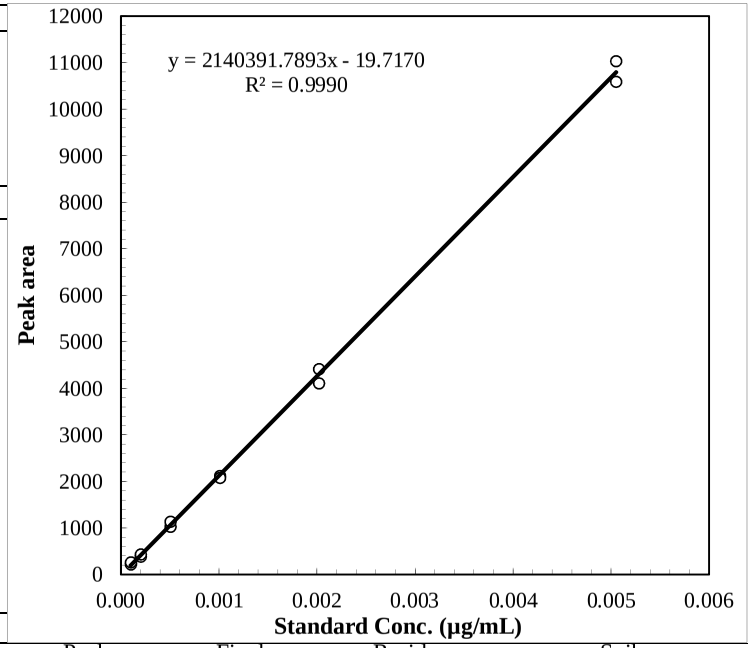
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2140392
Intercept, b = -19.72
R^2 = 0.9990
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 31-Jul-12 Analysis date: 02-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WI17-30	18473A-C-1	C	NA	5.00	NA	37	200	0.00106	NA
WI17-31	18473A-C-1	C	NA	5.00	NA	6	200	0.000481	NA
WI17-32	18473A-QC-0.02-1	F	0.1010	5.00	0.02020	613	200	0.01182	58.54
WI17-33	18473A-QC-0.02-1	F	0.1010	5.00	0.02020	621	200	0.01197	59.28
WI17-34	18474A-T-1	T		5.00		5146	1000	0.4827	
WI17-35	18474A-T-1	T		5.00		5522	1000	0.5178	
WI17-36	18475A-T-1	T		5.00		5235	1000	0.4910	
WI17-37	18475A-T-1	T		5.00		5520	1000	0.5176	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

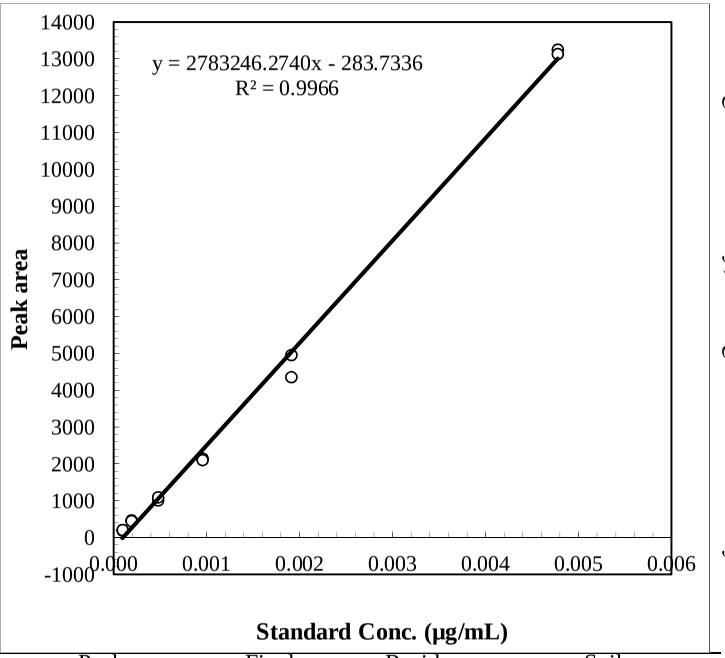
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WI17
Analysis for:	TFNG	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 02-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WI17-23	A307G-27	0.00009550	191
WI17-24	A307G-26	0.0001910	468
WI17-25	A307G-25	0.0004775	1013
WI17-26A	A307G-24	0.00095500	2147
WI17-27	A307G-23	0.0019100	4358
WI17-28	A307G-22	0.004775	13251
WI17-40	A307G-22	0.004775	13133
WI17-41	A307G-23	0.0019100	4958
WI17-42	A307G-24	0.00095500	2105
WI17-43	A307G-25	0.0004775	1094
WI17-44A	A307G-26	0.0001910	451
WI17-45A	A307G-27	0.00009550	207

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2783246
Intercept, b = -283.73
R^2 = 0.9966
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 31-Jul-12 Analysis date: 02-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WI17-30	18473A-C-1	C	NA	5.00	NA	514	200	0.0115	NA
WI17-31	18473A-C-1	C	NA	5.00	NA	474	200	0.0109	NA
WI17-32	18473A-QC-0.02-1	F	0.09550	5.00	0.01910	1243	200	0.02194	114.88
WI17-33	18473A-QC-0.02-1	F	0.09550	5.00	0.01910	1259	200	0.02217	116.08
WI17-34	18474A-T-1	T		5.00		2745	1000	0.2176	
WI17-35	18474A-T-1	T		5.00		2793	1000	0.2211	
WI17-36	18475A-T-1	T		5.00		2842	1000	0.2246	
WI17-37	18475A-T-1	T		5.00		2857	1000	0.2257	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

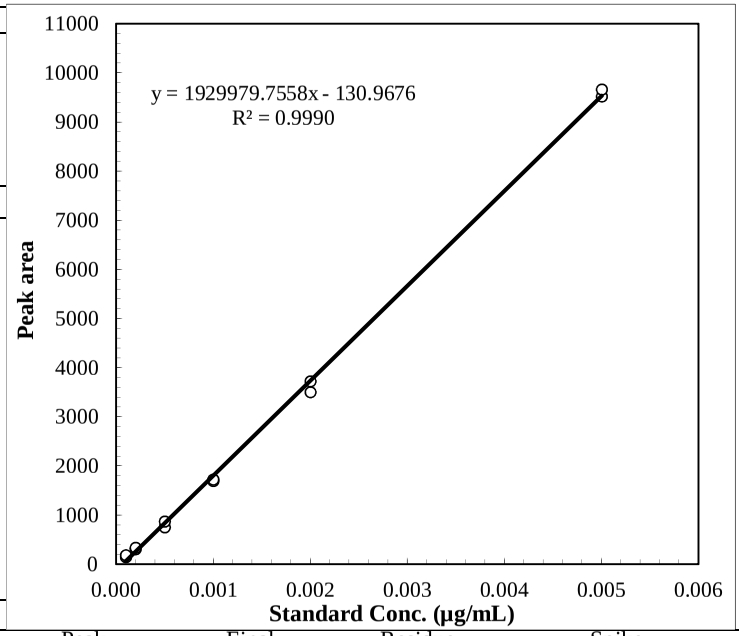
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WI17
Analysis for:	TFNA	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 02-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WI17-23	A307G-27	0.0001001	147
WI17-24	A307G-26	0.0002002	306
WI17-25	A307G-25	0.0005005	753
WI17-26	A307G-24	0.001001	1696
WI17-27	A307G-23	0.0020020	3503
WI17-28	A307G-22	0.005005	9521
WI17-40	A307G-22	0.005005	9663
WI17-41	A307G-23	0.0020020	3724
WI17-42	A307G-24	0.001001	1725
WI17-43	A307G-25	0.0005005	871
WI17-44	A307G-26	0.0002002	337
WI17-45	A307G-27	0.0001001	184

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1929980
Intercept, b = -130.97
R^2 = 0.9990
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 31-Jul-12 Analysis date: 02-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WI17-30	18473A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
WI17-31	18473A-C-1	C	NA	5.00	NA	80	200	0.00437	NA
WI17-32	18473A-QC-0.02-1	F	0.1001	5.00	0.02002	833	200	0.01998	99.79
WI17-33	18473A-QC-0.02-1	F	0.1001	5.00	0.02002	694	200	0.01710	85.40
WI17-34	18474A-T-1	T		5.00		919	1000	0.1088	
WI17-35	18474A-T-1	T		5.00		892	1000	0.1060	
WI17-36	18475A-T-1	T		5.00		930	1000	0.1099	
WI17-37	18475A-T-1	T		5.00		890	1000	0.1058	

NOTES

*1: C=Control, F=Fortified, T=Treated

*2: Spike amount = (Spike added) ÷ (Sample weight)

*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)

*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100

"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WI17
Analysis for:	TFNA-AM	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 06-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WI17-58	A307G-27	0.0001012	155
WI17-59	A307G-26	0.0002024	288
WI17-60	A307G-25	0.0005060	694
WI17-61	A307G-24	0.001012	1444
WI17-62	A307G-23	0.0020240	2625
WI17-63	A307G-22	0.005060	7730
WI17-74	A307G-22	0.005060	7202
WI17-75	A307G-23	0.0020240	2539
WI17-76A	A307G-24	0.001012	1380
WI17-77	A307G-25	0.0005060	800
WI17-78	A307G-26	0.0002024	329
WI17-79A	A307G-27	0.0001012	162

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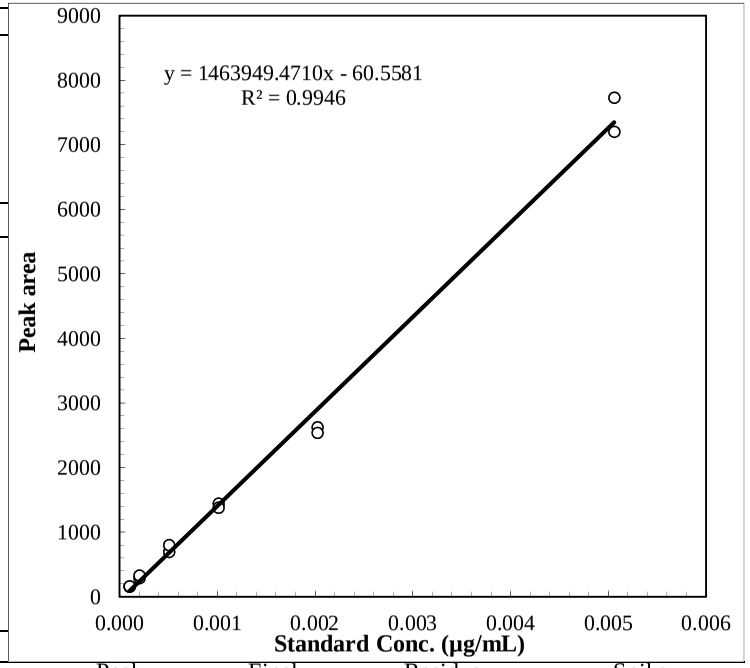
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1463949
Intercept, b = -60.56
R^2 = 0.9946
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 06-Aug-12 Analysis date: 06-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WI17-65	18476A-C-1	C	NA	2.50	NA	0	100	< 0.02	NA
WI17-66	18476A-C-1	C	NA	2.50	NA	0	100	< 0.02	NA
WI17-67	18476A-QC-0.02-1	F	0.05060	2.50	0.0202	531	100	0.01616	79.86
WI17-68	18476A-QC-0.02-1	F	0.05060	2.50	0.0202	555	100	0.01682	83.10
WI17-69	18476A-QC-0.2-1	F	0.5060	2.50	0.2024	1081	500	0.1560	77.05
WI17-70	18476A-QC-0.2-1	F	0.5060	2.50	0.2024	1180	500	0.1695	83.74
WI17-71	18477A-T-1	T		2.50		0	100	< 0.02	
WI17-72	18477A-T-1	T		2.50		0	100	< 0.02	

NOTES *1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WI17
Analysis for:	Flonicamid (IKI-220)	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 06-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WI17-58	A307G-27	0.0001010	194
WI17-59	A307G-26	0.0002020	411
WI17-60	A307G-25	0.0005050	1139
WI17-61	A307G-24	0.001010	2297
WI17-62	A307G-23	0.0020200	4453
WI17-63	A307G-22	0.005050	11530
WI17-74	A307G-22	0.005050	11140
WI17-75	A307G-23	0.0020200	4464
WI17-76	A307G-24	0.001010	2202
WI17-77	A307G-25	0.0005050	1095
WI17-78	A307G-26	0.0002020	466
WI17-79	A307G-27	0.0001010	247

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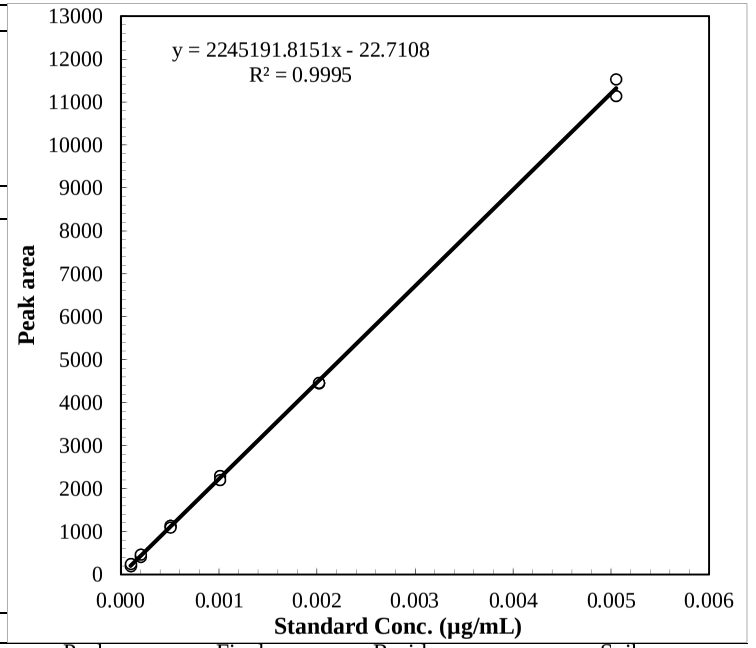
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2245192
Intercept, b = -22.71
R^2 = 0.9995
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 06-Aug-12 Analysis date: 06-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WI17-65	18476A-C-1	C	NA	2.50	NA	8	100	0.000547	NA
WI17-66	18476A-C-1	C	NA	2.50	NA	10	100	0.000583	NA
WI17-67	18476A-QC-0.02-1	F	0.05050	2.50	0.0202	966	100	0.0176	87.20
WI17-68	18476A-QC-0.02-1	F	0.05050	2.50	0.0202	1003	100	0.0183	90.46
WI17-69	18476A-QC-0.2-1	F	0.5050	2.50	0.2020	2016	500	0.1816	89.90
WI17-70	18476A-QC-0.2-1	F	0.5050	2.50	0.2020	1995	500	0.1797	88.98
WI17-71	18477A-T-1	T		2.50		27	100	0.000886	
WI17-72	18477A-T-1	T		2.50		16	100	0.000690	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

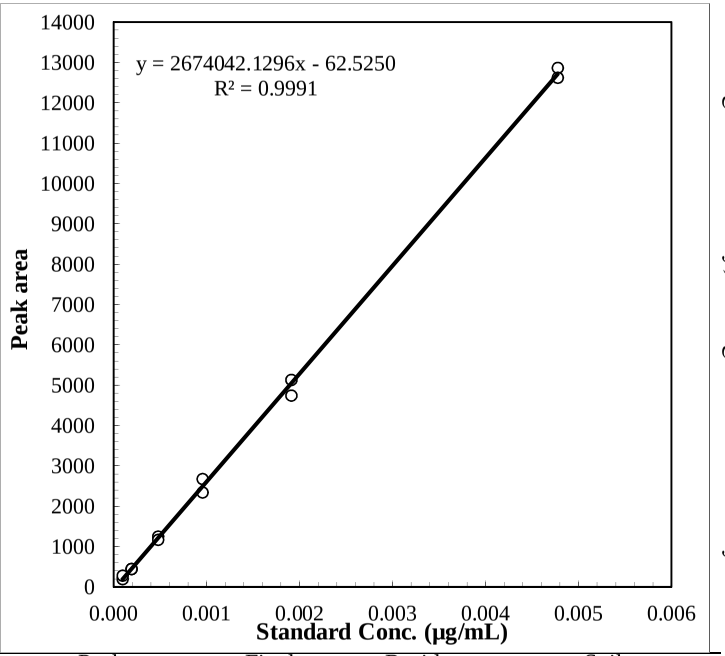
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WI17
Analysis for:	TFNG	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 06-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WI17-58	A307G-27	0.00009550	198
WI17-59	A307G-26	0.0001910	451
WI17-60	A307G-25	0.0004775	1253
WI17-61	A307G-24	0.00095500	2679
WI17-62	A307G-23	0.0019100	4746
WI17-63	A307G-22	0.004775	12865
WI17-74	A307G-22	0.004775	12621
WI17-75	A307G-23	0.0019100	5134
WI17-76	A307G-24	0.00095500	2345
WI17-77A	A307G-25	0.0004775	1172
WI17-78	A307G-26	0.0001910	448
WI17-79	A307G-27	0.00009550	283

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2674042
Intercept, b = -62.53
R^2 = 0.9991
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 06-Aug-12 Analysis date: 06-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WI17-65	18476A-C-1	C	NA	2.50	NA	11	100	0.001100	NA
WI17-66	18476A-C-1	C	NA	2.50	NA	13	100	0.001130	NA
WI17-67	18476A-QC-0.02-1	F	0.04775	2.50	0.0191	946	100	0.0151	78.99
WI17-68	18476A-QC-0.02-1	F	0.04775	2.50	0.0191	941	100	0.0150	78.59
WI17-69	18476A-QC-0.2-1	F	0.4775	2.50	0.1910	2216	500	0.1704	89.22
WI17-70	18476A-QC-0.2-1	F	0.4775	2.50	0.1910	2215	500	0.1703	89.18
WI17-71	18477A-T-1	T		2.50		70	100	0.001982	
WI17-72	18477A-T-1	T		2.50		76	100	0.002072	

NOTES

*1: C=Control, F=Fortified, T=Treated

*2: Spike amount = (Spike added) ÷ (Sample weight)

*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)

*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100

"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

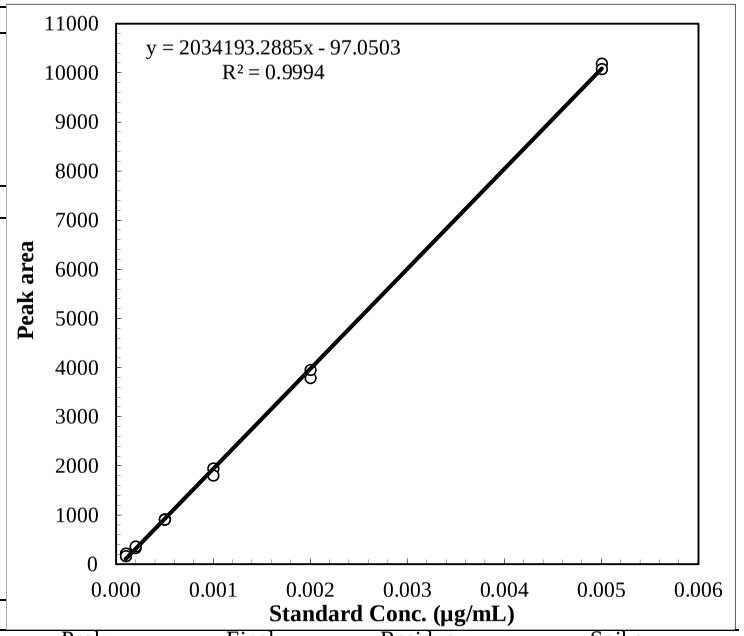
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WI17
Analysis for:	TFNA	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 06-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WI17-58	A307G-27	0.0001001	218
WI17-59	A307G-26	0.0002002	331
WI17-60	A307G-25	0.0005005	915
WI17-61	A307G-24	0.001001	1950
WI17-62	A307G-23	0.0020020	3791
WI17-63	A307G-22	0.005005	10191
WI17-74	A307G-22	0.005005	10079
WI17-75	A307G-23	0.0020020	3955
WI17-76	A307G-24	0.001001	1806
WI17-77	A307G-25	0.0005005	910
WI17-78	A307G-26	0.0002002	361
WI17-79	A307G-27	0.0001001	166

