

IR-4 Laboratory Guidance Document



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IR-4 Laboratory Guidance Document

Introduction

This Guidance Document is designed to provide consistency and to facilitate communication between the IR-4 Laboratory Research Directors (LRDs), Regional Directors (RDs, management), Quality Assurance Units (QA), and the IR-4 Study Directors (SDs). This document will serve as a resource for all facets of IR-4, through designating responsibilities and providing guidelines for implementation of procedures, to ensure that all studies conducted by IR-4 meet EPA Good Laboratory Practice (GLP) regulations. Once this document is approved by the IR-4 Project Management Committee, it becomes an official policy document for the conduct of studies across all 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 also 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 8.0

Original Committee members:

Daniel Kunkel, IR-4 Headquarters, Associate Director (Chair)
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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 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 or designee) 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 FRDs 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 (LRD): 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 (RLC): This person assigns laboratory-testing sites within his/her region for residue analyses conducted by the regional laboratory and private contract laboratories.

References

Good science is critical 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 CFR 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

Laboratory 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 LRD 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 require modification after this document is put in place or updated.

Standards and Solutions

Obtaining Standards: Current IR-4 policy requires that all reference standards are characterized under GLP before the completion of the study (signed by the study director), but preferably before the start of analysis. Due in part to the large number of registrants IR-4 works with, obtaining GLP standards can be difficult. It is therefore recommended that the LRD initiate discussions with the cooperating registrant as soon as possible after initiation of the study. If standards cannot be acquired in a 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. The purity value stated on the Certificate of Analysis should be used for all calculations of the standard concentration. In cases 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, then 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.*

Standard Solution Stability: If no stability information is available from the registrant or within the reference method, in-house stability data must be generated. The data generated must be valid for the solvent composition and storage conditions used, and analysis must be repeated if those conditions change. IR-4 will define a solution as stable for the interval measured if there is $\leq 10\%$ difference between a minimum of five replicate injections each of fresh and aged solutions. Labs are encouraged to reach out to other regions to see if they have any valid stability data to share.

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 Attachment 1).

Sample Receipt: Samples are generally received from a carrier such as ACDS or Fed Ex. 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 must check the condition of the samples to ensure they were kept frozen as well as 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 sample IDs. Shipping forms (Part 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 Field Data Book (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 homogeneity (for details, 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 entire 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 National Laboratory Director (NLD)¹ prior to making the change. During the course of method development, LRDs must be mindful of time and resources spent on a particular project. As part of the Backlog Response Policy (Attachment 2), a series of checkpoints have been developed to help keep projects on track and to identify potential solutions on difficult projects before falling into a backlogged status. The checkpoints are designed to keep all relevant groups (AC-AC, NLD and SD) informed of the method development progress such that ideas and experiences from the greater IR-4 community can be leveraged.

Other considerations: Approval for significant changes to the reference method must be requested from the SD, NLD 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 carrying out the same basic 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 NLD at HQ.

¹ The role of the NLD 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, NLD as well as the registrant so they can share their experiences. Chemists 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 recoveries are outside of the protocol range but consistent (standard deviation $\leq 10\%$), the Study Director may choose to accept the validation data. However, a protocol amendment should be issued to change the acceptance criteria.

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 cases where no residues are expected, fortifications should be at the lowest level of method validation (LLMV).

IR-4 laboratories agree that duplicate 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 duplicate injections provide a number of benefits such as enhanced instrument stability and better 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.

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 include samples that have no residues compared to other weathered (field) samples from treated plots, decline samples where no residues are detected, samples from untreated control plots with residues, and 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). Stability of the extracts must be proven via reanalysis after a given storage interval and comparison to the initial sample response. Always run samples with concurrent recoveries to demonstrate extract

stability.

Storage Stability

IR-4 carries out modified guideline storage stability studies as outlined in 860.1380. Our purpose is to show the samples are stable under the storage conditions used. Shortly after method validation, sufficient replicate samples covering all potential time points are fortified (at a level specified in the protocol) and three of those samples are analyzed alongside three concurrent fortifications to serve as a “Day 0” stability time point. At least 3 additional samples will be prepared and held for potential long-term analysis. Time points covering at least 90% of the storage time (from sampling date to extraction date) are typically sufficient per the protocol, though this must be confirmed with the SD prior to analysis. In some cases, the SD may be able to waive the storage stability analysis. Documentation of the waiver by the SD is required. The fortification standard solution used for stability sample preparation must be the same solution used for method validation. Currently, storage stability with analysis of one additional time point is carried out for most studies. When conducting storage stability analysis, a minimum of two concurrent fortifications will be analyzed, along with the untreated control used for storage stability fortifications. For many compounds, the registrant may have adequate storage stability data available. IR-4 will continue to work with EPA and the manufacturers to determine if a stability study is necessary. Ultimately, IR-4 will strive to conduct fewer storage stability studies where possible.

Communication of Results with SD:

Project Initiation and Response Needed to Proceed: Labs should contact the SD when R&D is starting on a given project, as well as when method validation will be conducted. The SD should use this notification as an opportunity to contact the registrant and check for any method revisions or other pertinent updates. Important modifications to the working method and levels of fortification should be made clear at this point. Upon successful method validation, and prior to treated sample analysis, the signed working method and validation data must be sent to the SD. If concurrent fortification recoveries are not within the approved protocol range (70-120%), 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 SD approval is needed or requested, the SD 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 with analysis may be sought from the NLD. However, the SD must also provide approval when he/she becomes available. LRDs and analysts should be ready to discuss possible causes for problems observed as well as proposed solutions. Practical options presented to the SD will often lead to a clearer, more efficient path forward.

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 SD as soon as possible, once all samples for the study have been analyzed. Acknowledgment of their receipt from the study director is expected.

Data Quality

The Mantle of Responsibility: IR-4 must continually strive for the highest levels of data quality and integrity, and all members of the program are responsible for the success of this mission. Chemists must ensure that the entirety of their work is reproducible and defensible in the face of an EPA audit. All data generated must therefore meet protocol, GLP, SOP, and the requirements of this document. LRDs are responsible for the careful review and approval of all project data, as well as the proper initial training of lab personnel. SDs must ensure that protocols are clear in their requirements and must remain updated on the status of all ongoing projects. All parties involved must maintain clear, honest, and open communication throughout the process. In addition, all parties must continue to educate themselves on new processes, developments, and applicable regulations.

Research and Development: Over the course of method development and refinement, care should be taken to document the different analytical approaches used, their results, and proposed next steps. Maintaining a clear record of the research process is beneficial for future projects involving similar commodities/chemistries, and such a record may be vital in the defense of method design/generated data during EPA's data review. All generated R&D data sets should be retained in at minimum a digital format, though some data sets (e.g., in-house standard stability data checks) may also be retained in a dated and initialed paper form for future use and potential archiving. This data may also need to be included in the ASR as justification of a change in acceptance criteria for recoveries.

R² values and Reproducibility: To maintain a level of consistency across all IR-4 regions, minimum data quality metrics have been set for R² values and injection reproducibility. Analytical sets that fail to meet these thresholds must be rerun. These are:

1. All generated calibration data subjected to linear regression must yield an R² value ≥ 0.985 .
2. Reproducibility between replicate injections of the same sample must be less than 20%.

Matrix-Matched (MM) Standards: Difficult matrices may impose significant enhancement or suppression effects on analytes of interest. These problems are typically solved with a more thorough sample cleanup prior to analysis. However, in cases where matrix effects cannot be overcome, or when the effects vary substantially from field to field, the use of MM standards may be beneficial. Seek LRD and SD approval prior to use of MM standards, and provide sufficient justification for their use (e.g., direct comparison of results using clean vs. MM standards). In general, a difference in concurrent recovery samples of more than $\pm 20\%$ between clean and MM standards can be used as an adequate justification for use. Verify that the control samples used for MM standard preparation are relatively clean prior to analysis. If significant ($>20\%$) differences in matrix effects are observed between fields, separate MM standards must be prepared for each field analyzed in order to account for field variability. Ensure that each MM standard is properly numbered and recorded according to GLP and SOP requirements.

Internal Standards: Internal standards should be used only if specifically required by the reference method or registrant. The SD should be contacted prior to any decision regarding use or disuse of internal standards.

Manual Integration: The use of manual integration is discouraged due to the subjective nature of individually drawn baselines. If analyte peaks are not being accurately integrated, analysts should first make every attempt to adjust the software integration parameters to fully capture peaks in a consistent manner. However, when no set of integration parameters will fully and accurately quantitate the analyte peak (e.g., missing fronts and tails, dropped baselines, inclusion of coeluting peaks or baseline noise), manual integration becomes necessary for

proper data analysis. Whenever manual integration is used, that data must be differentiated from computer-generated integrations. In software suites like Agilent MassHunter, the color of the peak changes, and an asterisk is added. Other options include handwritten notes, initialed and dated, on the manually integrated peaks, or computer-generated codes denoting manual integration. The LRD is responsible for proper manual integration training of laboratory personnel and must review all data packets to ensure that baselines (both computer-generated and manual) are properly applied.

Calculation of Parent Equivalents: Protocols may require residues to be reported in parent-equivalents. If asked to convert all residues to parent-equivalents, use the molecular weights provided by the Certificate of Analysis (CoA) or registrant to ensure accuracy. Any inconsistencies or other questions should be discussed with the SD.

Analytical Summary Report

A sample ASR is provided in Attachment 3.

Training

This document will be used as a training tool for new Laboratory Coordinators, IR-4 chemists, QA officers and Study Directors. Contract and company laboratories may also use this document as a tool to provide guidance for residue analysis. Additional online and in-person training on GLP regulations, advancements in laboratory technologies/techniques, and proper laboratory safety should be conducted when possible and made a priority for continuing education of staff across all labs.

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: Sample Processing Document

This instructional guideline has been prepared to aid in ensuring 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 ensure that samples remain representative when processed in the IR-4 laboratories.

Attachment 2: Backlog Response Policy

This policy defines when a project is considered backlogged and provides guidance on preventing future backlogs. This policy also caps method development to 3 months.

Attachment 3: 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 included. The tables 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. Please 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 visit <https://www.ir4project.org>

Attachment 4: Checklist for Review of Analytical Summary Reports

This checklist (version 1.1, 2/5/2013) is being provided as reference information 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 ensuring 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 homogeneous RAC sub-samples in a safe manner 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. 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 if unsafe working conditions exist.

Processing equipment often operates at high speeds 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. Cleaning should be performed even 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 (LN₂) 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 (LN₂ 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), $\frac{1}{4}$ 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), $\frac{1}{2}$ 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.