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2034193
Intercept, b = -97.05
R^2 = 0.9994
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 06-Aug-12 Analysis date: 06-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WI17-65	18476A-C-1	C	NA	2.50	NA	0	100	< 0.02	NA
WI17-66	18476A-C-1	C	NA	2.50	NA	0	100	< 0.02	NA
WI17-67	18476A-QC-0.02-1	F	0.05010	2.50	0.02004	798	100	0.01760	87.82
WI17-68	18476A-QC-0.02-1	F	0.05010	2.50	0.02004	763	100	0.01691	84.39
WI17-69	18476A-QC-0.2-1	F	0.5010	2.50	0.2004	1801	500	0.1866	93.12
WI17-70	18476A-QC-0.2-1	F	0.5010	2.50	0.2004	1669	500	0.1736	86.64
WI17-71	18477A-T-1	T		2.50		24	100	0.002380	
WI17-72	18477A-T-1	T		2.50		0	100	< 0.02	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

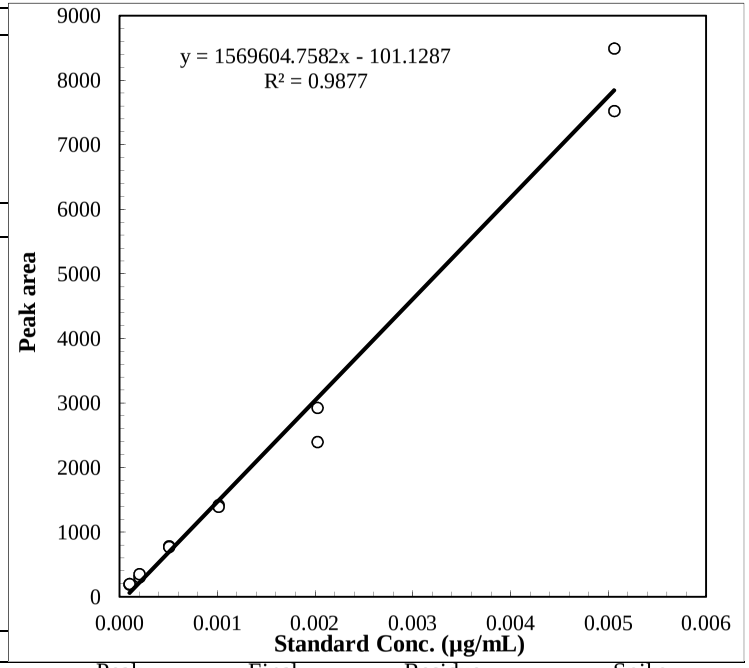
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WI18
Analysis for:	TFNA-AM	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 03-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WI18-21	A307G-27	0.0001012	189
WI18-22	A307G-26	0.0002024	302
WI18-23	A307G-25	0.0005060	782
WI18-24	A307G-24	0.001012	1419
WI18-25	A307G-23	0.0020240	2397
WI18-26	A307G-22	0.005060	8492
WI18-38	A307G-22	0.005060	7523
WI18-39	A307G-23	0.0020240	2926
WI18-40	A307G-24	0.001012	1394
WI18-41	A307G-25	0.0005060	771
WI18-42	A307G-26	0.0002024	350
WI18-43	A307G-27	0.0001012	198

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1569605
Intercept, b = -101.13
R^2 = 0.9877
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 03-Aug-12 Analysis date: 03-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WI18-28	18479A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
WI18-29	18479A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
WI18-30	18479A-QC-3-1	F	15.18	5.00	3.036	5640	3333	2.438	80.31
WI18-31	18479A-QC-3-1	F	15.18	5.00	3.036	5665	3333	2.449	80.66
WI18-32	18480A-T-1	T		5.00		537	2000	0.1626	
WI18-33	18480A-T-1	T		5.00		570	2000	0.1710	
WI18-34	18481A-T-1	T		5.00		554	2000	0.1670	
WI18-35	18481A-T-1	T		5.00		528	2000	0.1603	

NOTES *1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WI18
Analysis for:	Flonicamid (IKI-220)	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 03-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WI18-21	A307G-27	0.0001010	199
WI18-22	A307G-26	0.0002020	452
WI18-23	A307G-25	0.0005050	1093
WI18-24	A307G-24	0.001010	2238
WI18-25	A307G-23	0.0020200	4434
WI18-26	A307G-22	0.005050	11575
WI18-38	A307G-22	0.005050	11137
WI18-39	A307G-23	0.0020200	4539
WI18-40	A307G-24	0.001010	2179
WI18-41	A307G-25	0.0005050	1168
WI18-42	A307G-26	0.0002020	455
WI18-43	A307G-27	0.0001010	221

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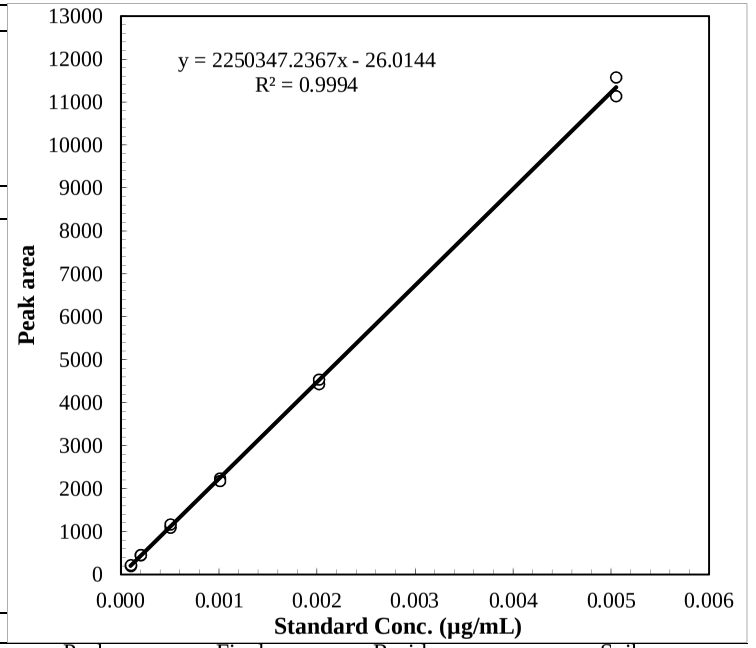
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2250347
Intercept, b = -26.01
R^2 = 0.9994
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 03-Aug-12 Analysis date: 03-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WI18-28	18479A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
WI18-29	18479A-C-1	C	NA	5.00	NA	66	200	0.00164	NA
WI18-30	18479A-QC-3-1	F	15.150	5.00	3.0300	8075	3333	2.400	79.20
WI18-31	18479A-QC-3-1	F	15.150	5.00	3.0300	8222	3333	2.443	80.63
WI18-32	18480A-T-1	T		5.00		10752	2000	1.916	
WI18-33	18480A-T-1	T		5.00		10570	2000	1.883	
WI18-34	18481A-T-1	T		5.00		10918	2000	1.945	
WI18-35	18481A-T-1	T		5.00		10762	2000	1.918	

NOTES *1: C=Control, F=Fortified, T=Treated

*2: Spike amount = (Spike added) ÷ (Sample weight)

*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)

*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100

"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

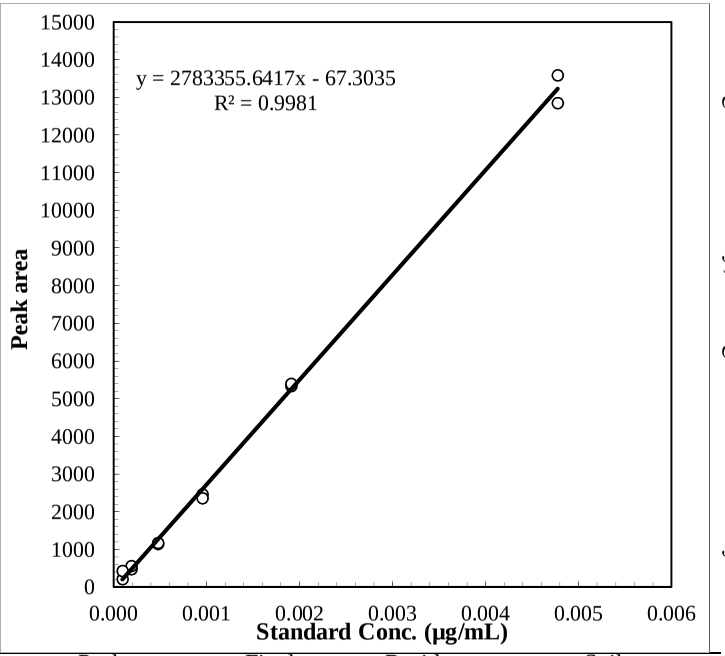
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WI18
Analysis for:	TFNG	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 03-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WI18-21	A307G-27	0.00009550	210
WI18-22	A307G-26	0.0001910	473
WI18-23	A307G-25	0.0004775	1143
WI18-24	A307G-24	0.00095500	2463
WI18-25	A307G-23	0.0019100	5338
WI18-26	A307G-22	0.004775	13587
WI18-38	A307G-22	0.004775	12849
WI18-39	A307G-23	0.0019100	5396
WI18-40	A307G-24	0.00095500	2358
WI18-41	A307G-25	0.0004775	1172
WI18-42	A307G-26	0.0001910	559
WI18-43	A307G-27	0.00009550	427

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2783356
Intercept, b = -67.30
R^2 = 0.9981
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 03-Aug-12 Analysis date: 03-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WI18-28	18479A-C-1	C	NA	5.00	NA	342	200	0.00588	NA
WI18-29	18479A-C-1	C	NA	5.00	NA	304	200	0.00534	NA
WI18-30	18479A-QC-3-1	F	14.3250	5.00	2.8650	10777	3333	2.597	90.65
WI18-31	18479A-QC-3-1	F	14.3250	5.00	2.8650	11365	3333	2.738	95.57
WI18-32	18480A-T-1	T		5.00		2505	2000	0.3697	
WI18-33	18480A-T-1	T		5.00		2461	2000	0.3633	
WI18-34	18481A-T-1	T		5.00		2451	2000	0.3619	
WI18-35	18481A-T-1	T		5.00		2369	2000	0.3501	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

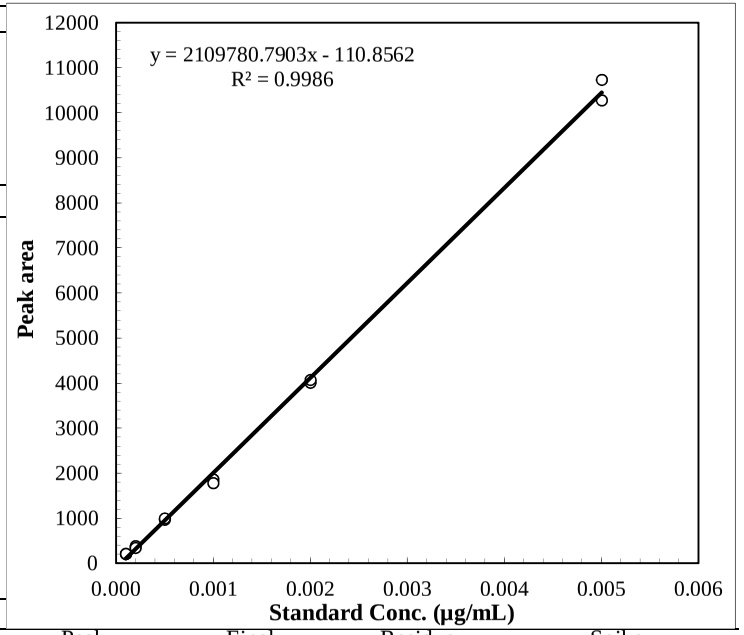
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WI18
Analysis for:	TFNA	Field Research Director:	Dr. Scott Chapman
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 03-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WI18-21	A307G-27	0.0001001	208
WI18-22	A307G-26	0.0002002	382
WI18-23	A307G-25	0.0005005	968
WI18-24	A307G-24	0.001001	1863
WI18-25	A307G-23	0.0020020	4008
WI18-26	A307G-22	0.005005	10734
WI18-38	A307G-22	0.005005	10278
WI18-39	A307G-23	0.0020020	4071
WI18-40	A307G-24	0.001001	1780
WI18-41	A307G-25	0.0005005	996
WI18-42	A307G-26	0.0002002	342
WI18-43	A307G-27	0.0001001	209

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2109781
Intercept, b = -110.86
R^2 = 0.9986
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 03-Aug-12 Analysis date: 03-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WI18-28	18479A-C-1	C	NA	5.00	NA	115	200	0.00428	NA
WI18-29	18479A-C-1	C	NA	5.00	NA	110	200	0.00419	NA
WI18-30	18479A-QC-3-1	F	15.015	5.00	3.003	9102	3333	2.9109	96.93
WI18-31	18479A-QC-3-1	F	15.015	5.00	3.003	9246	3333	2.9564	98.45
WI18-32	18480A-T-1	T		5.00		1026	2000	0.21554	
WI18-33	18480A-T-1	T		5.00		981	2000	0.20701	
WI18-34	18481A-T-1	T		5.00		1002	2000	0.21099	
WI18-35	18481A-T-1	T		5.00		929	2000	0.19715	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

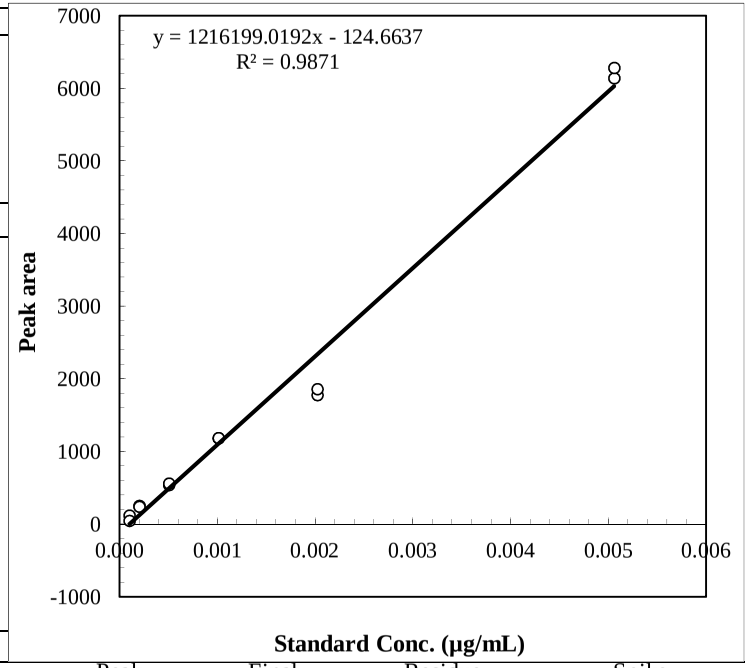
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WA*18
Analysis for:	TFNA-AM	Field Research Director:	John Harvey
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 30-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WA*18-23	A307G-27	0.0001012	118
WA*18-24A	A307G-26	0.0002024	252
WA*18-25	A307G-25	0.0005060	537
WA*18-26	A307G-24	0.001012	1180
WA*18-27	A307G-23	0.0020240	1775
WA*18-28	A307G-22	0.005060	6139
WA*18-39	A307G-22	0.005060	6281
WA*18-40	A307G-23	0.0020240	1858
WA*18-41A	A307G-24	0.001012	1184
WA*18-42	A307G-25	0.0005060	558
WA*18-43	A307G-26	0.0002024	239
WA*18-44	A307G-27	0.0001012	45

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1216199
Intercept, b = -124.66
R^2 = 0.9871
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 26-Jul-12 Analysis date: 30-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WA*18-30	18467A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
WA*18-31	18467A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
WA*18-32	18467A-QC-2-1	F	10.12	5.00	2.024	1946	5000	1.703	84.12
WA*18-33	18467A-QC-2-1	F	10.12	5.00	2.024	1959	5000	1.713	84.65
WA*18-34	18468A-T-1	T		5.00		411	2000	0.1762	
WA*18-35	18468A-T-1	T		5.00		371	2000	0.1630	
WA*18-36	18469A-T-1	T		5.00		351	2000	0.1564	
WA*18-37	18469A-T-1	T		5.00		477	2000	0.1979	

NOTES *1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WA*18
Analysis for:	Flonicamid (IKI-220)	Field Research Director:	John Harvey
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 30-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WA*18-23	A307G-27	0.0001010	115
WA*18-24	A307G-26	0.0002020	332
WA*18-25	A307G-25	0.0005050	648
WA*18-26	A307G-24	0.001010	1620
WA*18-27	A307G-23	0.0020200	3018
WA*18-28	A307G-22	0.005050	8821
WA*18-39	A307G-22	0.005050	9154
WA*18-40	A307G-23	0.0020200	3224
WA*18-41	A307G-24	0.001010	1884
WA*18-42	A307G-25	0.0005050	788
WA*18-43	A307G-26	0.0002020	360
WA*18-44	A307G-27	0.0001010	154

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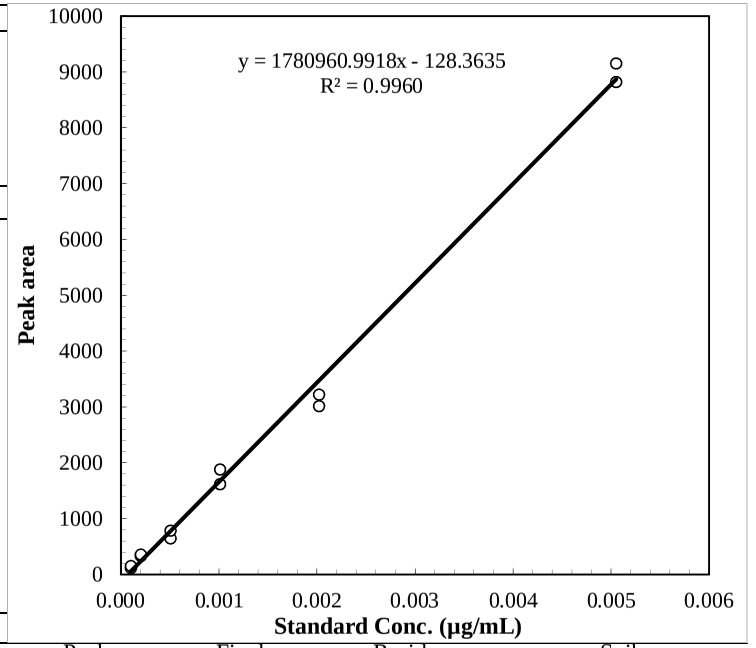
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1780961
Intercept, b = -128.36
R^2 = 0.9960
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 26-Jul-12 Analysis date: 30-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WA*18-30	18467A-C-1	C	NA	5.00	NA	10	200	0.00311	NA
WA*18-31	18467A-C-1	C	NA	5.00	NA	8	200	0.00306	NA
WA*18-32	18467A-QC-2-1	F	10.10	5.00	2.020	2939	5000	1.722	85.26
WA*18-33	18467A-QC-2-1	F	10.10	5.00	2.020	3026	5000	1.771	87.68
WA*18-34	18468A-T-1	T		5.00		6413	2000	1.469	
WA*18-35	18468A-T-1	T		5.00		7119	2000	1.628	
WA*18-36	18469A-T-1	T		5.00		6633	2000	1.519	
WA*18-37	18469A-T-1	T		5.00		7250	2000	1.657	

NOTES *1: C=Control, F=Fortified, T=Treated

*2: Spike amount = (Spike added) ÷ (Sample weight)

*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)

*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100

"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WA*18
Analysis for:	TFNG	Field Research Director:	John Harvey
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 30-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WA*18-23	A307G-27	0.00009550	216
WA*18-24A	A307G-26	0.0001910	331
WA*18-25	A307G-25	0.0004775	812
WA*18-26	A307G-24	0.00095500	1961
WA*18-27	A307G-23	0.0019100	3549
WA*18-28	A307G-22	0.004775	11830
WA*18-39	A307G-22	0.004775	11453
WA*18-40	A307G-23	0.0019100	3641
WA*18-41	A307G-24	0.00095500	2108
WA*18-42	A307G-25	0.0004775	857
WA*18-43	A307G-26	0.0001910	375
WA*18-44	A307G-27	0.00009550	179

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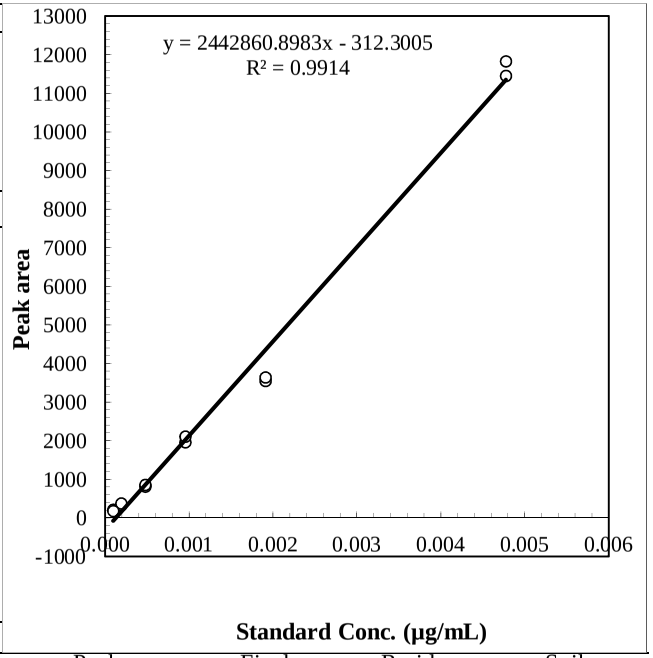
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2442861
Intercept, b = -312.30
R^2 = 0.9914
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 26-Jul-12 Analysis date: 30-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WA*18-30	18467A-C-1	C	NA	5.00	NA	326	200	0.0105	NA
WA*18-31	18467A-C-1	C	NA	5.00	NA	269	200	0.0095	NA
WA*18-32	18467A-QC-2-1	F	9.550	5.00	1.910	3964	5000	1.751	91.65
WA*18-33	18467A-QC-2-1	F	9.550	5.00	1.910	3995	5000	1.763	92.32
WA*18-34	18468A-T-1	T		5.00		1671	2000	0.3248	
WA*18-35	18468A-T-1	T		5.00		1725	2000	0.3336	
WA*18-36	18469A-T-1	T		5.00		1618	2000	0.3161	
WA*18-37	18469A-T-1	T		5.00		2022	2000	0.3822	

NOTES

*1: C=Control, F=Fortified, T=Treated

*2: Spike amount = (Spike added) ÷ (Sample weight)

*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)

*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100

"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

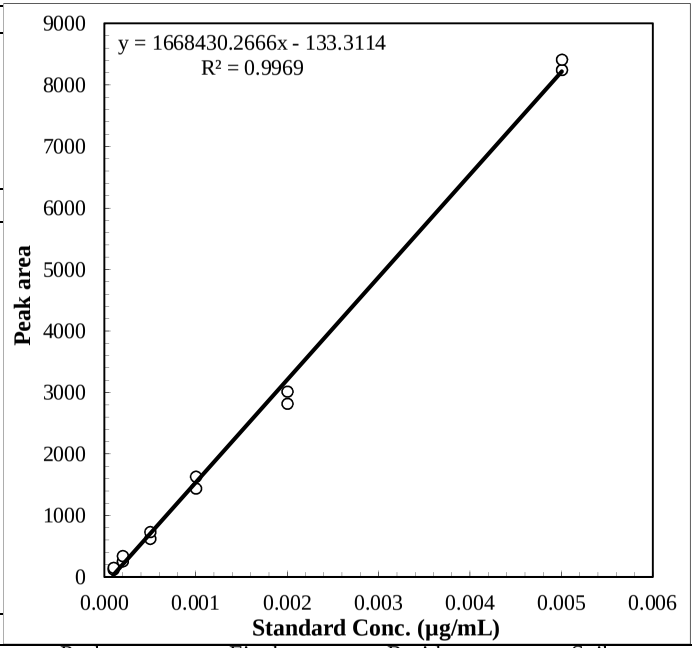
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WA*18
Analysis for:	TFNA	Field Research Director:	John Harvey
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

Date of calibration analysis: 30-Jul-12

	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WA*18-23	A307G-27	0.0001001	123
WA*18-24	A307G-26	0.0002002	256
WA*18-25	A307G-25	0.0005005	622
WA*18-26	A307G-24	0.001001	1439
WA*18-27	A307G-23	0.0020020	2819
WA*18-28	A307G-22	0.005005	8246
WA*18-39	A307G-22	0.005005	8413
WA*18-40	A307G-23	0.0020020	3016
WA*18-41	A307G-24	0.001001	1633
WA*18-42	A307G-25	0.0005005	733
WA*18-43	A307G-26	0.0002002	343
WA*18-44	A307G-27	0.0001001	151

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1668430
Intercept, b = -133.31
R^2 = 0.9969
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 26-Jul-12 Analysis date: 30-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WA*18-30	18467A-C-1	C	NA	5.00	NA	31	200	0.00394	NA
WA*18-31	18467A-C-1	C	NA	5.00	NA	62	200	0.00468	NA
WA*18-32	18467A-QC-2-1	F	10.01	5.00	2.002	3202	5000	1.999	99.85
WA*18-33	18467A-QC-2-1	F	10.01	5.00	2.002	3186	5000	1.989	99.37
WA*18-34	18468A-T-1	T		5.00		680	2000	0.1950	
WA*18-35	18468A-T-1	T		5.00		661	2000	0.1904	
WA*18-36	18469A-T-1	T		5.00		569	2000	0.1684	
WA*18-37	18469A-T-1	T		5.00		776	2000	0.2180	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

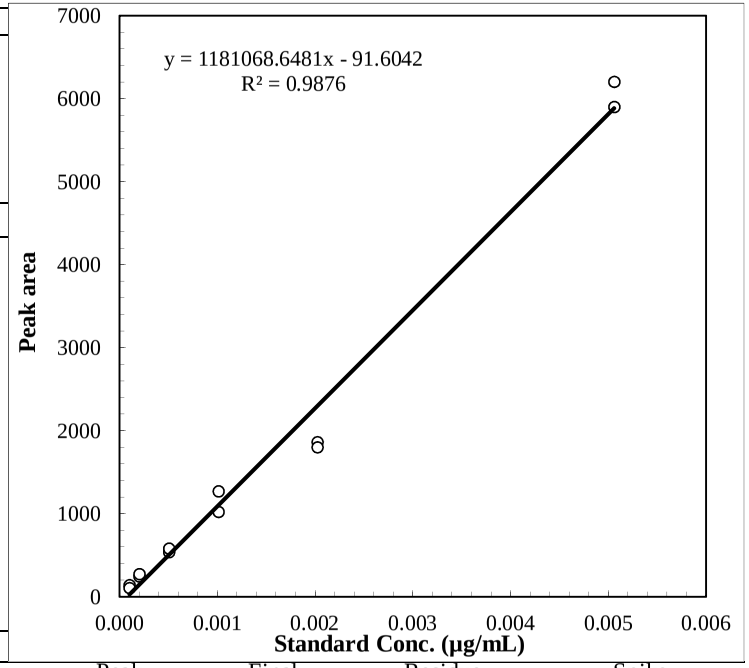
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WA*18
Analysis for:	TFNA-AM	Field Research Director:	John Harvey
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 30-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WA*18-56	A307G-27	0.0001012	140
WA*18-57	A307G-26	0.0002024	244
WA*18-58A	A307G-25	0.0005060	537
WA*18-59A	A307G-24	0.001012	1022
WA*18-60	A307G-23	0.0020240	1863
WA*18-61	A307G-22	0.005060	5900
WA*18-72	A307G-22	0.005060	6203
WA*18-73	A307G-23	0.0020240	1800
WA*18-74	A307G-24	0.001012	1270
WA*18-75	A307G-25	0.0005060	580
WA*18-76	A307G-26	0.0002024	273
WA*18-77	A307G-27	0.0001012	105

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1181069
Intercept, b = -91.60
R^2 = 0.9876
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 30-Jul-12 Analysis date: 30-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WA*18-63	18470A-C-2	C	NA	2.50	NA	0	100	< 0.02	NA
WA*18-64	18470A-C-2	C	NA	2.50	NA	0	100	< 0.02	NA
WA*18-65	18470A-QC-0.02-1	F	0.05060	2.50	0.02024	530	100	0.02105	104.01
WA*18-66	18470A-QC-0.02-1	F	0.05060	2.50	0.02024	525	100	0.02088	103.18
WA*18-67	18470A-QC-0.02-2	F	0.05060	2.50	0.02024	483	100	0.01946	96.15
WA*18-68	18470A-QC-0.02-2	F	0.05060	2.50	0.02024	524	100	0.02085	103.01
WA*18-69	18471A-T-1	T		2.50		0	100	< 0.02	
WA*18-70	18471A-T-1	T		2.50		0	100	< 0.02	

NOTES *1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

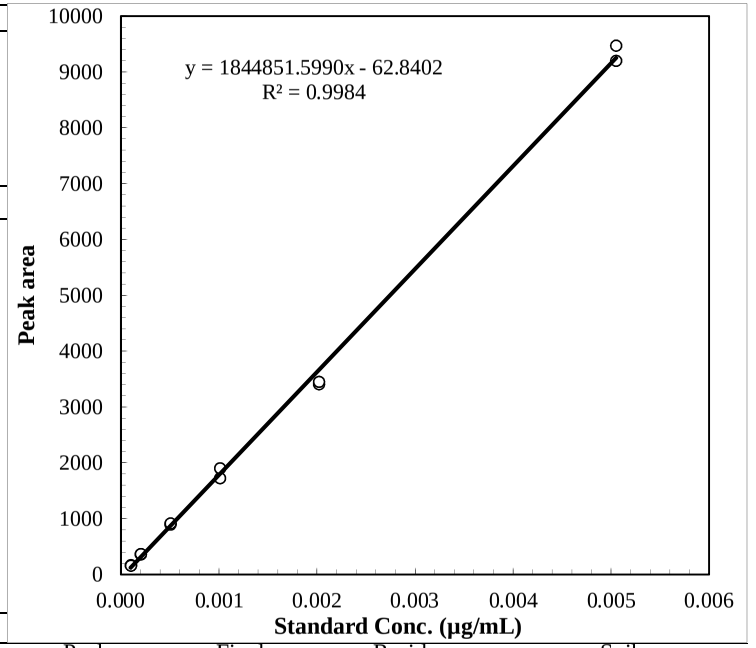
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WA*18
Analysis for:	Flonicamid (IKI-220)	Field Research Director:	John Harvey
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 30-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WA*18-56	A307G-27	0.0001010	170
WA*18-57	A307G-26	0.0002020	361
WA*18-58	A307G-25	0.0005050	897
WA*18-59	A307G-24	0.001010	1726
WA*18-60	A307G-23	0.0020200	3409
WA*18-61	A307G-22	0.005050	9202
WA*18-72	A307G-22	0.005050	9472
WA*18-73	A307G-23	0.0020200	3453
WA*18-74	A307G-24	0.001010	1904
WA*18-75	A307G-25	0.0005050	917
WA*18-76	A307G-26	0.0002020	369
WA*18-77	A307G-27	0.0001010	160

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1844852
Intercept, b = -62.84
R^2 = 0.9984
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 30-Jul-12 Analysis date: 30-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WA*18-63	18470A-C-2	C	NA	2.50	NA	71	100	0.00290	NA
WA*18-64	18470A-C-2	C	NA	2.50	NA	0	100	< 0.02	NA
WA*18-65	18470A-QC-0.02-1	F	0.05050	2.50	0.02020	867	100	0.02016	99.81
WA*18-66	18470A-QC-0.02-1	F	0.05050	2.50	0.02020	838	100	0.01953	96.69
WA*18-67	18470A-QC-0.02-2	F	0.05050	2.50	0.02020	775	100	0.01817	89.93
WA*18-68	18470A-QC-0.02-2	F	0.05050	2.50	0.02020	933	100	0.02159	106.89
WA*18-69	18471A-T-1	T		2.50		133	100	0.004246	
WA*18-70	18471A-T-1	T		2.50		48	100	0.002403	

NOTES *1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

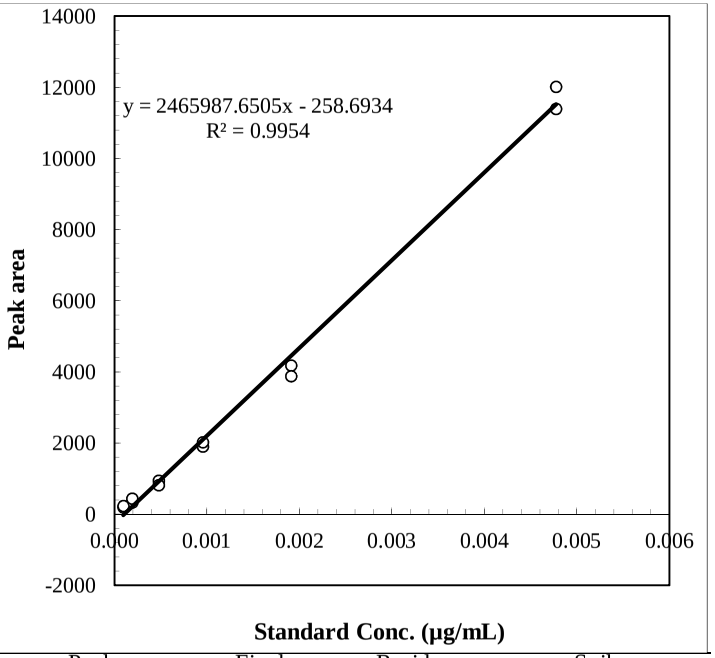
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WA*18
Analysis for:	TFNG	Field Research Director:	John Harvey
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 30-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WA*18-56	A307G-27	0.00009550	189
WA*18-57A	A307G-26	0.0001910	331
WA*18-58A	A307G-25	0.0004775	943
WA*18-59	A307G-24	0.00095500	1902
WA*18-60	A307G-23	0.0019100	4180
WA*18-61	A307G-22	0.004775	11394
WA*18-72	A307G-22	0.004775	12014
WA*18-73	A307G-23	0.0019100	3880
WA*18-74	A307G-24	0.00095500	2023
WA*18-75	A307G-25	0.0004775	817
WA*18-76	A307G-26	0.0001910	439
WA*18-77	A307G-27	0.00009550	232

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2465988
Intercept, b = -258.69
R^2 = 0.9954
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 30-Jul-12 Analysis date: 30-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WA*18-63	18470A-C-2	C	NA	2.50	NA	27	100	0.00463	NA
WA*18-64	18470A-C-2	C	NA	2.50	NA	2	100	0.00423	NA
WA*18-65	18470A-QC-0.02-1	F	0.04775	2.50	0.01910	698	100	0.01552	81.25
WA*18-66	18470A-QC-0.02-1	F	0.04775	2.50	0.01910	723	100	0.01592	83.37
WA*18-67	18470A-QC-0.02-2	F	0.04775	2.50	0.01910	860	100	0.01815	95.01
WA*18-68	18470A-QC-0.02-2	F	0.04775	2.50	0.01910	859	100	0.01813	94.92
WA*18-69	18471A-T-1	T		2.50		7	100	0.00431	
WA*18-70	18471A-T-1	T		2.50		2	100	0.00423	

NOTES

*1: C=Control, F=Fortified, T=Treated

*2: Spike amount = (Spike added) ÷ (Sample weight)

*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)

*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100

"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

PR Number: 09358 Laboratory ID: 09358.11-MIR05
Chemical: Flonicamid Field ID No.: 09358.11-WA*18
Analysis for: TFNA Field Research Director: John Harvey
Commodity: Mint Analyst(s): Eina Abouzied&Lester Geissel
Crop part: Mint Oil Instrument: UPLC/MS/MS

Date of calibration analysis: 30-Jul-12

	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WA*18-56	A307G-27	0.0001001	135
WA*18-57	A307G-26	0.0002002	273
WA*18-58	A307G-25	0.0005005	717
WA*18-59	A307G-24	0.001001	1502
WA*18-60	A307G-23	0.0020020	3133
WA*18-61	A307G-22	0.005005	8168
WA*18-72	A307G-22	0.005005	8758
WA*18-73	A307G-23	0.0020020	3076
WA*18-74	A307G-24	0.001001	1526
WA*18-75	A307G-25	0.0005005	704
WA*18-76	A307G-26	0.0002002	306
WA*18-77	A307G-27	0.0001001	177

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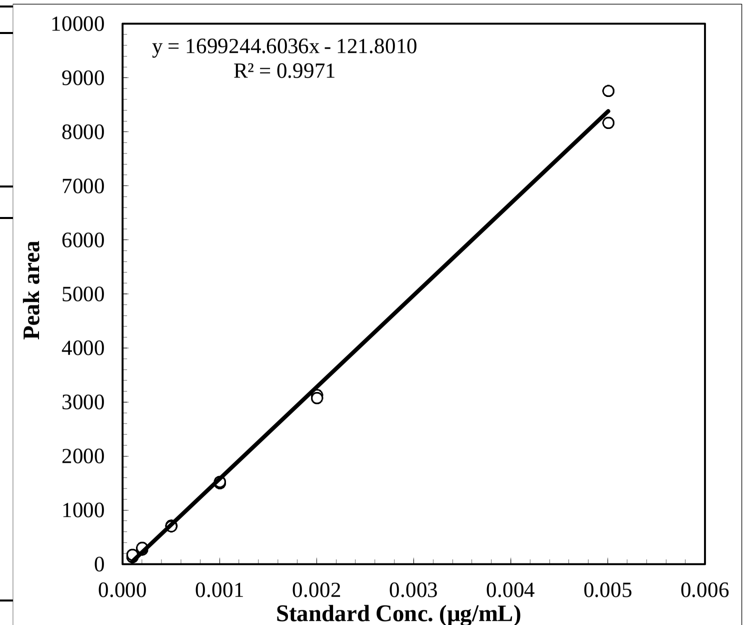
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1699245
Intercept, b = -121.80
R^2 = 0.9971
LOQ (µg/g) = 0.02



ANALYTICAL DATA

Extraction date: 30-Jul-12 Analysis date: 30-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WA*18-63	18470A-C-2	C	NA	2.50	NA	0	100	< 0.02	NA
WA*18-64	18470A-C-2	C	NA	2.50	NA	0	100	< 0.02	NA
WA*18-65	18470A-QC-0.02-1	F	0.05005	2.50	0.02002	588	100	0.01671	83.46
WA*18-66	18470A-QC-0.02-1	F	0.05005	2.50	0.02002	676	100	0.01878	93.81
WA*18-67	18470A-QC-0.02-2	F	0.05005	2.50	0.02002	759	100	0.02073	103.57
WA*18-68	18470A-QC-0.02-2	F	0.05005	2.50	0.02002	736	100	0.0202	100.86
WA*18-69	18471A-T-1	T		2.50		0	100	< 0.02	
WA*18-70	18471A-T-1	T		2.50		0	100	< 0.02	

NOTES

*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

Attachment B: Calculation Worksheets, Standard Curves and LOD/LOQ Calculations

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-ID12
Analysis for:	TFNA-AM	Field Research Director:	Will Meeks
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 24-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
ID12-21	A307G-27	0.0001012	139
ID12-22	A307G-26	0.0002024	234
ID12-23	A307G-25	0.0005060	451
ID12-24	A307G-24	0.001012	948
ID12-25	A307G-23	0.0020240	1874
ID12-26	A307G-22	0.005060	5978
ID12-38	A307G-22	0.005060	6000
ID12-39	A307G-23	0.0020240	1856
ID12-40	A307G-24	0.001012	1126
ID12-41	A307G-25	0.0005060	611
ID12-42	A307G-26	0.0002024	285
ID12-43	A307G-27	0.0001012	87