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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; tanager; 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; tanager; 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; tanager; 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; tanager; 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 final table

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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

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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 (<u>Phaseolus</u>), (succulent & dried), pea (<u>Pisum</u>) (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 (<u>Lupinus</u>) (includes grain lupin, sweet lupin, white lupin, and white sweet lupin); bean (<u>Phaseolus</u>) (includes field bean, kidney bean, lima bean, navy bean, pinto bean, runner bean, snap bean, tepary bean, wax bean); bean (<u>Vigna</u>) (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 (<u>Pisum</u>) (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 (<u>Phaseolus</u>) and any one succulent cultivar of edible-podded pea (<u>Pisum</u>)	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 (<u>Phaseolus</u>) (includes runner bean, snap bean, wax bean); bean (<u>Vigna</u>) (includes asparagus bean, Chinese longbean, moth bean, yardlong bean); jackbean; pea (<u>Pisum</u>) (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 (<u>Phaseolus</u>) and garden pea (<u>Pisum</u>)	Pre-processing not required	Robot Coupe, Grinder or Hobart with cryogen (LN2 or dry ice)	Bean (<u>Phaseolus</u>) (includes lima bean, green; broad bean, succulent); bean (<u>Vigna</u>) (includes blackeyed pea, cowpea, southern pea); pea (<u>Pisum</u>) (includes English pea, garden pea, green pea); pigeon pea
6C. Dried shelled pea and bean (except soybean) subgroup	Any one dried cultivar of bean (<u>Phaseolus</u>) and any one dried cultivar of pea (<u>Pisum</u>)	Pre-processing not required	Grinder type processor, coffee grinder or Robot Coupe with cryogen (LN2 or dry ice)	Dried cultivars of bean (<u>Lupinus</u>); bean (<u>Phaseolus</u>) (includes field bean, kidney bean, lima bean (dry), navy bean, pinto bean, tepary bean); bean (<u>Vigna</u>) (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 (<u>Pisum</u>) (includes field pea); pigeon pea
7. FOLIAGE OF LEGUME VEGETABLES	Any cultivar of bean (<u>Phaseolus</u>), field pea (<u>Pisum</u>) 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 (<u>Phaseolus</u>) and field pea (<u>Pisum</u>)	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.

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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 (<i>Physalis</i> 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); <i>Momordica</i> 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; <i>Momordica</i> 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

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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

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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 (<u>Trifolium</u>)	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 (<u>Trifolium</u> , <u>Mellilotus</u>); 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

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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 <i>Passiflora spp.</i> 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

SECTION 102

Pesticide Analytical Manual Vol. I

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)

Pesticide Analytical Manual Vol. I

SECTION 102

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.

Transmittal No. 96-1 (9/96)
Form FDA 2905a (6/92)

102-3

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

Backlog Response Policy

1. Definition of a backlog

Analytical work should be done within 12 months after the last batch of field samples are received by a lab. A “backlog” exists when the sample analysis and ASR are not completed within 12 months.

Projects will not be considered backlogged if the following situations have occurred:

- HQ management reprioritization of study timeline
- Unable to receive standards from registrant
- Government or University shutdown impacting IR-4 and/or ARS operations

2. Strategy for preventing future backlogs

- Better planning
 - As much as possible, assign projects based on previous experience and expertise, recognizing that workloads need to be balanced and new actives will be analyzed.
 - Each lab should plan for the anticipated projects in advance, for example, to do method development before samples from last trials arrive.
 - Management will assure that personnel, resources, facilities, equipment...are available as scheduled.
 - Management to evaluate status of labs during Spring PMC meeting to determine if all labs have sufficient resources such that IR-4 laboratories may accept outside contract work.
- Better communication and transparency
 - Management to foster a culture where timelines are met, and issues are raised and addressed to prevent backlogs.
 - Cap the time for method development to **3 months**
 - i. Suggested Method Development Timeline
 1. After 1st month: LRD contacts AC-AC to engage other chemists for ideas. Concurrently, LRD reaches out to registrant (chemist and IR-4 representative) for assistance. The Study Director (SD) and National Laboratory Director (NLD) are kept in the loop either as part of AC-AC discussions or direct communications.
 2. After 2nd month: LRD re-engages with AC-AC to report back results for various attempts and discusses possible next steps. LRD shares efforts with IR-4 and regional management (Regional Director (RD), NLD and SD).
 3. After 3rd month: LRD meets with RD, NLD and SD to discuss options to facilitate the timely completion of the project.
 - a. Transfer project to different IR-4 lab
 - b. Transfer project to contract lab
 4. For the purposes of reporting, color levels will be used.
 - a. After 1st month: Yellow
 - b. After 2nd month: Orange
 - c. After 3rd month: Red

3. Consequences for backlog

Once a project or projects become backlogged per the definition above, an email note will be sent by the National Lab Director to the IR-4 Executive Director, the Lab Director and the Regional Director. This note will provide notification that there is a backlog and request a formal response containing an explanation of the current status, and a proposal for corrective actions to meet the analytical timeline(s). The email response will be generated by the Lab director, approved by the Regional Director, and should be sent to the IR-4 Executive Director and National Lab Director within two weeks. HQ will decide if a meeting between the Lab Director, Regional Director, IR-4 Executive Director (and others as requested) is needed to approve the proposal or to discuss alternatives to make corrections to meet study timelines. If a meeting is not deemed necessary, approval for the plan will be provided via email. Once the backlog situation has been successfully addressed, a follow-up email will be sent from the National Lab Director to the Lab Director, with copies to the Regional Director and the IR-4 Executive Director.