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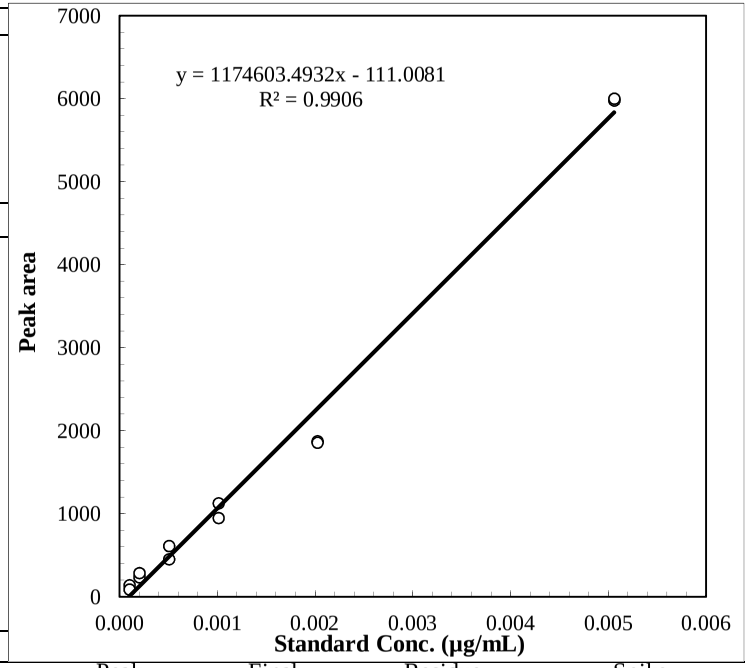
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1174603
Intercept, b = -111.01
R^2 = 0.9906
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 24-Jul-12 Analysis date: 24-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
ID12-28	18353A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
ID12-29	18353A-C-1	C	NA	5.00	NA	15	200	0.00429	NA
ID12-30	18353A-QC-0.02-1	F	0.1012	5.00	0.0202	362	200	0.01611	79.58
ID12-31	18353A-QC-0.02-1	F	0.1012	5.00	0.0202	401	200	0.01744	86.15
ID12-32	18354A-T-1	T		5.00		162	2000	0.0930	
ID12-33	18354A-T-1	T		5.00		119	2000	0.0783	
ID12-34	18355A-T-1	T		5.00		167	2000	0.09467	
ID12-35	18355A-T-1	T		5.00		199	2000	0.1056	

NOTES *1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

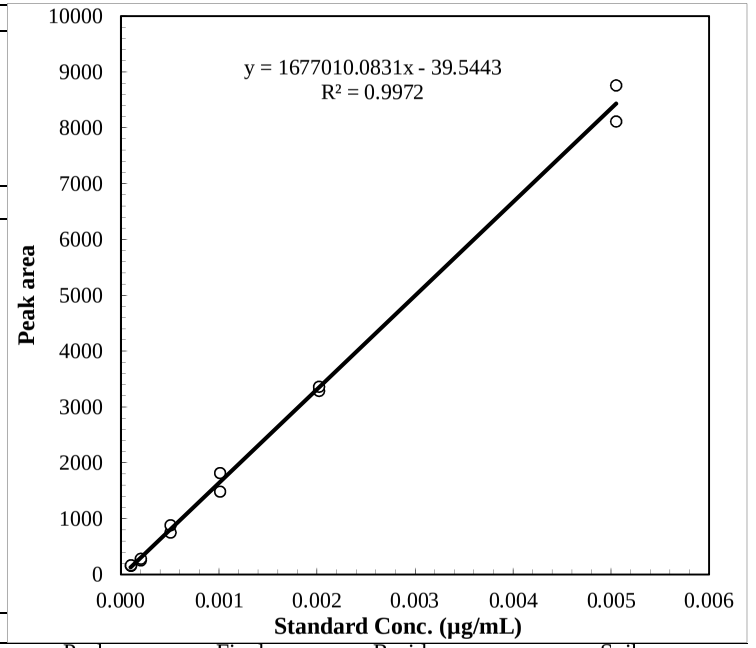
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-ID12
Analysis for:	Flonicamid (IKI-220)	Field Research Director:	Will Meeks
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 24-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
ID12-21	A307G-27	0.0001010	157
ID12-22	A307G-26	0.0002020	256
ID12-23	A307G-25	0.0005050	753
ID12-24	A307G-24	0.001010	1486
ID12-25	A307G-23	0.0020200	3290
ID12-26	A307G-22	0.005050	8114
ID12-38	A307G-22	0.005050	8761
ID12-39	A307G-23	0.0020200	3364
ID12-40	A307G-24	0.001010	1819
ID12-41	A307G-25	0.0005050	884
ID12-42	A307G-26	0.0002020	285
ID12-43	A307G-27	0.0001010	167

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1677010
Intercept, b = -39.54
R^2 = 0.9972
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 24-Jul-12 Analysis date: 24-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
ID12-28	18353A-C-1	C	NA	5.00	NA	36	200	0.00180	NA
ID12-29	18353A-C-1	C	NA	5.00	NA	71	200	0.00264	NA
ID12-30	18353A-QC-0.02-1	F	0.101	5.00	0.0202	547	200	0.01399	69.26
ID12-31	18353A-QC-0.02-1	F	0.101	5.00	0.0202	540	200	0.01382	68.43
ID12-32	18354A-T-1	T		5.00		8638	2000	2.070	
ID12-33	18354A-T-1	T		5.00		~~9389~~	~~2000~~	~~2.248894~~	
ID12-34	18355A-T-1	T		5.00		~~8932~~	~~2000~~	~~2.139890~~	
ID12-35	18355A-T-1	T		5.00		~~9260~~	~~2000~~	~~2.218125~~	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

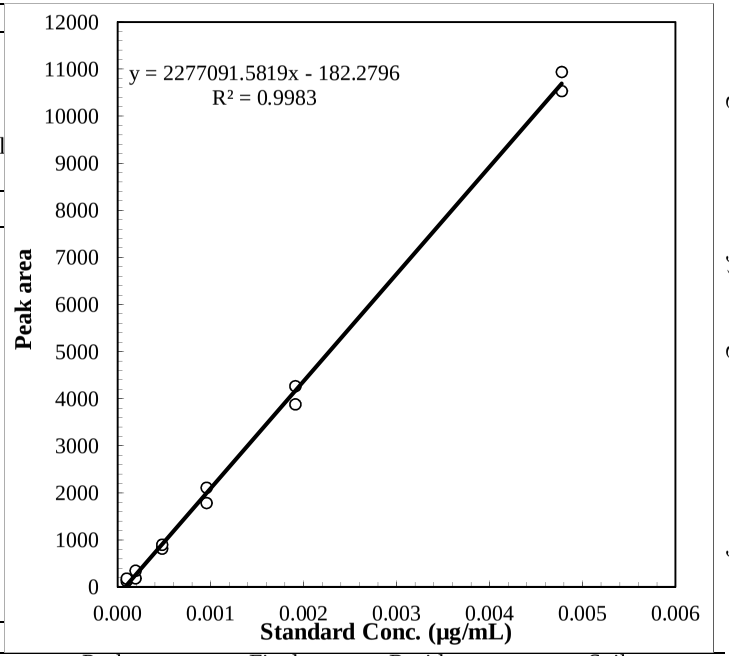
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-ID12
Analysis for:	TFNG	Field Research Director:	Will Meeks
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 24-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
ID12-21	A307G-27	0.00009550	130
ID12-22	A307G-26	0.0001910	187
ID12-23	A307G-25	0.0004775	818
ID12-24	A307G-24	0.00095500	1786
ID12-25	A307G-23	0.0019100	3882
ID12-26	A307G-22	0.004775	10535
ID12-38	A307G-22	0.004775	10941
ID12-39	A307G-23	0.0019100	4268
ID12-40	A307G-24	0.00095500	2111
ID12-41A	A307G-25	0.0004775	900
ID12-42	A307G-26	0.0001910	348
ID12-43A	A307G-27	0.00009550	180

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2277092
Intercept, b = -182.28
R^2 = 0.9983
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 24-Jul-12 Analysis date: 24-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
ID12-28	18353A-C-1	C	NA	5.00	NA	280	200	0.00812	NA
ID12-29	18353A-C-1	C	NA	5.00	NA	306	200	0.00858	NA
ID12-30	18353A-QC-0.02-1	F	0.0955	5.00	0.0191	1054	200	0.0217	113.70
ID12-31	18353A-QC-0.02-1	F	0.0955	5.00	0.0191	1058	200	0.0218	114.07
ID12-32	18354A-T-1	T		5.00		1851	2000	0.3572	
ID12-33	18354A-T-1	T		5.00		1962	2000	0.3767	
ID12-34	18355A-T-1	T		5.00		1823	2000	0.3523	
ID12-35	18355A-T-1	T		5.00		1880	2000	0.3623	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

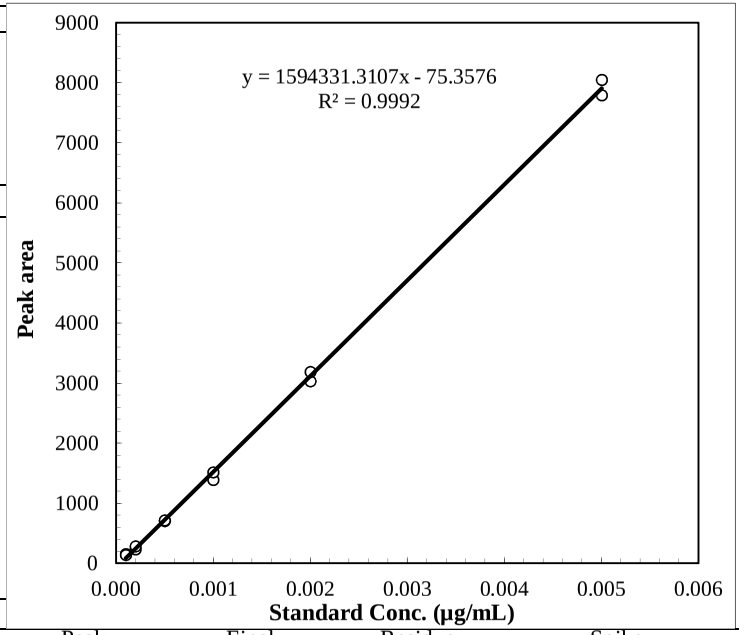
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-ID12
Analysis for:	TFNA	Field Research Director:	Will Meeks
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 24-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
ID12-21	A307G-27	0.0001001	154
ID12-22	A307G-26	0.0002002	231
ID12-23	A307G-25	0.0005005	703
ID12-24	A307G-24	0.001001	1388
ID12-25	A307G-23	0.0020020	3031
ID12-26	A307G-22	0.005005	7792
ID12-38	A307G-22	0.005005	8050
ID12-39	A307G-23	0.0020020	3186
ID12-40	A307G-24	0.001001	1514
ID12-41	A307G-25	0.0005005	715
ID12-42	A307G-26	0.0002002	282
ID12-43	A307G-27	0.0001001	138

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1594331
Intercept, b = -75.36
R^2 = 0.9992
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 24-Jul-12 Analysis date: 24-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
ID12-28	18353A-C-1	C	NA	5.00	NA	84	200	0.00400	NA
ID12-29	18353A-C-1	C	NA	5.00	NA	98	200	0.00435	NA
ID12-30	18353A-QC-0.02-1	F	0.100	5.00	0.02002	847	200	0.02314	115.59
ID12-31	18353A-QC-0.02-1	F	0.100	5.00	0.02002	808	200	0.02216	110.70
ID12-32	18354A-T-1	T		5.00		453	2000	0.1326	
ID12-33	18354A-T-1	T		5.00		524	2000	0.1504	
ID12-34	18355A-T-1	T		5.00		401	2000	0.1195	
ID12-35	18355A-T-1	T		5.00		436	2000	0.1283	

NOTES

*1: C=Control, F=Fortified, T=Treated

*2: Spike amount = (Spike added) ÷ (Sample weight)

*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)

*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100

"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

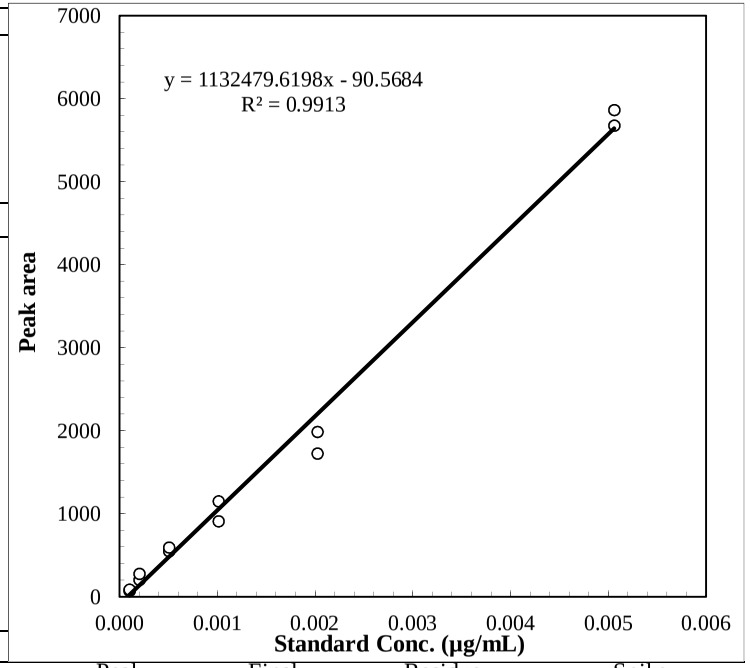
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-ID12
Analysis for:	TFNA-AM	Field Research Director:	Will Meeks
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 25-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
ID12-51	A307G-27	0.0001012	69
ID12-52	A307G-26	0.0002024	203
ID12-53	A307G-25	0.0005060	548
ID12-54	A307G-24	0.001012	909
ID12-55	A307G-23	0.0020240	1725
ID12-56	A307G-22	0.005060	5863
ID12-67	A307G-22	0.005060	5676
ID12-68	A307G-23	0.0020240	1986
ID12-69	A307G-24	0.001012	1150
ID12-70A	A307G-25	0.0005060	593
ID12-71	A307G-26	0.0002024	276
ID12-72	A307G-27	0.0001012	86

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1132480
Intercept, b = -90.57
R^2 = 0.9913
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 24-Jul-12 Analysis date: 25-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
ID12-58	18353A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
ID12-59	18353A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
ID12-60	18353A-QC-0.02-1	F	0.101	5.00	0.0202	341	200	0.01524	75.31
ID12-61	18353A-QC-0.02-1	F	0.101	5.00	0.0202	376	200	0.01648	81.42
ID12-62	18354A-T-1	T		5.00		0	4000	< 0.02	
ID12-63	18354A-T-1	T		5.00		~~54~~	~~4000~~	~~0.1021~~	
ID12-64	18355A-T-1	T		5.00		~~65~~	~~4000~~	~~0.1099~~	
ID12-65	18355A-T-1	T		5.00		121	4000	0.1495	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

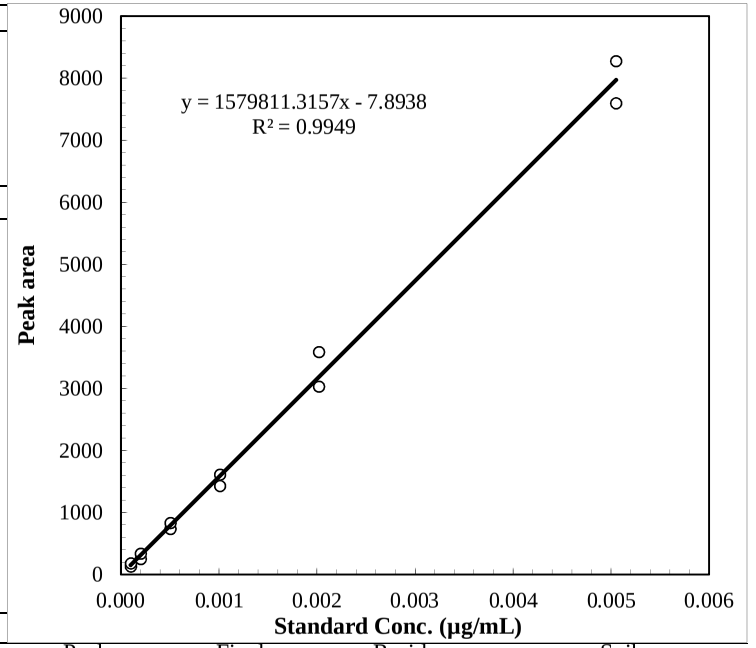
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-ID12
Analysis for:	Flonicamid (IKI-220)	Field Research Director:	Will Meeks
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 25-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
ID12-51	A307G-27	0.0001010	129
ID12-52	A307G-26	0.0002020	251
ID12-53	A307G-25	0.0005050	736
ID12-54	A307G-24	0.001010	1428
ID12-55	A307G-23	0.0020200	3029
ID12-56	A307G-22	0.005050	7596
ID12-67	A307G-22	0.005050	8275
ID12-68	A307G-23	0.0020200	3586
ID12-69	A307G-24	0.001010	1610
ID12-70	A307G-25	0.0005050	831
ID12-71	A307G-26	0.0002020	337
ID12-72	A307G-27	0.0001010	180

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1579811
Intercept, b = -7.89
R^2 = 0.9949
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 24-Jul-12 Analysis date: 25-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
ID12-58	18353A-C-1	C	NA	5.00	NA	40	200	0.001213	NA
ID12-59	18353A-C-1	C	NA	5.00	NA	14	200	0.000554	NA
ID12-60	18353A-QC-0.02-1	F	0.101	5.00	0.0202	612	200	0.0157	77.70
ID12-61	18353A-QC-0.02-1	F	0.101	5.00	0.0202	550	200	0.0141	69.93
ID12-62	18354A-T-1	T		5.00		4426	4000	2.245	
ID12-63	18354A-T-1	T		5.00		4678	4000	2.373	
ID12-64	18355A-T-1	T		5.00		4678	4000	2.373	
ID12-65	18355A-T-1	T		5.00		4829	4000	2.449	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

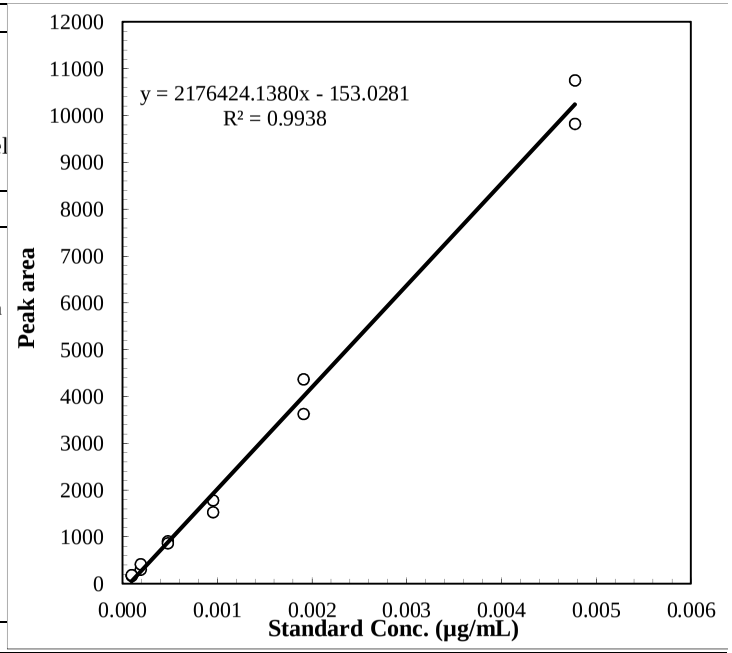
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-ID12
Analysis for:	TFNG	Field Research Director:	Will Meeks
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 25-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
ID12-51	A307G-27	0.00009550	169
ID12-52	A307G-26	0.0001910	304
ID12-53	A307G-25	0.0004775	910
ID12-54	A307G-24	0.00095500	1530
ID12-55	A307G-23	0.0019100	3629
ID12-56	A307G-22	0.004775	9824
ID12-67	A307G-22	0.004775	10753
ID12-68	A307G-23	0.0019100	4369
ID12-69	A307G-24	0.00095500	1783
ID12-70	A307G-25	0.0004775	867
ID12-71	A307G-26	0.0001910	420
ID12-72	A307G-27	0.00009550	187

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2176424
Intercept, b = -153.03
R^2 = 0.9938
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 24-Jul-12 Analysis date: 25-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
ID12-58	18353A-C-1	C	NA	5.00	NA	293	200	0.00820	NA
ID12-59	18353A-C-1	C	NA	5.00	NA	292	200	0.00818	NA
ID12-60	18353A-QC-0.02-1	F	0.0955	5.00	0.0191	862	200	0.0187	97.67
ID12-61	18353A-QC-0.02-1	F	0.0955	5.00	0.0191	972	200	0.0207	108.25
ID12-62	18354A-T-1	T		5.00		885	4000	0.3816	
ID12-63	18354A-T-1	T		5.00		900	4000	0.3871	
ID12-64	18355A-T-1	T		5.00		902	4000	0.3878	
ID12-65	18355A-T-1	T		5.00		946	4000	0.4040	

NOTES

*1: C=Control, F=Fortified, T=Treated

*2: Spike amount = (Spike added) ÷ (Sample weight)

*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)

*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100

"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

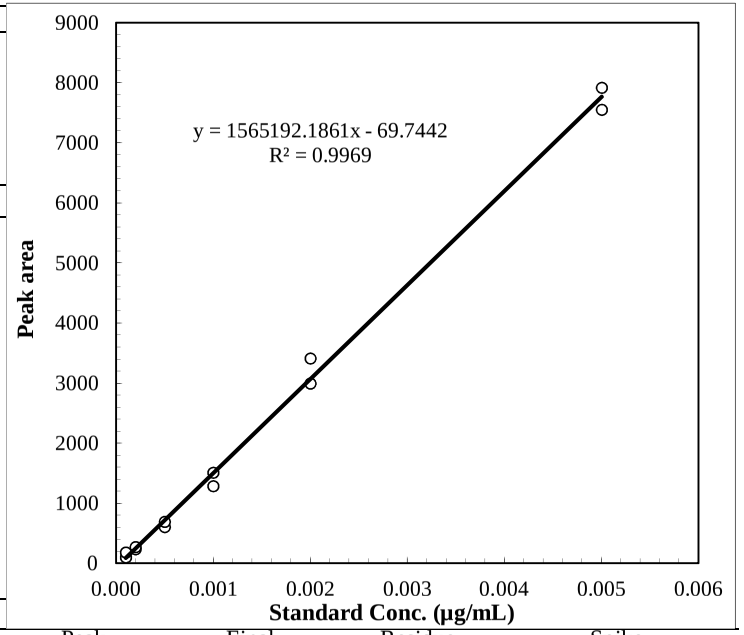
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-ID12
Analysis for:	TFNA	Field Research Director:	Will Meeks
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 25-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
ID12-51	A307G-27	0.0001001	97
ID12-52	A307G-26	0.0002002	234
ID12-53	A307G-25	0.0005005	603
ID12-54	A307G-24	0.001001	1284
ID12-55	A307G-23	0.0020020	2993
ID12-56	A307G-22	0.005005	7551
ID12-67	A307G-22	0.005005	7918
ID12-68	A307G-23	0.0020020	3410
ID12-69	A307G-24	0.001001	1508
ID12-70	A307G-25	0.0005005	690
ID12-71	A307G-26	0.0002002	270
ID12-72	A307G-27	0.0001001	180

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1565192
Intercept, b = -69.74
R^2 = 0.9969
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 24-Jul-12 Analysis date: 25-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
ID12-58	18353A-C-1	C	NA	5.00	NA	79	200	0.00380	NA
ID12-59	18353A-C-1	C	NA	5.00	NA	124	200	0.00495	NA
ID12-60	18353A-QC-0.02-1	F	0.100	5.00	0.02002	753	200	0.0210	105.03
ID12-61	18353A-QC-0.02-1	F	0.100	5.00	0.02002	820	200	0.0227	113.58
ID12-62	18354A-T-1	T		5.00		189	4000	0.13225	
ID12-63	18354A-T-1	T		5.00		259	4000	0.1680	
ID12-64	18355A-T-1	T		5.00		216	4000	0.1460	
ID12-65	18355A-T-1	T		5.00		202	4000	0.1389	

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

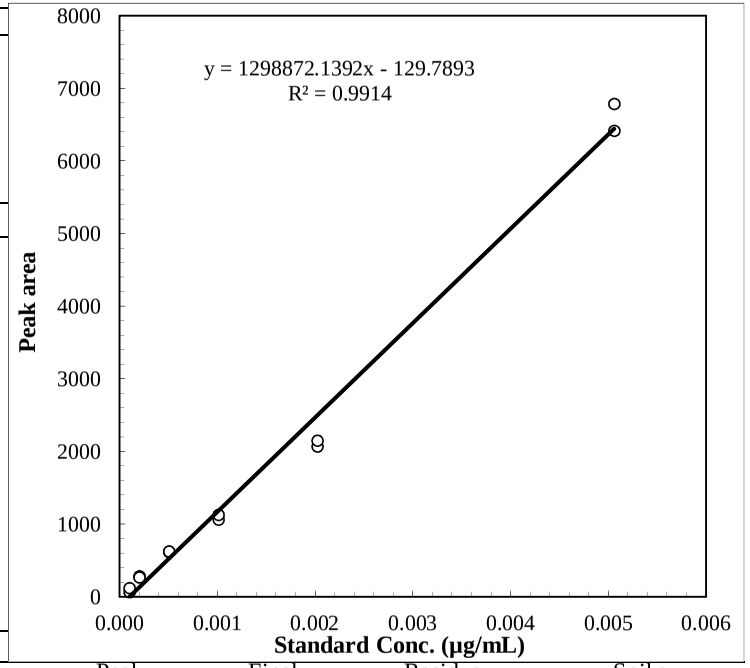
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WA17
Analysis for:	TFNA-AM	Field Research Director:	Dan Groenendale
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 25-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WA17-21	A307G-27	0.0001012	64
WA17-22	A307G-26	0.0002024	282
WA17-23	A307G-25	0.0005060	617
WA17-24	A307G-24	0.001012	1061
WA17-25	A307G-23	0.0020240	2069
WA17-26	A307G-22	0.005060	6415
WA17-37	A307G-22	0.005060	6785
WA17-38	A307G-23	0.0020240	2149
WA17-39	A307G-24	0.001012	1127
WA17-40	A307G-25	0.0005060	623
WA17-41	A307G-26	0.0002024	266
WA17-42	A307G-27	0.0001012	119

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1298872
Intercept, b = -129.79
R^2 = 0.9914
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 25-Jul-12 Analysis date: 25-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WA17-28	18463A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
WA17-29	18463A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
WA17-30	18463A-QC-0.02-1	F	0.1012	5.00	0.0202	387	200	0.01592	78.63
WA17-31	18463A-QC-0.02-1	F	0.1012	5.00	0.0202	403	200	0.01641	81.07
WA17-32	18464A-T-1	T		5.00		188	4000	0.195732	
WA17-33	18464A-T-1	T		5.00		248	4000	0.23269	
WA17-34	18465A-T-1	T		5.00		278	4000	0.25117	
WA17-35	18465A-T-1	T		5.00		287	4000	0.25671	

NOTES *1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

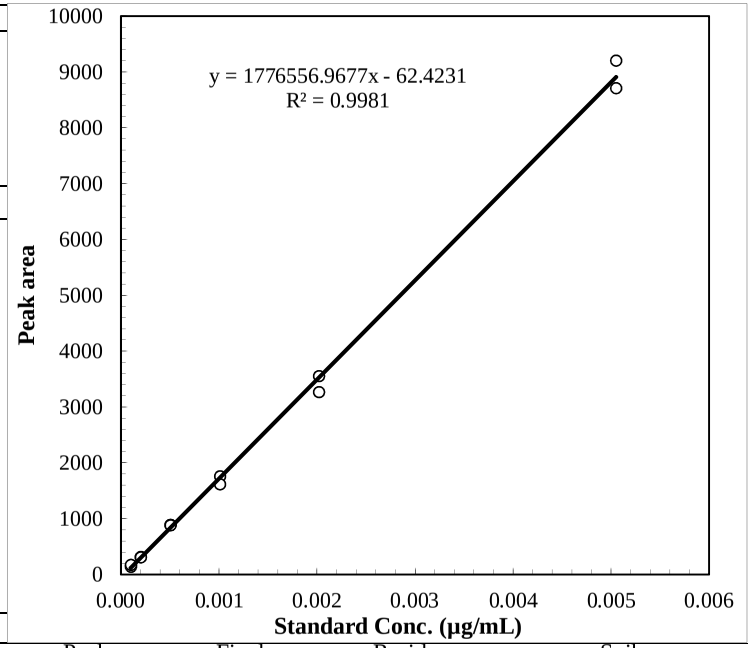
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WA17
Analysis for:	Flonicamid (IKI-220)	Field Research Director:	Dan Groenendale
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 25-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WA17-21	A307G-27	0.0001010	141
WA17-22	A307G-26	0.0002020	317
WA17-23	A307G-25	0.0005050	892
WA17-24	A307G-24	0.001010	1760
WA17-25	A307G-23	0.0020200	3268
WA17-26	A307G-22	0.005050	9204
WA17-37	A307G-22	0.005050	8710
WA17-38	A307G-23	0.0020200	3555
WA17-39	A307G-24	0.001010	1617
WA17-40	A307G-25	0.0005050	883
WA17-41	A307G-26	0.0002020	309
WA17-42	A307G-27	0.0001010	175

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1776557
Intercept, b = -62.42
R^2 = 0.9981
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 25-Jul-12 Analysis date: 25-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WA17-28	18463A-C-1	C	NA	5.00	NA	29	200	0.00206	NA
WA17-29	18463A-C-1	C	NA	5.00	NA	37	200	0.00224	NA
WA17-30	18463A-QC-0.02-1	F	0.1010	5.00	0.02020	480	200	0.01221	60.46
WA17-31	18463A-QC-0.02-1	F	0.1010	5.00	0.02020	474	200	0.01208	59.79
WA17-32	18464A-T-1	T		5.00		3578	4000	1.639	
WA17-33	18464A-T-1	T		5.00		3716	4000	1.701	
WA17-34	18465A-T-1	T		5.00		3891	4000	1.780	
WA17-35	18465A-T-1	T		5.00		3666	4000	1.679	

NOTES *1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

PROJECT INFORMATION

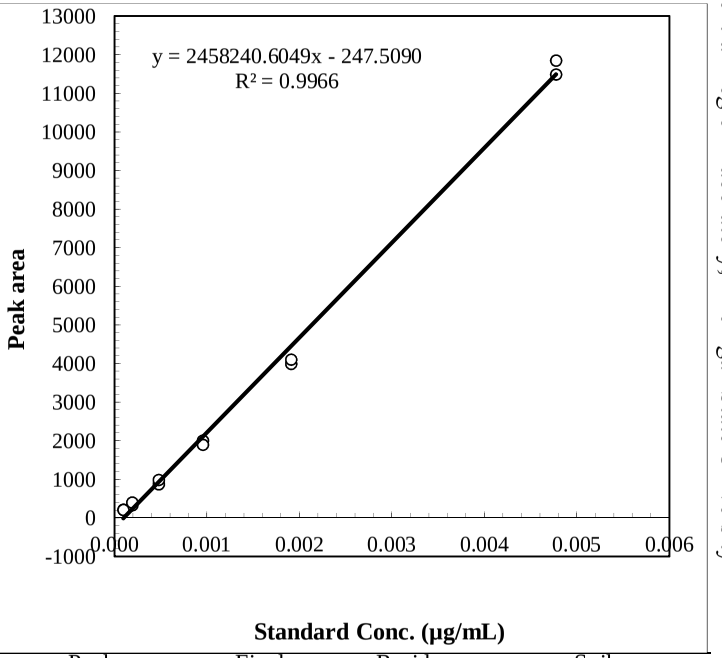
PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WA17
Analysis for:	TFNG	Field Research Director:	Dan Groenendale
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 25-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WA17-21A	A307G-27	0.00009550	214
WA17-22	A307G-26	0.0001910	329
WA17-23	A307G-25	0.0004775	873
WA17-24	A307G-24	0.00095500	2005
WA17-25	A307G-23	0.0019100	3992
WA17-26	A307G-22	0.004775	11850
WA17-37	A307G-22	0.004775	11490
WA17-38	A307G-23	0.0019100	4103
WA17-39	A307G-24	0.00095500	1897
WA17-40A	A307G-25	0.0004775	986
WA17-41A	A307G-26	0.0001910	400
WA17-42	A307G-27	0.00009550	209

---- ----

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2458241
Intercept, b = -247.51
R^2 = 0.9966
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 25-Jul-12 Analysis date: 25-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WA17-28	18463A-C-1	C	NA	5.00	NA	459	200	0.0115	NA
WA17-29	18463A-C-1	C	NA	5.00	NA	437	200	0.01114	NA
WA17-30	18463A-QC-0.02-1	F	0.0955	5.00	0.01910	1108	200	0.02206	115.48
WA17-31	18463A-QC-0.02-1	F	0.0955	5.00	0.01910	1017	200	0.02058	107.73
WA17-32	18464A-T-1	T		5.00		1085	4000	0.4336	
WA17-33	18464A-T-1	T		5.00		1191	4000	0.4681	
WA17-34	18465A-T-1	T		5.00		1137	4000	0.4506	
WA17-35	18465A-T-1	T		5.00		1204	4000	0.4724	

NOTES

*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

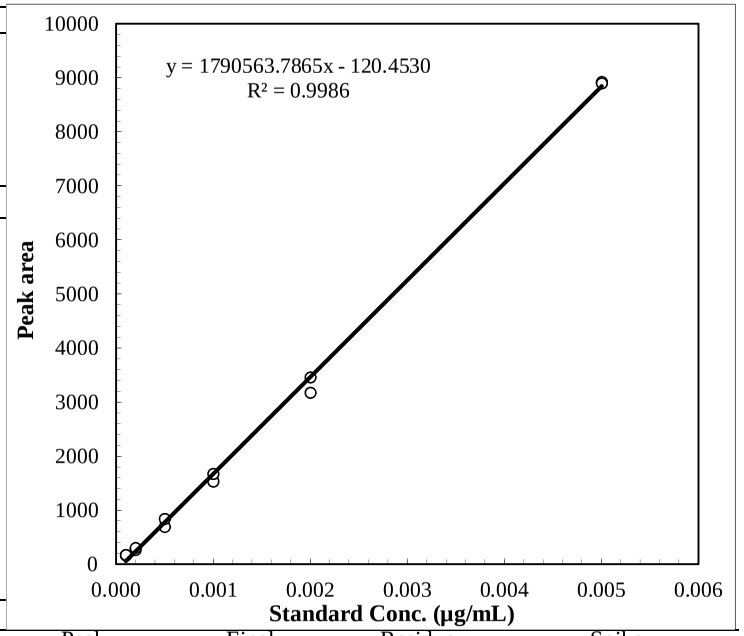
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.:	09358.11-WA17
Analysis for:	TFNA	Field Research Director:	Dan Groenendale
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 25-Jul-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
WA17-21	A307G-27	0.0001001	169
WA17-22	A307G-26	0.0002002	266
WA17-23	A307G-25	0.0005005	692
WA17-24	A307G-24	0.001001	1532
WA17-25	A307G-23	0.0020020	3174
WA17-26	A307G-22	0.005005	8924
WA17-37	A307G-22	0.005005	8899
WA17-38	A307G-23	0.0020020	3458
WA17-39	A307G-24	0.001001	1672
WA17-40	A307G-25	0.0005005	840
WA17-41	A307G-26	0.0002002	302
WA17-42	A307G-27	0.0001001	172

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1790564
Intercept, b = -120.45
R^2 = 0.9986
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 25-Jul-12 Analysis date: 25-Jul-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
WA17-28	18463A-C-1	C	NA	5.00	NA	66	200	0.00417	NA
WA17-29	18463A-C-1	C	NA	5.00	NA	57	200	0.00396	NA
WA17-30	18463A-QC-0.02-1	F	0.1001	5.00	0.02002	743	200	0.0193	96.35
WA17-31	18463A-QC-0.02-1	F	0.1001	5.00	0.02002	777	200	0.0200	100.14
WA17-32	18464A-T-1	T		5.00		364	4000	0.2164	
WA17-33	18464A-T-1	T		5.00		388	4000	0.2272	
WA17-34	18465A-T-1	T		5.00		439	4000	0.2500	
WA17-35	18465A-T-1	T		5.00		372	4000	0.2200	

NOTES

*1: C=Control, F=Fortified, T=Treated

*2: Spike amount = (Spike added) ÷ (Sample weight)

*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)

*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100

"NA" stands for "Not applicable"

Field Trial Data

CALCULATION PAGE

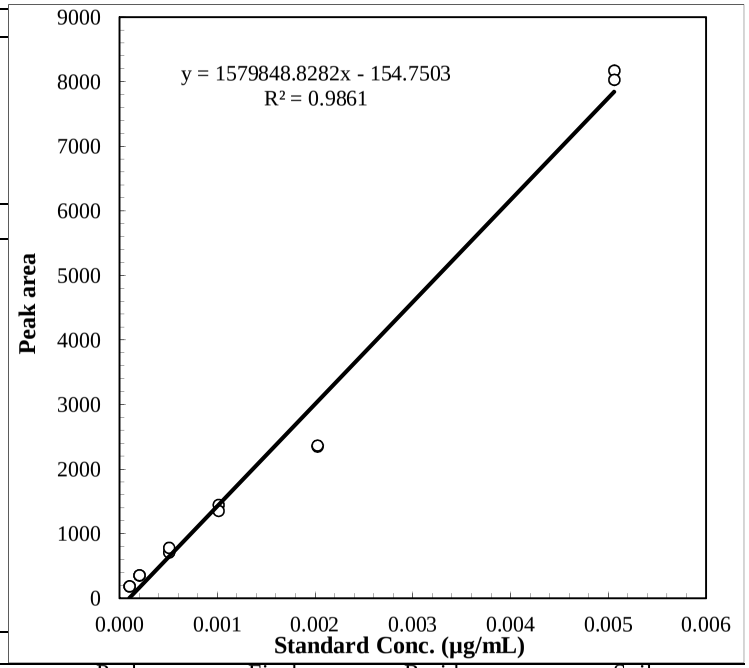
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.for SS:	09358.11-WA*18
Analysis for:	TFNA-AM	Field Research Director:	John Harvey
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 07-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
SS-10	A307G-27	0.0001012	185
SS-11	A307G-26	0.0002024	352
SS-12	A307G-25	0.0005060	708
SS-13	A307G-24	0.001012	1445
SS-14	A307G-23	0.0020240	2349
SS-15	A307G-22	0.005060	8173
SS-23	A307G-22	0.005060	8030
SS-24	A307G-23	0.0020240	2365
SS-25	A307G-24	0.001012	1353
SS-26	A307G-25	0.0005060	781
SS-27A	A307G-26	0.0002024	356
SS-28	A307G-27	0.0001012	185

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1579849
Intercept, b = -154.75
R^2 = 0.9861
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 07-Aug-12 Analysis date: 07-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
SS-17	18470A-C-3	C	NA	5.00	NA	0	200	< 0.02	NA
SS-18	18470A-QC-0.2-1	F	1.012	5.00	0.2024	1138	1000	0.1637	80.86
SS-19	18470A-0.2-SS 1	F	1.012	5.00	0.2024	696	1000	0.1077	53.21
SS-20	18470A-0.2-SS 2	F	1.012	5.00	0.2024	574	1000	0.0923	45.58
SS-21	18470A-0.2-SS 3	F	1.012	5.00	0.2024	689	1000	0.1068	52.77