Attachment 3

Sample Analytical Summary Report

ANALYTICAL SUMMARY REPORT
Flonicamid: Magnitude of the Residue on Onion

PR# 08550

Author(s)

Alexander McFall

Laboratory Research Director

Matt Hengel

Testing Laboratory

IR-4 Western Region Laboratory
Department of Environmental Toxicology
University of California, Davis
4218 Meyer Hall
Davis, CA 95616

Laboratory ID# 08550.20-CAR10

Study Director

Cristina Marchesan Marconi

Sponsor

Interregional Research Project #4 (IR-4)
IR-4 Project Headquarters
1730 Varsity Drive, Venture IV, Suite 110
Raleigh, NC 27606

Field ID Numbers

08550.20-CA*19	08550.20-CA*20
08550.20-WA*403	08550.20-TX380
08550.20-CA16	08550.20-CA17
08550.20-CA18	08550.20-ID181
08550.20-WA404	08550.20-OR405
08550.20-CO461	

Study Timetable

Study Initiation Date: 02/20/20
Experimental Termination Date: 07/20/21

Report Date

GLP STANDARDS COMPLIANCE STATEMENT

PR#: 08550

Lab ID#: 08550.20-CAR10

The study reported herein for residues of flonicamid and its metabolites on onion was conducted and reported in compliance with GLP Title 40 CFR, part 160 of the Code of Federal Regulations of the United States of America.

 11/23/2021

Signature/Date

Matt Hengel

Laboratory Research Director

*IR-4 Western Region Laboratory**Department of Environmental Toxicology**University of California, Davis**4218 Meyer Hall**Davis, CA 95616**Tel. No.: (530) 752-2402* 11/23/2021

Signature/Date

Alexander McFall

Analyst

*IR-4 Western Region Laboratory**Department of Environmental Toxicology**University of California, Davis**4218 Meyer Hall**Davis, CA 95616**Tel. No.: (530) 752-2402*

IR-4 NATIONAL PESTICIDE CLEARANCE RESEARCH PROGRAM
RESIDUE DATA REPORTING FORM (Part 2)
RESIDUE STUDY QUALITY ASSURANCE REPORT SHEET

INSTRUCTION: For the residue study captioned below, the Quality Assurance Unit (QAU) must enter the type of inspections or audits performed, the date each was done, and the date the findings were reported to Management and the Study Director. The completed form becomes part of the study record. QAU records are to be maintained and indexed as per 40 CFR Part 160.

PR#: 08550

Lab ID #: 08550.20-CAR10

Commodity: Onion

Analysis for residues of: Flonicamid

Laboratory Research Director: Matt Hengel

INSPECTION/AUDIT**

<u>TYPE</u>	<u>DATE</u>	<u>BY</u>	<u>DATE REPORTED*</u>
1. <u>CPI-L</u>	<u>5/19/21</u>	<u>M. Beran</u>	<u>5/20/21</u>
2. <u>RDA -ASR</u>	<u>11/3-5, 8-9/21</u>	<u>M. Beran</u>	<u>11/10/21</u>

*Date findings reported to management and study director

**CPI= Critical Phase Inspection; RDA= Raw Data Audit; ASR= Analytical Summary Report.

Signature: _____ Date: 11/23/21

Name: (Print): Martin Beran
Quality Assurance Coordinator

Phone: (530) 754-8525

Address: Dept of Environmental Toxicology, University of California. Davis, CA 95616-8588

LABORATORY PERSONNEL

Name	Designation
Matt Hengel	Laboratory Research Director
Bronson Hung	Analyst, Sample Control Officer
Paul Kuzmicky	Analyst
Alexander McFall	Analyst
Tey Montalvo	Analyst
Riza Punongbayan	Analyst, Report Preparation
Salvador Chava Torres	Asst. Sample Control Officer
Fabiola G. Zuno	Analyst

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LOCATION OF RAW DATA

Original raw data, a certified copy of the signed protocol, amendments, correspondence logs and all relevant information for the study titled: “Flonicamid: Magnitude of the Residue on Onion, PR# 08550” along with a certified copy of the signed analytical summary report will be maintained in the archives of the testing laboratory. The original copy of the analytical summary report will be forwarded to the sponsor.

Portions of the field samples will be retained at the testing laboratory in a freezer generally -20°C for at least 12 months after submission of the laboratory report. The long term storage stability samples will be stored for at least 5 years at generally -20°C . The study director will be consulted before the field samples or the storage stability samples are discarded.

Laboratory Research Director: Matt Hengel

Testing Laboratory: IR-4 Western Region Laboratory
Department of Environmental Toxicology
University of California, Davis
4218 Meyer Hall
Davis, CA 95616
Tel. No.: (530) 752-2402

**IR-4 NATIONAL PESTICIDE CLEARANCE RESEARCH PROGRAM
ANALYTICAL SUMMARY REPORT PR#08550:
FLONICAMID/ONION**

I. Objective/Introduction

At the request of IR-4 Headquarters, the Western Region Laboratory at the University of California, Davis (UCD) has assayed onion for residues of Flonicamid (CAS# 158062-67-0) and its metabolites to provide data to support the establishment of a pesticide tolerance. The method used in this study was derived from "Analytical Methodology for IKI-220 (F1785) and its Major Metabolites in/on Peach, Potato Tuber, and Wheat Straw", Audrey W. Chen, Ph.D., Report Number P-3561M, FMC Corporation Agricultural Products Group, Princeton, NJ. August 28, 2002. Steps where the UCD working method significantly diverges from the method referenced in the protocol are noted in Section V. Modifications. The study followed IR-4 National Pesticide Clearance Laboratory Phase Protocol PR# 08550 as amended. The validated method sensitivity is 0.01 ppm flonicamid and its metabolites TFNG-AM, TFNA, TFNG.

II. Sample Inventory/History

Upon arrival at the laboratory, samples were opened, inspected, and checked against the enclosed shipping form. Unique laboratory sample ID numbers were assigned as listed in Table II.1. Samples were stored frozen. Samples from field trial CA*20 were received with untrimmed roots. At the request of the Study Director, the roots were removed while frozen with a clean knife before processing.

Raw Agricultural Commodity (RAC) samples were processed with dry ice in either a floor model Hobart food chopper or a Robot Coupe food chopper. After the entire sample was chopped, a portion was placed in labeled glass pint jars and surplus was put back into the sample bag. Samples chopped with the Hobart food chopper were sifted through a #6 wire mesh screen into glass pint jars and surplus was put back into the sample bag. Glass jars and sample bags were returned to the freezer and stored frozen (generally -20°C).

Table II.1: Sample Inventory

Field Trial	Crop Fraction	Field Sample ID	Lab Sample ID	Sampling Date	Lab Receipt Date	Processing Date
CA*19	Bulbs	DBA	28989	12/03/20	12/08/20	01/04/21
		DBB	28990	12/03/20	12/08/20	01/04/21
		DBC	28991	12/03/20	12/08/20	01/04/21
		DBD	28992	12/03/20	12/08/20	01/04/21
CA*20	Plants	GA	28849	08/31/20	11/10/20	12/21/20
		GB	28850	08/31/20	11/10/20	12/21/20
		GC	28851	08/31/20	11/10/20	12/21/20
		GD	28852	08/31/20	11/10/20	12/21/20
WA*403	Bulbs	DBA	28861	10/02/20	11/10/20	01/04/21
		DBB	28862	10/02/20	11/10/20	01/04/21
		DBC	28863	10/02/20	11/10/20	01/05/21
		DBD	28864	10/02/20	11/10/20	01/05/21
TX380	Bulbs	DBA	29148	06/07/21	07/14/21	07/19/21
		DBB	29149	06/07/21	07/14/21	07/19/21
		DBC	29150	06/07/21	07/14/21	07/19/21
		DBD	29151	06/07/21	07/14/21	07/19/21
CA16	Bulbs	DBA	28241	06/30/20	07/07/20	07/16/20
		DBB	28242	06/30/20	07/07/20	07/16/20
		DBC	28243	06/30/20	07/07/20	07/16/20
		DBD	28244	06/30/20	07/07/20	07/16/20
CA17	Bulbs	DBA	28304	08/07/20	08/12/20	01/04/21
		DBB	28305	08/07/20	08/12/20	01/04/21
		DBC	28306	08/07/20	08/12/20	01/05/21
		DBD	28307	08/07/20	08/12/20	01/05/21
CA18	Plants	GA	28225	05/11/20	06/04/20	07/27/20
		GB	28226	05/11/20	06/04/20	07/27/20
		GC	28227	05/11/20	06/04/20	07/27/20
		GD	28228	05/11/20	06/04/20	07/27/20
ID181	Bulbs	DBA	28857	09/14/20	11/10/20	01/05/21
		DBB	28858	09/14/20	11/10/20	01/05/21
		DBC	28859	09/14/20	11/10/20	01/05/21
		DBD	28860	09/14/20	11/10/20	01/05/21
WA404	Bulbs	DBA	28654	09/08/20	09/24/20	01/05/21
		DBB	28655	09/08/20	09/24/20	01/05/21
		DBC	28656	09/08/20	09/24/20	01/06/21
		DBD	28657	09/08/20	09/24/20	01/06/21
OR405	Plants	GA	28265	06/11/20	07/13/20	08/03/20
		GB	28266	06/11/20	07/13/20	08/03/20
		GC	28267	06/11/20	07/13/20	08/03/20
		GD	28268	06/11/20	07/13/20	08/03/20
CO461	Bulbs	DBA	28853	08/23/20	11/10/20	12/08/20
		DBB	28854	08/23/20	11/10/20	12/08/20
		DBC	28855	08/23/20	11/10/20	12/08/20
		DBD	28856	08/23/20	11/10/20	12/08/20

III. Preparation of Storage Stability Samples

Storage stability samples were prepared by the laboratory. The analysis of these samples was not required because the samples were stored for less than the 23 month period covered by existing storage stability data.

Table III.1: Preparation of Storage Stability Samples

Field Trial	Field Sample ID	Crop Fraction	No. Prepared	Sample Size (g)	Std #	Conc. $\mu\text{g/mL}$	μL Added	μg Added	Fort. Level ppm	Date Fortified
CA16	DBA	Bulbs	3	2.50	687-1M3	1.0	250	0.25	0.1	07/17/20
CA16	DBB	Bulbs	3	2.50	687-1M3	1.0	250	0.25	0.1	07/17/20

Note: All samples were weighed into 50 mL polypropylene tubes and stored in the dark at generally -20°C .

IV. Standard Preparation

Stock Solutions:

Prepare a primary stock solution for each compound: flonicamid, TFNA-AM, TFNA, and TFNG. For all compounds, 25 mg (corrected for purity) of analytical standard is accurately weighed and transferred to a 25 mL volumetric flask. The standards are brought to volume with acetonitrile. The resulting solution concentrations are 1.0 mg/mL. These solutions are stored in amber glass bottles in the freezer (ca. -20°C) when not in use. When stored in the freezer in amber bottles, these stock solutions are stable for 1 year, per the reference method.

Fortification Standards:

Typically, the following analyte concentrations are prepared. All solutions are stored in amber bottles in the freezer when not in use and are stable for 6 months, per the reference method.

100 $\mu\text{g/mL}$ Mix: Transfer 2.50 mL of each 1.0 mg/mL standard into a 25 mL volumetric flask. Bring to volume with acetonitrile. Mix well.

10 $\mu\text{g/mL}$ Mix: Transfer 2.50 mL of the 100 $\mu\text{g/mL}$ mixed standard solution into a 25 mL volumetric flask. Bring to volume with acetonitrile. Mix well.