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Storage Stability Data

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.for SS:	09358.11-WA*18
Analysis for:	Flonicamid (IKI-220)	Field Research Director:	John Harvey
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 07-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
SS-10	A307G-27	0.0001010	207
SS-11	A307G-26	0.0002020	491
SS-12	A307G-25	0.0005050	1062
SS-13	A307G-24	0.001010	2361
SS-14	A307G-23	0.0020200	4495
SS-15	A307G-22	0.005050	11201
SS-23	A307G-22	0.005050	11030
SS-24	A307G-23	0.0020200	4579
SS-25	A307G-24	0.001010	2333
SS-26	A307G-25	0.0005050	1118
SS-27	A307G-26	0.0002020	412
SS-28	A307G-27	0.0001010	215

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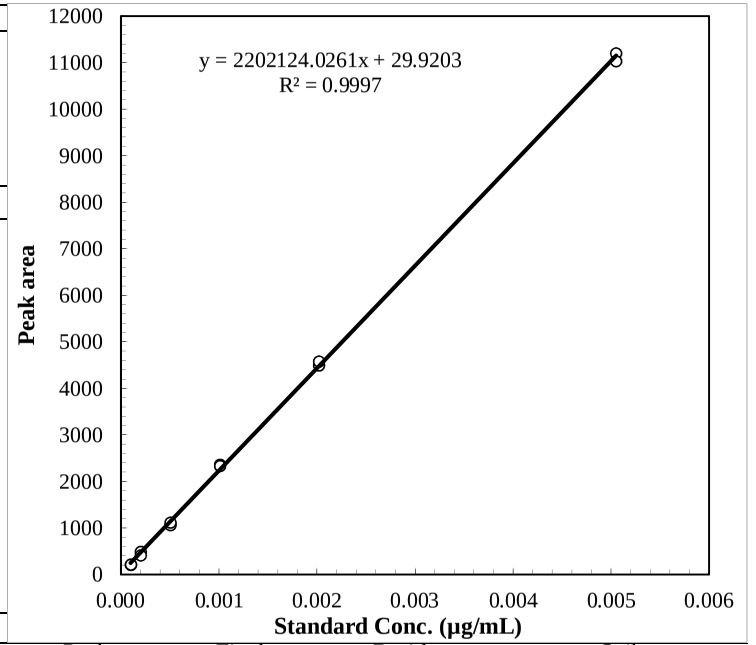
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2202124
Intercept, b = 29.92
R^2 = 0.9997
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 07-Aug-12 Analysis date: 07-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
SS-17	18470A-C-3	C	NA	5.00	NA	8	200	-0.000398166	NA
SS-18	18470A-QC-0.2-1	F	1.010	5.00	0.2020	2139	1000	0.1915	94.83
SS-19	18470A-0.2-SS 1	F	1.006	5.00	0.2012	1050	1000	0.0926	46.05
SS-20	18470A-0.2-SS 2	F	1.006	5.00	0.2012	988	1000	0.0870	43.25
SS-21	18470A-0.2-SS 3	F	1.006	5.00	0.2012	992	1000	0.0874	43.43

NOTES *1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Storage Stability Data

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.for SS:	09358.11-WA*18
Analysis for:	TFNG	Field Research Director:	John Harvey
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 07-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
SS-10	A307G-27	0.00009550	272
SS-11	A307G-26	0.0001910	520
SS-12	A307G-25	0.0004775	1408
SS-13	A307G-24	0.00095500	2548
SS-14	A307G-23	0.0019100	5257
SS-15	A307G-22	0.004775	13378
SS-23	A307G-22	0.004775	13032
SS-24	A307G-23	0.0019100	5227
SS-25	A307G-24	0.00095500	2772
SS-26	A307G-25	0.0004775	1196
SS-27	A307G-26	0.0001910	529
SS-28A	A307G-27	0.00009550	241

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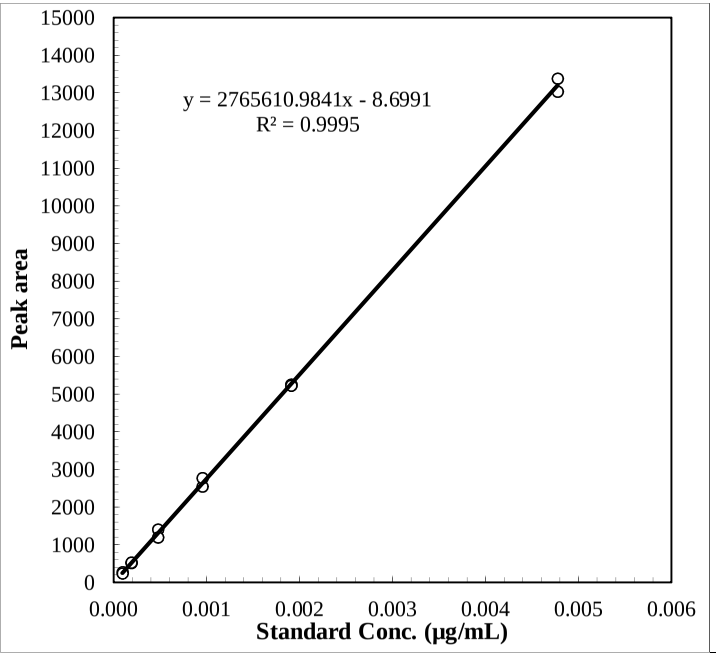
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2765611
Intercept, b = -8.70
R^2 = 0.9995
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 07-Aug-12 Analysis date: 07-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
SS-17	18470A-C-3	C	NA	5.00	NA	26	200	0.00050	NA
SS-18	18470A-QC-0.2-1	F	0.9550	5.00	0.1910	2330	1000	0.1691	88.55
SS-19	18470A-0.2-SS 1	F	0.9970	5.00	0.1994	1153	1000	0.0840	42.13
SS-20	18470A-0.2-SS 2	F	0.9970	5.00	0.1994	1235	1000	0.0899	45.11
SS-21	18470A-0.2-SS 3	F	0.9970	5.00	0.1994	1240	1000	0.0903	45.29

NOTES

*1: C=Control, F=Fortified, T=Treated

*2: Spike amount = (Spike added) ÷ (Sample weight)

*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)

*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100

"NA" stands for "Not applicable"

Storage Stability Data

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.for SS:	09358.11-WA*18
Analysis for:	TFNA	Field Research Director:	John Harvey
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Oil	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 07-Aug-12

RTSDB Page	Calibration standard ID (or name)	Calibration standard Conc.(µg/mL)	Peak Area
SS-10	A307G-27	0.0001001	209
SS-11	A307G-26	0.0002002	384
SS-12	A307G-25	0.0005005	935
SS-13	A307G-24	0.001001	1961
SS-14	A307G-23	0.0020020	3832
SS-15	A307G-22	0.005005	10410
SS-23	A307G-22	0.005005	10098
SS-24	A307G-23	0.0020020	3848
SS-25	A307G-24	0.001001	1936
SS-26	A307G-25	0.0005005	924
SS-27	A307G-26	0.0002002	393
SS-28	A307G-27	0.0001001	222

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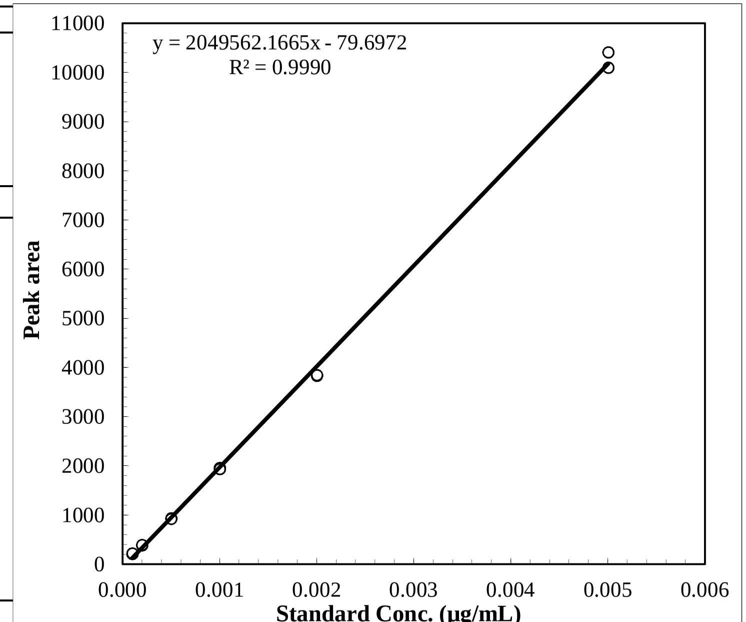
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2049562
Intercept, b = -79.70
R^2 = 0.9990
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 07-Aug-12 Analysis date: 07-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
SS-17	18470A-C-3	C	NA	5.00	NA		200	< 0.02	NA
SS-18	18470A-QC-0.2-1	F	1.001	5.00	0.2002	1968	1000	0.1998	99.81
SS-19	18470A-0.2-SS 1	F	1.001	5.00	0.2002	786	1000	0.0845	42.20
SS-20	18470A-0.2-SS 2	F	1.001	5.00	0.2002	918	1000	0.0974	48.63
SS-21	18470A-0.2-SS 3	F	1.001	5.00	0.2002	836	1000	0.0894	44.63

NOTES

*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Storage Stability Data

CALCULATION PAGE

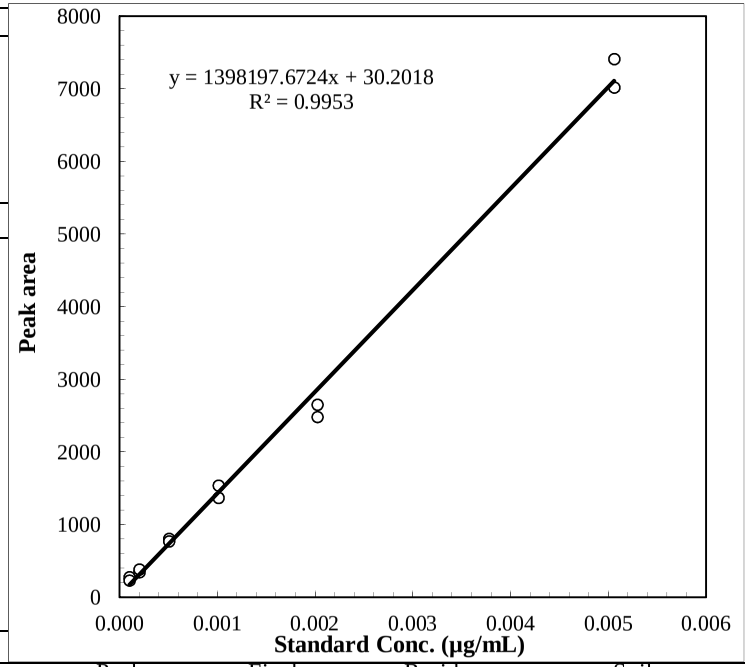
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.for SS:	09358.11-ID12
Analysis for:	TFNA-AM	Field Research Director:	Will Meeks
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 08-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
SS-39	A307G-27	0.0001012	276
SS-40	A307G-26	0.0002024	343
SS-41	A307G-25	0.0005060	804
SS-42	A307G-24	0.001012	1537
SS-43	A307G-23	0.0020240	2481
SS-44	A307G-22	0.005060	7409
SS-52	A307G-22	0.005060	7017
SS-53	A307G-23	0.0020240	2650
SS-54	A307G-24	0.001012	1368
SS-55	A307G-25	0.0005060	769
SS-56	A307G-26	0.0002024	382
SS-57	A307G-27	0.0001012	230

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 1398198
Intercept, b = 30.20
R^2 = 0.9953
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 08-Aug-12 Analysis date: 08-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
SS-46	18352A-C-1	C	NA	5.00	NA	0	200	< 0.02	NA
SS-47	18352A-QC-0.2-1	F	1.012	5.00	0.2024	1177	1000	0.1640	81.05
SS-48	18352A-0.2-SS 1	F	1.012	5.00	0.2024	1168	1000	0.1628	80.41
SS-49	18352A-0.2-SS 2	F	1.012	5.00	0.2024	1097	1000	0.1526	75.39
SS-50	18352A-0.2-SS 3	F	1.012	5.00	0.2024	1195	1000	0.1666	82.32

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Storage Stability Data

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.for SS:	09358.11-ID12
Analysis for:	Flonicamid (IKI-220)	Field Research Director:	Will Meeks
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 08-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
SS-39	A307G-27	0.0001010	224
SS-40	A307G-26	0.0002020	439
SS-41	A307G-25	0.0005050	1101
SS-42	A307G-24	0.001010	2285
SS-43	A307G-23	0.0020200	4310
SS-44	A307G-22	0.005050	11818
SS-52	A307G-22	0.005050	11179
SS-53	A307G-23	0.0020200	4295
SS-54	A307G-24	0.001010	2181
SS-55	A307G-25	0.0005050	1025
SS-56	A307G-26	0.0002020	444
SS-57	A307G-27	0.0001010	198

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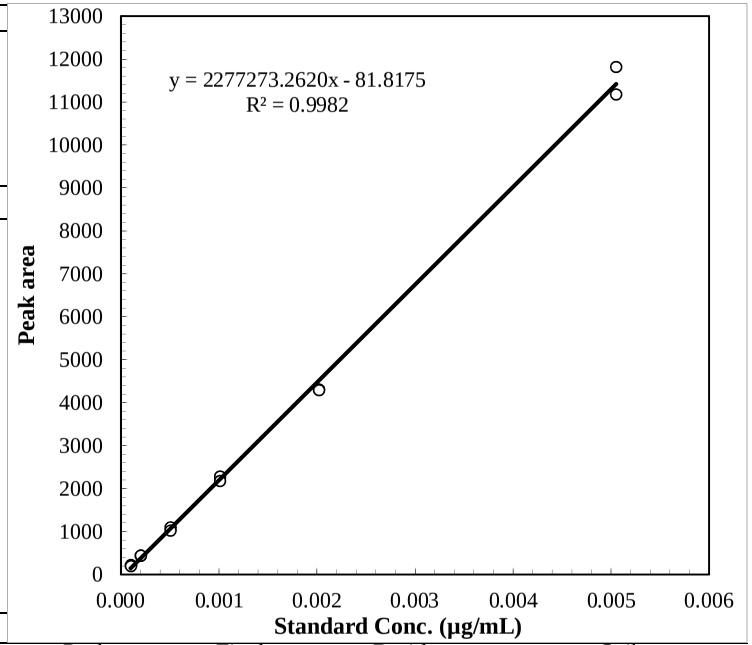
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2277273
Intercept, b = -81.82
R^2 = 0.9982
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 08-Aug-12 Analysis date: 08-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
SS-46	18352A-C-1	C	NA	5.00	NA	33	200	0.00202	NA
SS-47	18352A-QC-0.2-1	F	1.010	5.00	0.2020	1773	1000	0.1629	80.64
SS-48	18352A-0.2-SS 1	F	1.006	5.00	0.2012	1896	1000	0.1737	86.33
SS-49	18352A-0.2-SS 2	F	1.006	5.00	0.2012	1792	1000	0.1646	81.79
SS-50	18352A-0.2-SS 3	F	1.006	5.00	0.2012	1898	1000	0.1739	86.42

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Storage Stability Data

CALCULATION PAGE

PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.for SS:	09358.11-ID12
Analysis for:	TFNG	Field Research Director:	Will Meeks
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 08-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
SS-39	A307G-27	0.00009550	411
SS-40	A307G-26	0.0001910	470
SS-41	A307G-25	0.0004775	1116
SS-42	A307G-24	0.00095500	2830
SS-43	A307G-23	0.0019100	5152
SS-44	A307G-22	0.004775	13335
SS-52	A307G-22	0.004775	12809
SS-53	A307G-23	0.0019100	5293
SS-54	A307G-24	0.00095500	2490
SS-55	A307G-25	0.0004775	1563
SS-56	A307G-26	0.0001910	474
SS-57	A307G-27	0.00009550	220

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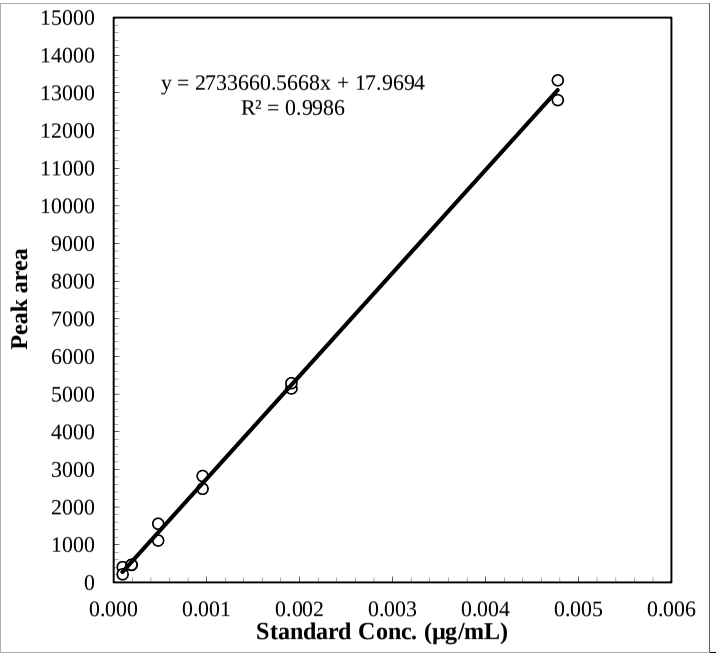
Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2733661
Intercept, b = 17.97
R^2 = 0.9986
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 08-Aug-12 Analysis date: 08-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
SS-46	18352A-C-1	C	NA	5.00	NA	415	200	0.00581	NA
SS-47	18352A-QC-0.2-1	F	0.9550	5.00	0.1910	2274	1000	0.1651	86.42
SS-48	18352A-0.2-SS 1	F	0.9970	5.00	0.1994	2282	1000	0.1656	83.07
SS-49	18352A-0.2-SS 2	F	0.9970	5.00	0.1994	2169	1000	0.1574	78.92
SS-50	18352A-0.2-SS 3	F	0.9970	5.00	0.1994	2202	1000	0.1598	80.13

NOTES

*1: C=Control, F=Fortified, T=Treated

*2: Spike amount = (Spike added) ÷ (Sample weight)

*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)

*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100

"NA" stands for "Not applicable"

Storage Stability Data

CALCULATION PAGE

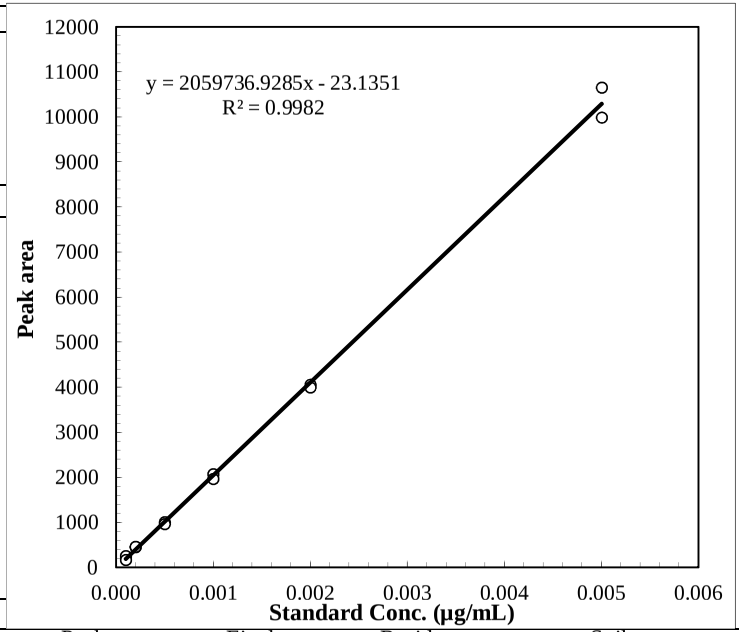
PROJECT INFORMATION

PR Number:	09358	Laboratory ID:	09358.11-MIR05
Chemical:	Flonicamid	Field ID No.for SS:	09358.11-ID12
Analysis for:	TFNA	Field Research Director:	Will Meeks
Commodity:	Mint	Analyst(s):	Eina Abouzied&Lester Geissel
Crop part:	Mint Tops (leaves&stems)	Instrument:	UPLC/MS/MS