1.0 $\mu\text{g/mL}$ Mix: Transfer 2.50 mL of the 10 $\mu\text{g/mL}$ mixed standard solution into a 25 mL volumetric flask. Bring to volume with acetonitrile. Mix well.

0.10 $\mu\text{g/mL}$ Mix: Transfer 2.50 mL of the 1.0 $\mu\text{g/mL}$ mixed standard solution into a 25 mL volumetric flask. Bring to volume with acetonitrile. Mix well.

0.010 µg/mL Mix: Transfer 2.50 mL of the 0.10 µg/mL mixed standard solution into a 25 mL volumetric flask. Bring to volume with acetonitrile. Mix well.

LC-MS/MS (Calibration) Standard Solutions:

All calibration standard solutions are stored in amber bottles in the freezer when not in use. Solutions are prepared in 5:95 acetonitrile:water, and are considered stable in the freezer for 14 days. Typically, the following concentrations of calibration solutions are prepared:

1.0 pg/µL: Transfer 2.50 mL of 0.010 µg/mL mixed standard solution into a 25 mL volumetric flask. Bring to volume with 5:95 acetonitrile:water. Mix well.

0.50 pg/µL: Transfer 1.25 mL of 0.010 µg/mL mixed standard solution into a 25 mL volumetric flask. Bring to volume with 5:95 acetonitrile:water. Mix well.

0.25 pg/µL: Transfer 625 µL of 0.010 µg/mL mixed standard solution into a 25 mL volumetric flask. Bring to volume with 5:95 acetonitrile:water. Mix well.

0.10 pg/µL: Transfer 250 µL of 0.010 µg/mL mixed standard solution into a 25 mL volumetric flask. Bring to volume with 5:95 acetonitrile:water. Mix well.

0.050 pg/µL: Transfer 125 µL of 0.010 µg/mL mixed standard solution into a 25 mL volumetric flask. Bring to volume with 5:95 acetonitrile:water. Mix well.

V. Analytical Procedure

Principle of Method

Residues of flonicamid (aka IKI-220), 4-trifluoromethylnicotinamide (TFNA-AM), 4-trifluoromethylnicotinic acid (TFNA), and *N*-(4-trifluoromethylnicotinoyl) glycine (TFNG) are extracted from samples via sequential shake extractions with a 50/50 water/acetonitrile mixture. The acetonitrile is removed via evaporation, the extract is acidified, the volume is adjusted, and then the samples are cleaned up using C-18 solid phase extraction (SPE). The extracts are then evaporated once more to remove ACN, diluted as necessary, and then analyzed using liquid chromatography coupled with positive-ion electrospray mass spectrometry (LC-MS/MS). The lowest level of method validation (LLMV) is 0.01 ppm and the limit of detection is ten percent below the lowest calibration standard.

Materials and Reagents

Acetonitrile, Optima LC-MS grade, Fisher

Methanol, Optima LC-MS grade, Fisher

Water, Type I (18.2 MΩcm, Milli-Q)

Formic acid LC-MS grade, Thermo Scientific

Hydrochloric acid, GR ACS grade, EMD

Mega Bond Elut C18 SPE Cartridges, 1g/6mL, Agilent

50 mL polypropylene tubes, Corning

Flint glass test tubes, 16 x 100 mm, Fisher

50 mL graduated cylinders, Corning

LC column: InfinityLab Poroshell 120 EC-18, 2.1 x 100 mm, 2.7 μm, Agilent

Guard column: Poroshell 120 EC-C18, 2.1 x 5 mm, 2.7 μm, Agilent

Method Procedure

1. Extraction

- 1.1. Weigh out 2.5 g of sample into a 50 mL polypropylene tube, fortify at this point for concurrent recovery samples (fortified as a mixture).
- 1.2. Add 40 mL of 50:50 ACN:Water (v:v).
- 1.3. Using a platform shaker, shake the samples for 30 minutes at 200 RPM.
- 1.4. Centrifuge the samples for 5 mins at 4000 RPM, and then decant the extract through a funnel containing Whatman #1 filter paper into a clean TurboVap tube (Note 1).
- 1.5. Add 40 mL of 50:50 ACN:Water to the original sample pellet, and shake using a platform shaker for 30 minutes at 200 RPM.
- 1.6. Centrifuge the samples for 5 mins at 4000 RPM, and pass through the same filter to combine with the step 1.4 extract. Rinse the filter paper with 5 mL of 50:50 ACN:Water.

2. Evaporation

- 2.1. Evaporate the samples using a TurboVap (50°C, 24 PSI) until ~ 30 mL remain (to ensure no ACN is present, approximately 60 minutes, Note 2).
- 2.2. Add 0.50 mL of concentrated HCl to each sample and transfer to a 50 mL graduated cylinder.
- 2.3. Rinse the TurboVap tube twice with 5 mL of Milli-Q water and add to the graduated cylinder. Adjust the volume to 50 mL with additional Milli-Q water and transfer to a 50-mL polypropylene centrifuge tube for storage.

3. C-18 SPE Cleanup

- 3.1. Condition each C-18 cartridge (1g/6mL) with 1 CV of methanol followed by 1 CV of 0.25N HCl in water.
- 3.2. Aliquot 2 mL of sample extract onto the cartridge and load (1-2 drops/sec), discarding the eluate. Briefly use full vacuum to pull all possible solution through the packing.
- 3.3. Elute the analytes (1-2 drops/sec) with 6 mL of 20:80 ACN:Water (v:v) into a glass test tube.
- 3.4. Using a TurboVap LV (45°C, ~12 PSI), evaporate the samples until 4-5 mL remain (approximately 10 minutes, Note 2).
- 3.5. Transfer to a graduated centrifuge tube and bring up to 10 mL with 0.25N HCl in water (Note 3). Submit to LC-MS/MS for analysis.