CALIBRATION DATA Date of calibration analysis: 08-Aug-12

RTSDB Page	Calibration standard ID (or name)	Conc.(µg/mL)	Peak Area
SS-39	A307G-27	0.0001001	251
SS-40	A307G-26	0.0002002	449
SS-41	A307G-25	0.0005005	1004
SS-42	A307G-24	0.001001	2070
SS-43	A307G-23	0.0020020	4056
SS-44	A307G-22	0.005005	10653
SS-52	A307G-22	0.005005	9987
SS-53	A307G-23	0.0020020	3993
SS-54	A307G-24	0.001001	1964
SS-55	A307G-25	0.0005005	962
SS-56	A307G-26	0.0002002	457
SS-57	A307G-27	0.0001001	164

Type: Linear regression
Equation: $y = mx + b$
Slope, m = 2059737
Intercept, b = -23.14
R^2 = 0.9982
LOQ (µg/g) = 0.02



ANALYTICAL DATA Extraction date: 08-Aug-12 Analysis date: 08-Aug-12

RTSDB Page No.	Sample ID	Sample Type *1	Spike Added (µg)	Sample Weight (g)	Spike Conc. (µg/g)*2	Peak Area (counts)	Final Volume (mL)	Residue Found (µg/g)*3	Spike Recovery (%)*4
SS-46	18352A-C-1	C	NA	5.00	NA	31	200	0.00105	NA
SS-47	18352A-QC-0.2-1	F	1.001	5.00	0.2002	2111	1000	0.2072	103.51
SS-48	18352A-0.2-SS 1	F	1.001	5.00	0.2002	1535	1000	0.1513	75.57
SS-49	18352A-0.2-SS 2	F	1.001	5.00	0.2002	1307	1000	0.1292	64.51
SS-50	18352A-0.2-SS 3	F	1.001	5.00	0.2002	1579	1000	0.1556	77.71

NOTES
*1: C=Control, F=Fortified, T=Treated
*2: Spike amount = (Spike added) ÷ (Sample weight)
*3: Residue amount = [(Area - b) ÷ m] x (Final vol/Sample wt)
*4. Recovery (%) =(Residue found) ÷ (spike conc) * 100
"NA" stands for "Not applicable"

Storage Stability Data

IR-4 LOD / LOQ Calculator		Analysis Date	Observed Amount	Percent Recovery	Number of Observations	One-Tailed 't' statistic
Project Information		19-Jul-12	0.01494	73.94	1	∞
		19-Jul-12	0.01448	71.69	2	31.821
IR-4 LOD / LOQ Calculator	09358	19-Jul-12	0.01411	69.87	3	6.965
	Flonicamid	02-Aug-12	0.01462	72.40	4	4.541
	TFNA-AM	24 and 25 Jul*	0.01632	80.78	5	3.747
	Mint	25-Jul-12	0.01616	80.01	6	3.365
	Mint Tops (leaves&stems)				7	3.143
	09358.11-MIR05				8	2.998
Analyst:	Eina Abouzied&Lester Geissel				9	2.896
Date Printed:					10	2.821
Calculations					11	2.764
					12	2.718
Spike Amount:	0.02020 ppm				13	2.681
Number of Observations:	6				14	2.650
Average Amount Observed:	0.01511 ppm				15	2.624
Standard Deviation:	0.000918 ppm				16	2.602
Average Recovery:	74.78 ± 4.5 %				17	2.583
One-Tailed 't' statistic:	3.365 for n = 6				18	2.567
Limit of Detection (LOD):	0.003090 ppm				19	2.552
Limit of Quantitation (LOQ):	0.00927 ppm				20	2.539
Formulas					21	2.528
					22	2.518
LOD = (Standard Deviation) x (One-Tailed t-Statistic)					23	2.508
LOQ = (3) x (LOD)					24	2.500
Based on the method described in Handbook of Environmental Analysis, Fourth Edition, Genium Publishing Corporation, 1999 by Roy-Keith Smith						

*same set injected on two different days

IR-4 LOD / LOQ Calculator		Analysis Date	Observed Amount	Percent Recovery	Number of Observations	One-Tailed 't' statistic
Project Information		19-Jul-12	0.01486	73.56	1	∞
		19-Jul-12	0.01358	67.21	2	31.821
IR-4 LOD / LOQ Calculator	09358	19-Jul-12	0.01432	70.89	3	6.965
	Flonicamid	02-Aug-12	0.01190	58.91	4	4.541
	Flonicamid(IKI-220)	24- and 25-Jul-12*	0.01441	71.33	5	3.747
	Mint	25-Jul-12	0.01215	60.13	6	3.365
	Mint Tops (leaves&stems)				7	3.143
	09358.11-MIR05				8	2.998
Analyst:	Eina Abouzied&Lester Geissel				9	2.896
Date Printed:					10	2.821
Calculations					11	2.764
					12	2.718
Spike Amount:	0.02020 ppm				13	2.681
Number of Observations:	6				14	2.650
Average Amount Observed:	0.01353 ppm				15	2.624
Standard Deviation:	0.001244 ppm				16	2.602
Average Recovery:	67.00 ± 6.2 %				17	2.583
One-Tailed 't' statistic:	3.365 for n = 6				18	2.567
Limit of Detection (LOD):	0.004187 ppm				19	2.552
Limit of Quantitation (LOQ):	0.01256 ppm				20	2.539
Formulas					21	2.528
					22	2.518
LOD = (Standard Deviation) x (One-Tailed t-Statistic)					23	2.508
LOQ = (3) x (LOD)					24	2.500

Based on the method described in Handbook of Environmental Analysis, Fourth Edition, Genium Publishing Corporation, 1999 by Roy-Keith Smith

*same set injected on two different days

IR-4 LOD / LOQ Calculator		Analysis Date	Observed Amount	Percent Recovery	Number of Observations	One-Tailed 't' statistic
Project Information		19-Jul-12	0.02093	109.60	1	∞
		19-Jul-12	0.02048	107.20	2	31.821
IR-4 LOD / LOQ Calculator	09358	19-Jul-12	0.02149	112.51	3	6.965
	Flonicamid	02-Aug-12	0.02206	115.48	4	4.541
	TFNG	24 and 25 Jul-12*	0.02071	108.43	5	3.747
	Mint	25-Jul-12	0.02132	111.60	6	3.365
	Mint Tops (leaves&stems)				7	3.143
	09358.11-MIR05				8	2.998
Analyst:	Eina Abouzied&Lester Geissel				9	2.896
Date Printed:					10	2.821
Calculations					11	2.764
					12	2.718
Spike Amount:	0.01910 ppm				13	2.681
Number of Observations:	6				14	2.650
Average Amount Observed:	0.02116 ppm				15	2.624
Standard Deviation:	0.000576 ppm				16	2.602
Average Recovery:	110.81 ± 3 %				17	2.583
One-Tailed 't' statistic:	3.365 for n = 6				18	2.567
Limit of Detection (LOD):	0.001938 ppm				19	2.552
Limit of Quantitation (LOQ):	0.00581 ppm				20	2.539
Formulas					21	2.528
					22	2.518
LOD = (Standard Deviation) x (One-Tailed t-Statistic)					23	2.508
LOQ = (3) x (LOD)					24	2.500
Based on the method described in Handbook of Environmental Analysis, Fourth Edition, Genium Publishing Corporation, 1999 by Roy-Keith Smith						

*same set injected on two different days

Attachment B: Calculation Worksheets, Standard Curves and LOD/LOQ Calculations

IR-4 LOD / LOQ Calculator		Analysis Date	Observed Amount	Percent Recovery	Number of Observations	One-Tailed 't' statistic
Project Information		19-Jul-12	0.02054	102.58	1	∞
		19-Jul-12	0.02086	104.20	2	31.821
IR-4 LOD / LOQ Calculator	09358	19-Jul-12	0.02146	107.19	3	6.965
	Flonicamid	02-Aug-12	0.01854	92.60	4	4.541
	TFNA	24 and 25-Jul-12*	0.02227	111.22	5	3.747
	Mint	25-Jul-12	0.01967	98.25	6	3.365
	Mint Tops (leaves&stems)				7	3.143
	09358.11-MIR05				8	2.998
Analyst:	Eina Abouzied&Lester Geissel				9	2.896
Date Printed:					10	2.821
Calculations					11	2.764
					12	2.718
Spike Amount:	0.02002 ppm				13	2.681
Number of Observations:	6				14	2.650
Average Amount Observed:	0.02056 ppm				15	2.624
Standard Deviation:	0.001318 ppm				16	2.602
Average Recovery:	102.67 ± 6.6 %				17	2.583
One-Tailed 't' statistic:	3.365 for n = 6				18	2.567
Limit of Detection (LOD):	0.004437 ppm				19	2.552
Limit of Quantitation (LOQ):	0.01331 ppm				20	2.539
Formulas					21	2.528
					22	2.518
LOD = (Standard Deviation) x (One-Tailed t-Statistic)					23	2.508
LOQ = (3) x (LOD)					24	2.500
Based on the method described in Handbook of Environmental Analysis, Fourth Edition, Genium Publishing Corporation, 1999 by Roy-Keith Smith						

*same set injected on two different days

IR-4 LOD / LOQ Calculator		Analysis Date	Observed Amount	Percent Recovery	Number of Observations	One-Tailed 't' statistic
Project Information		11-Jul-12	0.01876	92.70	1	∞
		11-Jul-12	0.01969	97.26	2	31.821
IR-4 LOD / LOQ Calculator	09358	11-Jul-12	0.02063	101.92	3	6.965
	Flonicamid	06-Aug-12	0.01649	81.48	4	4.541
	TFNA-AM	30-Jul-12	0.02097	103.59	5	3.747
	Mint	30-Jul-12	0.02015	99.58	6	3.365
	Mint Oil				7	3.143
	09358.11-MIR05				8	2.998
Analyst:	Eina Abouzied&Lester Geiss				9	2.896
Date Printed:					10	2.821
Calculations					11	2.764
					12	2.718
Spike Amount:	0.02020 ppm				13	2.681
Number of Observations:	6				14	2.650
Average Amount Observed:	0.01945 ppm				15	2.624
Standard Deviation:	0.001642 ppm				16	2.602
Average Recovery:	96.09 ± 8.1 %				17	2.583
One-Tailed 't' statistic:	3.365 for n = 6				18	2.567
Limit of Detection (LOD):	0.005524 ppm				19	2.552
Limit of Quantitation (LOQ):	0.01657 ppm				20	2.539
Formulas					21	2.528
					22	2.518
LOD = (Standard Deviation) x (One-Tailed t-Statistic)					23	2.508
LOQ = (3) x (LOD)					24	2.500
Based on the method described in Handbook of Environmental Analysis, Fourth Edition, Genium Publishing Corporation, 1999 by Roy-Keith Smith						

IR-4 LOD / LOQ Calculator		Analysis Date	Observed Amount	Percent Recovery	Number of Observations	One-Tailed 't' statistic
Project Information		11-Jul-12	0.02083	103.14	1	∞
		11-Jul-12	0.01756	86.93	2	31.821
IR-4 LOD / LOQ Calculator	09358	11-Jul-12	0.02053	101.63	3	6.965
	Flonicamid	06-Aug-12	0.01794	88.83	4	4.541
	Flonicamid(IKI-220)	30-Jul-12	0.01985	98.25	5	3.747
	Mint	30-Jul-12	0.01988	98.41	6	3.365
	Mint Oil				7	3.143
	09358.11-MIR05				8	2.998
Analyst:	Eina Abouzied&Lester Geiss				9	2.896
Date Printed:					10	2.821
Calculations					11	2.764
					12	2.718
Spike Amount:	0.02020 ppm				13	2.681
Number of Observations:	6				14	2.650
Average Amount Observed:	0.01943 ppm				15	2.624
Standard Deviation:	0.001361 ppm				16	2.602
Average Recovery:	96.20 ± 6.7 %				17	2.583
One-Tailed 't' statistic:	3.365 for n = 6				18	2.567
Limit of Detection (LOD):	0.004579 ppm				19	2.552
Limit of Quantitation (LOQ):	0.01374 ppm				20	2.539
Formulas					21	2.528
					22	2.518
LOD = (Standard Deviation) x (One-Tailed t-Statistic)					23	2.508
LOQ = (3) x (LOD)					24	2.500

Based on the method described in Handbook of Environmental Analysis, Fourth Edition, Genium Publishing Corporation, 1999 by Roy-Keith Smith

IR-4 LOD / LOQ Calculator		Analysis Date	Observed Amount	Percent Recovery	Number of Observations	One-Tailed 't' statistic
Project Information		11-Jul-12	0.01778	93.08	1	∞
		11-Jul-12	0.01668	87.31	2	31.821
IR-4 LOD / LOQ Calculator	09358	11-Jul-12	0.01781	93.26	3	6.965
	Flonicamid	06-Aug-12	0.01505	78.79	4	4.541
	TFNG	30-Jul-12	0.01572	82.31	5	3.747
	Mint	30-Jul-12	0.01814	94.96	6	3.365
	Mint Oil				7	3.143
	09358.11-MIR05				8	2.998
Analyst:	Eina Abouzied&Lester Geisse				9	2.896
Date Printed:					10	2.821
Calculations					11	2.764
					12	2.718
Spike Amount:	0.01910 ppm				13	2.681
Number of Observations:	6				14	2.650
Average Amount Observed:	0.01686 ppm				15	2.624
Standard Deviation:	0.001264 ppm				16	2.602
Average Recovery:	88.28 ± 6.6 %				17	2.583
One-Tailed 't' statistic:	3.365 for n = 6				18	2.567
Limit of Detection (LOD):	0.004255 ppm				19	2.552
Limit of Quantitation (LOQ):	0.01276 ppm				20	2.539
Formulas					21	2.528
					22	2.518
LOD = (Standard Deviation) x (One-Tailed t-Statistic)					23	2.508
LOQ = (3) x (LOD)					24	2.500
Based on the method described in Handbook of Environmental Analysis, Fourth Edition, Genium Publishing Corporation, 1999 by Roy-Keith Smith						

IR-4 LOD / LOQ Calculator		Analysis Date	Observed Amount	Percent Recovery	Number of Observations	One-Tailed 't' statistic
Project Information		11-Jul-12	0.01771	88.47	1	∞
		11-Jul-12	0.01807	90.27	2	31.821
IR-4 LOD / LOQ Calculator	09358	11-Jul-12	0.01854	92.62	3	6.965
	Flonicamid	06-Aug-12	0.01726	86.11	4	4.541
	TFNA	30-Jul-12	0.01774	88.63	5	3.747
	Mint	30-Jul-12	0.02046	102.21	6	3.365
	Mint Oil				7	3.143
	09358.11-MIR05				8	2.998
Analyst:	Eina Abouzied&Lester Geiss				9	2.896
Date Printed:					10	2.821
Calculations					11	2.764
					12	2.718
Spike Amount:	0.02002 ppm				13	2.681
Number of Observations:	6				14	2.650
Average Amount Observed:	0.01830 ppm				15	2.624
Standard Deviation:	0.001143 ppm				16	2.602
Average Recovery:	91.39 ± 5.7 %				17	2.583
One-Tailed 't' statistic:	3.365 for n = 6				18	2.567
Limit of Detection (LOD):	0.003847 ppm				19	2.552
Limit of Quantitation (LOQ):	0.01154 ppm				20	2.539
Formulas					21	2.528
					22	2.518
LOD = (Standard Deviation) x (One-Tailed t-Statistic)					23	2.508
LOQ = (3) x (LOD)					24	2.500
Based on the method described in Handbook of Environmental Analysis, Fourth Edition, Genium Publishing Corporation, 1999 by Roy-Keith Smith						

MIDWEST RESEARCH INSTITUTE
425 Volker Boulevard
Kansas City, Missouri 64110
Telephone (816) 753-7600
Telefax (816) 753-5519

Amended Certificate of Analysis

IKI-220 PAI, Lot No. 9803

Original data and GLP reserve sample are archived under MRI Project No. 310260.1.093.01

Data Requirement

Good Laboratory Practice Standards of the U.S. Environmental Protection Agency's Federal Insecticide, Fungicide, and Rodenticide Act, 40 *CFR* Part 160.

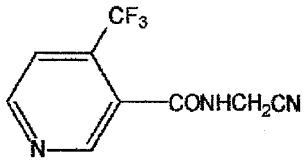
Performing Laboratory

Midwest Research Institute
425 Volker Blvd.
Kansas City, MO 64110

Study Sponsor

Ishihara Sangyo Kaisha, Ltd.
Biosciences Business Headquarters
3-15 Edobori 1-Chome, Nishi-ku
Osaka 550-0002 JAPAN

Compound Identification



Common Name:	IKI-220-PAI; Flonicamid
IUPAC Chemical Name:	N-(cyanomethyl)-4-(trifluoromethyl)nicotinamide
CA Chemical Name:	N-(cyanomethyl)-4-(trifluoromethyl)-3-pyridinecarboxamide
Empirical Formula:	C9H6F3N3O
Molecular Weight:	229.17
CAS Number:	158062-67-0
Lot No.:	9803

Experimental Techniques

Purity was determined using DSC and HPLC purity profiling methodology

Quality

Purity (%):	99.9 ± 0.1 %
Identity:	Conforms
Storage Conditions:	Frozen (~-20 °C)
Date of Analysis:	May 15, 2009
Expiration Date:	May 15, 2014
Initial Date of Issue[1]:	June 18, 2009

Approved: Paul J. Weller Date: July 22, 2009

Paul J. Weller, Study Director
Senior Chemist
Midwest Research Institute

ARS: 307
REC: 17MAR10
ATK

[1] Amended Certificate of Analysis issued to correct a typographical error on original issue of the Certificate of Analysis dated June 18, 2009.

MIDWEST RESEARCH INSTITUTE
425 Volker Boulevard
Kansas City, Missouri 64110
Telephone (816) 753-7600
Telefax (816) 753-5519

Certificate of Analysis

TFNA-AM, Lot No. 0006

Original data and GLP reserve sample are archived under MRI Project No. 310260.1.048

Data Requirement

Good Laboratory Practice Standards of the U.S. Environmental Protection Agency's Federal Insecticide, Fungicide, and Rodenticide Act, 40 *CFR* Part 160.105.

Performing Laboratory

Midwest Research Institute
425 Volker Blvd.
Kansas City, MO 64110

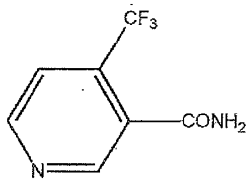
Study Sponsor

Ishihara Sangyo Kaisha, Ltd.
3-15, Edobori 1-Chome, Nishi-ku
Osaka, 550-0002 JAPAN

Compound Identification

Common Name:	TFNA-AM
IUPAC Chemical Name:	4-Trifluoromethylnicotinamide
Empirical Formula:	$C_7H_5F_3N_2O$
Molecular Weight:	190.12
CAS Number:	158062-71-6
Lot No.:	0006

Structure:



Experimental Techniques

Structure was verified using infrared spectrometry
Purity was determined using HPLC purity profiling methodology

Quality

Purity (%):	99.87% (HPLC)
Identity:	Conforms
Storage Conditions:	Frozen (–20 °C)
Date of Analysis:	January 6, 2006
Expiration Date:	January 6, 2011

EXACT COPY OF DATA
Signed C. Wiley
Date 2/24/06

Approved: Paul J. Weller Date: January 25, 2006
Paul J. Weller, Study Director
Senior Chemist
Midwest Research Institute

ARS: 308
RTK
REC: 17 MAR 10

MIDWEST RESEARCH INSTITUTE
425 Volker Boulevard
Kansas City, Missouri 64110
Telephone (816) 753-7600
Telefax (816) 753-5519

Certificate of Analysis

TFNA-AM, Lot No. 0006

Original data and GLP reserve sample are archived under MRI Project No. 310260.1.114.02

Data Requirement

Good Laboratory Practice Standards (40 *CFR* 160) of the U.S. Environmental Protection Agency's Federal Insecticide, Fungicide and Rodenticide Act, and 40 *CFR* 160.105, Test, Control, and Reference Substance Characterization.

Performing Laboratory

Midwest Research Institute
425 Volker Blvd.
Kansas City, MO 64110

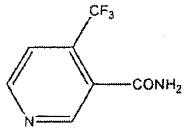
Study Sponsor

Ishihara Sangyo Kaisha, Ltd.
Biosciences Business Headquarters
3-15 Edobori 1-Chome, Nishi-ku
Osaka 550-0002 JAPAN

Compound Identification

Common Name:	TFNA-AM
IUPAC Chemical Name:	4-Trifluoromethylnicotinamide
Empirical Formula:	$C_7H_5F_3N_2O$
Molecular Weight:	190.12
CAS Number:	158062-21-6
Lot No.:	0006

Structure:



Recertification
ARS: 308
Rec: 28 Jan 11
RTK

Experimental Techniques

Purity was determined using HPLC purity profiling methodology results
Structure was previously verified by infrared spectrometry on this lot on January 6, 2006

Quality

Purity (%):	99.7%
Storage Conditions:	Frozen (~ -20°C)
Date of HPLC Analysis:	January 3, 2011
Expiration Date:	January 3, 2016

Approved: Walter R. Vandaveer Date: 1-14-11
Walter R. Vandaveer, Ph.D., Study Director
Senior Chemist
Midwest Research Institute

harlan™

CERTIFICATE OF ANALYSIS

Harlan Laboratories Study Number:	C16441
Sponsor:	Ishihara Sangyo Kaisha, Ltd, 3-15, Edobori 1-chome Nishi-ku, Osaka, 550-0002 Japan
Test Facility:	Harlan Laboratories Ltd. Zelgliweg 1 4452 Itingen Switzerland

Data of Test Item as supplied by the sponsor:

Identity:	TFNG
Batch:	0006-1
Storage:	In a freezer at -20 °C

Results:

Date of Analysis by Harlan Laboratories Ltd.:	December 10, 2008
Purity:	92.4%
Expiry Date:	December, 2013 *(as given by the sponsor based on the results of this study)*

The Result described in this certificate was achieved in compliance with the Swiss Ordinance relating to GLP, based on the OECD Principles of Good Laboratory Practice.

Issued by:

Dr. Nicole Tobler
(Study Director)
Date: March 05, 2009

Mr. Ramanan Sarvanantthan
(Quality Assurance)
Date: March 04, 2009

ARS: 309
REC: 17 MAR 10
RTK

MIDWEST RESEARCH INSTITUTE
425 Volker Boulevard
Kansas City, Missouri 64110
Telephone (816) 753-7600
Telefax (816) 753-5519

Certificate of Analysis

TFNA, Lot No. 0006

Original data and GLP reserve sample are archived under MRI Project No. 310260.1.047

Data Requirement

Good Laboratory Practice Standards of the U.S. Environmental Protection Agency's Federal Insecticide, Fungicide, and Rodenticide Act, 40 *CFR* Part 160.105..

Performing Laboratory

Midwest Research Institute
425 Volker Blvd.
Kansas City, MO 64110

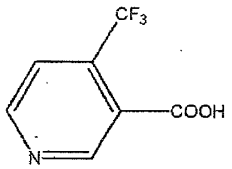
Study Sponsor

Ishihara Sangyo Kaisha, Ltd.
3-15, Edobori 1-Chome, Nishi-ku
Osaka, 550-0002 JAPAN

Compound Identification

Common Name:	TFNA
IUPAC Chemical Name:	4-Trifluoromethylnicotinic acid
Empirical Formula:	$C_7H_4F_3NO_2$
Molecular Weight:	191.11
CAS Number:	158063-66-2
Lot No.:	0006

Structure:



Experimental Techniques

Structure was verified using infrared spectrometry
Purity was determined using HPLC purity profiling methodology

Quality

Purity (wt.%):	100.00% (HPLC)
Identity:	Conforms
Storage Conditions:	Frozen (–20 °C)
Date of Analysis:	January 4, 2006
Expiration Date:	January 5, 2011

Approved: Paul J. Weller Date: 02/16/2006
Paul J. Weller, Study Director
Senior Chemist
Midwest Research Institute

ARS: 310
Rec: 17 MAR 10
RTK

MIDWEST RESEARCH INSTITUTE
425 Volker Boulevard
Kansas City, Missouri 64110
Telephone (816) 753-7600
Telefax (816) 753-5519

Certificate of Analysis

TFNA, Lot No. 0006

Original data and GLP reserve sample are archived under MRI Project No. 310260.1.114.01

Data Requirement

Good Laboratory Practice Standards (40 *CFR* 160) of the U.S. Environmental Protection Agency's Federal Insecticide, Fungicide and Rodenticide Act, and 40 *CFR* 160.105, Test, Control, and Reference Substance Characterization.

Performing Laboratory

Midwest Research Institute
425 Volker Blvd.
Kansas City, MO 64110

Study Sponsor

Ishihara Sangyo Kaisha, Ltd.
Biosciences Business Headquarters
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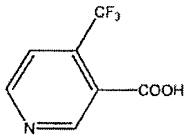
Osaka 550-0002 JAPAN

Compound Identification

Common Name:	TFNA
IUPAC Chemical Name:	4-Trifluoromethylnicotinic acid
Empirical Formula:	$C_7H_4F_3NO_2$
Molecular Weight:	191.11
CAS Number:	158063-66-2
Lot No.:	0006

Structure:



Recertification
ARS; 310
Rec; 28 Jan 11
RTK

Experimental Techniques

Purity was determined using HPLC purity profiling methodology results
Structure was verified by infrared spectrometry on this lot on January 4, 2006

Quality

Purity (%):	100.0%
Storage Conditions:	Frozen (~ –20°C)
Date of HPLC Analysis:	January 3, 2011
Expiration Date:	January 3, 2016

Approved: Walter R. Vandaveer Date: 1-14-11
Walter R. Vandaveer, Ph.D., Study Director
Senior Chemist
Midwest Research Institute

WORKING OUTLINES

Version #: 1 By: EG/LDG Date: 20-Jul-2012 Supersedes: None

9.0 WORKING OUTLINES FOR SPECIFIC ANALYTICAL PROCEDURES

9.166 Flonicamid and its Metabolites TFNA-AM, TFNA AND TFNG Analysis Procedure for Mint (Tops and Oil)

This working outline is an adaptation of two FMC Corporation Reports. Number P-3561M for the mint tops (leaves and stems) and number P-3567 for the mint oil.

Tops: Report Number P-3561M, "Analytical Methodology for IKI-220 (F1785) and its Major Metabolites in/on Peach, Potato Tuber, and Wheat Straw", was written by Audrey W. Chen on August 28, 2002, Princeton NJ, 08543, USA.

Oil: Report Number P-3567. "Magnitude of the Residues of IKI-220 on Cotton – USA in 2001" was written by Karen D. Dow on December 18, 2002, Princeton NJ, 08543, USA.

Abstract:
For tops, following method outline of appendix 1.A. Five (5.0) g of sample is extracted using an extraction solvent (acetonitrile:water, 50:50, v/v). The sample is shaken, decanted into flat bottom boiling flask.

For oil, following method outline of appendix 1 for Cotton Refined Oil. Two and a half (2.5) g of sample is partition twice using hexane and an extraction solvent (acetonitrile:water, 50:50, v/v). Extraction solvent is combined in a flat bottom boiling flask.

For both tops and oil, extraction solvent is evaporated to its aqueous remainder, filtered, acidified and made up to 50 mL. A portion of the extract (5 out of 50 mL) is used to go through the rest of procedure that includes partitioning with ethyl acetate, evaporation of the ethyl acetate to near dryness using N-EVAP Evaporator. The residues are dissolved in actonitrile:water, 50:50, v/v and then analyzed by HPLC/MS/MS.

9.166.1 Extraction and Cleanup

For Mint tops:

1. Accurately weigh 5.00 g of tops in a 50 mL centrifuge tube. If preparing a concurrent recovery, spike with flonicamid (IKI-220) and its three metabolites TFNA-AM, TFNA, TFNG by adding 1 mL or less of standard as needed to achieve desired concentrations. Allow sample to sit several minutes before proceeding for solvent to evaporate.

2. Using a graduated cylinder add 40 mL of an extraction solvent (acetonitrile:water, 50:50, v/v) to the centrifuge tube containing the sample.

3. Shake for 30 minutes on a wrist action shaker (Eberbach) set at 75.

4. Centrifuge for 10 minutes at approximately 5,500 rpm.

5. Decant solvent into 250 mL flat bottom boiling flask.

6. Using a graduated cylinder add 40 mL of the extraction solvent (acetonitrile:water, 50:50, v/v) to the flask. If needed, manually shake to disrupt pellet.

7. Repeat steps 3 – 5, combining extracts in the 250 mL flat bottom boiling flask. Skip to Step 12.

For Mint Oil:

8. Accurately weigh 2.50 g of mint oil in a 15 mL centrifuge tube. If preparing a concurrent recovery, spike with flonicamid (IKI-220) and its three metabolites TFNA-AM, TFNA, TFNG by adding 1 mL or less of standard as needed to achieve desired concentrations. Allow sample to sit several minutes before proceeding for solvent to evaporate. Transfer the 2.5 g oil sample to a 125 mL separatory funnel.

9. Rinse the centrifuge tube with 2 x 5 mL of hexane and transfer the rinsate to the separatory funnel.

10. Partition twice with 50 mL of 50% acetonitrile in milli- Q water. Shake for 30 – 40 s.

11. Drain 50% acetonitrile in Milli- Q water layers into the same 250 mL flat bottom boiling flask.

For Mint Tops and Oil:

12. Rotovap to aqueous remainder, 15 – 20 mL (water bath, 45 ± 5 °C). Then, add 0.6 mL of concentrated hydrochloric acid to the remaining solution.

13. Filter through a paper filter (Whatman No. 1, 7 cm) into a 100 mL graduated cylinder. Rinse flat bottom boiling flask with at least 2 x 10 mL water. Using water, bring the volume up to 50 mL.

14. Mix and transfer a 5.0 mL aliquot of filtered sample (step 13) using a volumetric pipet into a 15 mL polypropylene centrifuge tube.

15. Partition with ethyl acetate by vortexing three times (4 mL for 30 s, 4 mL for 30 s and 2 mL for 15 s). To separate layers, centrifuge for 3 min at approximately 5,500 rpm. After separation, collect ethyl acetate portions in another 15 mL polypropylene centrifuge tube.

16. Use an N-EVAP Evaporator (water bath, 25 ± 5 °C) under low nitrogen (just enough to produce a ripple on the surface) to remove the solvent to near dryness. DO NOT OVER DRY.

17. Dissolve residues in actonitrile:water, 50:50, v/v.

18. Transfer the residue to a 10 mL volumetric flask along with rinses of the 15 mL centrifuge tube and dilute it to the mark with the actonitrile:water, 50:50, v/v. This 10 mL represents a 5 g equivalent final volume of 100 mL final volume for tops and a 2.5 g equivalent final volume of 100 mL for oil. Note: A different final volume is permissible and so is dilution of extract to bring the residue concentration into the range of the analytical standards.

19. Transfer a portion of the sample extract to an autosampler vial. A 0.2µM syringe filter may be used if the samples appear to be cloudy.

9.166.2 LC/MS/MS Analysis

Samples are analyzed using UPLC/MS/MS.

20. The typical parameters for UPLC/MS/MS analysis are listed in the tables below.

Instrument: Waters ACQUITY Ultra Performance Liquid Chromatography (UPLC) equipped with Micromass Quattro Micro triple quadruple mass spectrometer, with MassLynx Software 4.1 SCN 714 (or equivalent).

Ionization: Electrospray Ionization, Positive mode (ESI +)

Solvent Delay: Start 1 at 0.2 min and end at 1.5 min
Start 2 at 9.0 min and end at 12.9 min

Scan Mode: Multiple Reaction Monitoring (MRM). Masses used for each compound are shown below

Compounds	Mass (m/z)		Cone Voltage	Collision Energy
	Parent	Daughter		
Flonicamid (IKI-220)	229.80	202.80	37.00	17.00
TFNA-AM	190.80	147.80	37.00	17.00
TFNA	191.80	147.80	37.00	17.00
TFNG	248.80	202.80	33.00	17.00

LC Column: A Luna C18 (2) 100 A, 2.00 × 150 mm, 5 µm particle (or equivalent)

Injection volume: 10.0 µL

Column Temperature: 30°C

Mobile Phase: A= 0.3% Acetic Acid in, HPLC Water.
B= Acetonitrile

Gradient Program:

Time (min)	Flow Rate (mL/min)	A (%)	B (%)	Curve
Initial	0.350	95.0	5.0	-
3.00	0.350	95.0	5.0	10
9.00	0.350	5.0	95.0	6
11.00	0.350	5.0	95.0	6
11.10	0.350	95.0	5.0	6
13.00	0.350	95.0	5.0	10

Approximate Retention Times:
~6.20 min for (Flonicamid, IKI-220)
~5.35 min for (TFNA-AM)
~5.65 min for (TFNA)
~5.72 min for (TFNG)

The retention time may vary with fluctuations in temperature, column batches, mobile phase composition, and etc.

9.166.3 Calculation and Reporting

21. Flonicamid may be quantitated against a standard curve fitted linearly with a range typically between 0.001 and 0.05 ng using a 10.0 µL injection (0.0001 and 0.005 µg/mL).

The concentrations of calibration standards may be changed, as needed but should be confirmed for sensitivity and linearity.

22. Residues are determined by comparison of unknown sample data to a standard curve generated by spreadsheet calculations. A linear fit is typically used to fit the calibration standards with an r^2 of 0.975 or better.

9.166.4 Differences from reference method:

(1) A Rotovap was used to concentrate the samples (water bath, 45 °C) instead of TurboVap (step 12). To facilitate this, the extract was decanted into 250 mL flat bottom boiling flask instead of TurboVap vessel as in steps 5, 7 and 11. Also, 0.6 mL of concentrated HCl was used instead of 0.5 mL to improve recoveries.

(2) A smaller size of Whatman No. 1 filter was used, 7 cm instead of 11 cm (Step 13) to accommodate existing laboratory equipment.

(3) In step 14, 5 mL of sample extract was used instead of 2 mL to increase amount going on the LC/MS/MS due to differences in sensitivity between instrumentation. Because of the increased amount of sample extract, three partitions (4 mL, 4 mL and 2 mL) of ethyl acetate instead of two partitions with 2 mL ethyl acetate were conducted in order to increase the efficiency of extraction.

(4) A reduced water bath temperature in Step 16 from 45 °C to 25 °C to increase % recoveries.

(5) Extraction final volume in 50% acetonitrile instead of 30% acetonitrile due to optimization of the chromatography for our system.

(6) For the UPLC mobile phase, use 0.3% acetic acid in HPLC water and acetonitrile instead of 0.2% acetic acid in acetonitrile, 0.2% acetic acid and methanol in original method. This original separation required the use of three channels. The LC pump in use only has two channels.

(7) Calibration Standards were prepared in 50% Acetonitrile for compatibility with the optimized chromatography system.

Approved by: Susan Erhardt 20 Jul 2012

Susan Erhardt Date

Laboratory Research Director

Attachment 3

Checklist for Review of Analytical Summary Reports

Checklist for Review of Analytical Summary Reports

PR #: _____ Active Ingredient/Crop: _____

	Yes	No	NA	Notes
1)Sample Preparation				
1.1 For each sample, was the full sample ground, and mixed thoroughly?				
2)Instrument Condition				
2.1 For GC/MS, are tune files or other appropriate documentation available for each run, to show that the instrument was in good working order at the time the run was made?				
If 2.1 is no, or if the analyst has concerns regarding the instrument condition, the LRD must be consulted. Was the LRD consulted?				
2.2 For other detectors, was the instrument in good working order for each run? The answer to this question will rely on the analyst's professional judgment and will include an evaluation of appropriate data obtained throughout the study, for example, the standard curve, the peak retention times, the area counts of the standards and the signal to noise ratio. Note what data was considered.				
If 2.2 is no, or if the analyst has concerns regarding the instrument condition, the LRD must be consulted. Was the LRD consulted?				

	Yes	No	NA	Notes
3)Analysis				
3.1 Is the peak of interest distinct on each chromatogram? (No shoulder peaks on the peak of interest and no interfering peaks.)				
3.2 Is the S/N ratio adequate? For example, when viewing the chromatograms for the standards through the course of the study, are there any runs where the S/N ratio has dropped significantly? A low S/N ratio is a concern. The answer to this question will rely on the analyst's professional judgment.				
If 3.2 is no, or if the analyst has concerns regarding a change in S/N ratio during a study, the LRD must be consulted. Was the LRD consulted?				
3.31 Are recoveries during method validation comparable to the recoveries in the reference method? (When the average recoveries are compared, the difference is <20%. Spot check the data, detailed calculations are not needed)				
3.32 Are concurrent recoveries during analysis comparable to those seen in method validation? (When the average recoveries are compared the difference is <15%. Spot check the data, detailed calculations are not needed)				
3.4 Did the r-squared value remain consistent during method validation and analysis of samples (Range $\leq$ 0.02)?				
If 3.4 is no, what is the range of the r-squared values? Provide an explanation.				
3.5 Are there manual integrations?				
If 3.5 is yes, were any standards manually integrated?				
If 3.5 is yes, is a reason provided in the ASR?				

	Yes	No	NA	Notes
3.61 Were duplicate injections used for concurrent fortifications?				
If 3.61 is yes, were duplicate injections within 30% of each other?				
3.62 Were duplicate injections used for unknowns?				
If 3.62 is yes, were duplicate injections within 30% of each other?				
4)Results				
4.1 Are control values non-detectable?				
If 4.1 is no, are they <20% of the highest residue value? (860.1340, p.2)				
If 4.1 is no, is this noted in the ASR and the pertinent chromatograms included?				
4.21 Are method validation recoveries and concurrent recoveries consistently >100%? (860.1340, p.2)				
4.22 For method validation and concurrent recoveries, is the CV (defined as the standard deviation/average) <20%? (860.1340, p.3)				

	Yes	No	NA	Notes
5)Analytical Summary Report				
5.1 Were any samples re-extracted and rerun?				
If 5.1 is yes, was the LRD consulted? The LRD needs to approve samples needing re-extraction due to judgment calls, for example, samples with unexpectedly high or low residue results, or a need for manual integration.)				
If 5.1 is yes, was the study director notified? (If the situation is covered in an SOP i.e. samples needing dilution, reanalysis due to a power failure or the vial location was incorrect for injection, document in the study file. The study director does not need to be notified). If answer to this question is no, please note why.				
If 5.1 is yes, is there information explaining the situation and its resolution in the ASR?				
5.2 Is there documentation in the raw data regarding any unexpected circumstances during the run?				
If 5.2 is yes, was the LRD consulted? The LRD needs to approve judgment calls, for example, samples with unexpectedly high or low residue results, or a need for manual integration.)				
If 5.2 is yes, was the study director notified? (If the situation is covered in an SOP i.e. reanalysis due to a power failure or the vial location was incorrect for injection, the study director does not need to be notified). If answer to this question is no, please note why.				
If 5.2 is yes, is there information explaining the situation and its resolution in the ASR?				
If 5.2 is yes, are chromatograms of samples with unusual or inconsistent results included in the ASR? (860.1000, p.18)				

	Yes	No	NA	Notes
5.3 Are standard curves and peak heights/areas for all standards available in the ASR for each run? (860.1000, p. 18)				
If 5.3 is no, please attach a copy of any missing standard curves with peak heights/areas, so the study director may add them as an appendix to the final report.				
5.4 Are the dates the test compounds (standard solutions) were prepared included in the ASR? (860.1500, #3, p. 37)				
If 5.4 is no, please include this information with this checklist (A copy of the standards prep form(s) is fine).				
5.5Are all residue values reported in the ASR bracketed by the standard curve?				
If 5.5 is no, was the study director contacted?				

Analytical Summary Report Reviewed by:

_____

Signature Date