Modifications

1. Filtered samples prior to TurboVap, not after. Onion samples often have loose bits of skin/crop material that float to the surface and should be removed once extraction is completed.
2. Reduced sample aliquot size used in SPE from 10 mL to 2 mL. Greater instrument sensitivity allows for less sample extract to be used.
3. Removed liquid-liquid partition step after SPE cleanup. Testing showed extract was sufficiently clean after C18 cleanup.

Notes

1. Some onion crops possess very fine particle sizes that are more prone to clogging filter paper. To assist in filtration, a Buchner funnel with moderate vacuum may be substituted for gravity filtration.
2. May adjust evaporation times to ensure samples are evaporated to the proper remaining volume.
3. Typically, fortification samples at LLMV (0.01 ppm) and unknowns will be brought up to a 10 mL final volume using 0.25N HCl in water to approximate a final composition of 5:95 ACN:Water. Further dilution will use 5:95 ACN:Water to maintain approximate composition. Unknown and concurrent fortification sample volumes and dilutions may be adjusted as necessary.

VI. Quantitation:

Calculations:

Prepare a five-point standard curve by injecting constant volumes of standard solutions. Use constant volume injections for sample extracts as well. Sample responses not within 10% of the standard curve require volume adjustment and re-injection. Samples will not be adjusted below the equivalent volume of the LLMV. Inject a calibration standard after every four sample injections. Calculations for instrumental analysis are conducted by Agilent "MassHunter" software to create a standard curve based on linear regression. The regression functions are used to calculate a best fit line (from a set of standard concentrations in pg/ μ L versus peak response) and to determine sample analyte concentrations.

The equation used for the least squares fit is: $y = mx + b$, where y = peak response, x = pg/ μ L found for peak of interest, m = slope and b = y -intercept. Concurrent recovery samples are control samples fortified with known amounts of analyte prior to extraction.

Percent recovery (if calculated by measuring the peak area) is calculated as shown below:

$$\frac{\text{pg}/\mu\text{L determined} \times \mu\text{L injected}}{\text{mg crop injected} \times 1000 \text{ conv. factor}} = \text{actual ppm analyte}$$

$$\frac{\text{actual ppm analyte}}{\text{expected ppm analyte}} \times 100 = \% \text{ Recovery}$$

Example Calculation:

Sample: 28857V0.01R9 (Flonicamid, 05042021a023.d)

$$\frac{0.0909 \text{ pg}/\mu\text{L} \times 10 \text{ injected}}{0.100 \text{ mg} \times 1000 \text{ conv. factor}} = 0.00909 \text{ ppm Flonicamid}$$

$$\frac{0.00909 \text{ actual ppm flonicamid}}{0.0100 \text{ expected ppm flonicamid}} \times 100 = 91\% \text{ Recovery}$$

For expressing in equivalents;

Metabolite residues are expressed as parent equivalents and are calculated by using the formula:

$$\text{Average ppm} * (\text{conversion factor}) = \text{ppm found}$$

The conversion factor was calculated using the formula:

$$\text{Flonicamid MW} \div \text{metabolite MW} = \text{conversion factor}$$

Compound	Molecular Weight (MW)	Conversion Factor
Flonicamid	229.17	--
TFNA-AM	190.12	1.205
TFNA	191.11	1.199
TFNG	248.16	0.9235

Instrument Parameters:

Instrumentation: "Speeder" Agilent 6460 LC-MS/MS
 Autosampler: Agilent 1200 Series
 Pumps: Agilent 1200 Series
 Data System: Agilent MassHunter software (b.06 running on Windows 7). Data exported to Microsoft Excel.
 Mobile Phase: A = 0.1% formic acid in water
 B = 0.1% formic acid in methanol
 Column temperature: 40 °C
 Injection size: 10 µL

Gradient Program:

Total Time (min)	Flow Rate (mL/min)	Solvent A (%)	Solvent B (%)
0.00	0.400	98.0	2.0
0.50	0.400	98.0	2.0
2.50	0.400	90.0	10.0
3.50	0.400	90.0	10.0
5.50	0.400	70.0	30.0
6.50	0.400	70.0	30.0
8.50	0.400	2.0	98.0
10.50	0.400	2.0	98.0
10.60	0.400	98.0	2.0
18.00	0.400	98.0	2.0

LC-MS/MS Interface: ESI + Agilent Jet Stream
 Ionization Mode: Positive
 Gas Temperature: 300 °C
 Gas Flow: 10 L/min (N₂)
 Nebulizer: 45 psi (N₂)
 Capillary: 3000 V (+)
 Sheath Gas Temperature: 300 °C
 Sheath Gas Flow: 12 (L/min)
 Nozzle Voltage: 500 V

Analyte Parameters:

Compound	Transition	MS1 (m/z)	MS2 (m/z)	Dwell	Frag (V)	CE (V)	~ Rt (min)
TFNA	Quantifier	192.1	148.0	200	45	20	6.0
TFNA	Qualifier	192.1	98.1	200	45	32	6.0
TFNA-AM	Quantifier	191.1	148.0	200	45	20	5.3
TFNA-AM	Qualifier	191.1	98.1	200	45	32	5.3
TFNG	Quantifier	249.2	203.0	200	45	16	7.2
TFNG	Qualifier	249.2	148.0	200	45	28	7.2
Flonicamid	Quantifier	230.2	98.1	200	45	44	7.7
Flonicamid	Qualifier	230.2	148.0	200	45	28	7.7

Diverter Valve Program:

Total Time	Valve Position	Analyte(s)
0.0	Waste	N/A
4.0	MS	TFNA, TFNA-AM
6.6	MS	TFNG, Flonicamid
9.0	Waste	N/A

Needle Wash Program (flushing solution: 0.1% formic acid in 45:45:10 MeOH:ACN:Water):

Step	Action
Wash	Wash needle in flushport for 10s
Draw	Draw default volume from sample with default speed using default offset
Wash	Wash needle in flushport for 10s
Inject	Inject

VII. Results and Discussion:

The fortified sample results are reported below as ppm flonicamid, TFNA-AM, TFNA, TFNG and total flonicamid. For field samples, flonicamid residues are reported as such and metabolite residues are reported as parent equivalents. Summary of results are listed below:

Table VII.1.1: Summary of Recoveries, Flonicamid

Crop Fraction	Spike Level ppm	Lab Sample ID	Type of Recovery ¹	Flonicamid Found ppm	Average ppm	Recoveries (%)	Average Recovery (%) ²
Bulbs	0.01	28857V0.01R7	MV	0.00948	0.00939	95	94±6
		28857V0.01R8	MV	0.00864		86	
		28857V0.01R9	MV	0.00958		96	
		28853C0.01R1	CR	0.00917		92	
		28241C0.01R2	CR	0.00907		91	
		28861C0.01R3	CR	0.00937		94	
		29148C0.01R4	CR	0.0104		104	
	0.1	28857V0.1R4	MV	0.0945	0.0943	94	94±1
		28857V0.1R5	MV	0.0936		94	
		28857V0.1R6	MV	0.0948		95	
	1.0	28857V1.0R4	MV	0.965	0.962	97	96±6
		28857V1.0R5	MV	0.961		96	
		28857V1.0R6	MV	0.969		97	
		28853C1.0R1	CR	0.987		99	
		28241C1.0R2	CR	0.851		85	
		28861C1.0R3	CR	1.05		105	
		29148C1.0R4	CR	0.951		95	
Plants	0.01	28849V0.01R1	MV	0.00960	0.00908	96	91±6
		28849V0.01R2	MV	0.00924		92	
		28849V0.01R3	MV	0.00909		91	
		28265C0.01R1	CR	0.00913		91	
		28265C0.01R2	CR	0.00797		80	
		28225C0.01R3	CR	0.00945		94	
	0.1	28849V0.1R1	MV	0.0890	0.0907	89	91±2
		28849V0.1R2	MV	0.0894		89	
		28849V0.1R3	MV	0.0925		93	
		28225C0.10R1	CR	0.0920		92	
	1.0	28849V1.0R1	MV	0.990	0.987	99	99±1
		28849V1.0R2	MV	0.981		98	
		28849V1.0R3	MV	0.989		99	
	2.0	28849V2.0R1	MVE	2.32	2.20	116	110±7
		28849V2.0R2	MVE	2.23		111	
		28849V2.0R3	MVE	2.05		103	

¹MV=Method Validation, CR=Concurrent Recovery, MVE=Method Validation Extension

²Average % recovery and standard deviation calculations are based on rounded % recoveries.

Table VII.1.2: Summary of Recoveries, TFNA-AM

Crop Fraction	Spike Level ppm	Lab Sample ID	Type of Recovery ¹	TFNA-AM Found ppm	Average ppm	Recoveries (%)	Average Recovery (%) ²
Bulbs	0.01	28857V0.01R7	MV	0.0104	0.00958	104	96±7
		28857V0.01R8	MV	0.00948		95	
		28857V0.01R9	MV	0.00940		94	
		28853C0.01R1	CR	0.00937		94	
		28241C0.01R2	CR	0.00865		86	
		28861C0.01R3	CR	0.00916		92	
		29148C0.01R4	CR	0.0106		106	
	0.1	28857V0.1R4	MV	0.0901	0.0904	90	90±1
		28857V0.1R5	MV	0.0906		91	
		28857V0.1R6	MV	0.0904		90	
	1.0	28857V1.0R4	MV	0.935	0.952	94	95±7
		28857V1.0R5	MV	0.944		94	
		28857V1.0R6	MV	0.931		93	
		28853C1.0R1	CR	0.934		93	
		28241C1.0R2	CR	0.829		83	
		28861C1.0R3	CR	1.05		105	
		29148C1.0R4	CR	1.04		104	
Plants	0.01	28849V0.01R1	MV	0.0106	0.00950	106	95±6
		28849V0.01R2	MV	0.00965		96	
		28849V0.01R3	MV	0.00905		90	
		28265C0.01R1	CR	0.00919		92	
		28265C0.01R2	CR	0.00881		88	
		28225C0.01R3	CR	0.00969		97	
	0.1	28849V0.1R1	MV	0.0838	0.0883	84	88±4
		28849V0.1R2	MV	0.0870		87	
		28849V0.1R3	MV	0.0892		89	
		28225C0.10R1	CR	0.0931		93	
	1.0	28849V1.0R1	MV	0.934	0.948	93	95±2
		28849V1.0R2	MV	0.958		96	
		28849V1.0R3	MV	0.953		95	
	2.0	28849V2.0R1	MVE	2.17	2.10	108	105±5
		28849V2.0R2	MVE	1.98		99	
		28849V2.0R3	MVE	2.16		108	

¹MV=Method Validation, CR=Concurrent Recovery, MVE=Method Validation Extension²Average % recovery and standard deviation calculations are based on rounded % recoveries.

Table VII.1.3: Summary of Recoveries, TFNA

Crop Fraction	Spike Level ppm	Lab Sample ID	Type of Recovery ¹	TFNA Found ppm	Average ppm	Recoveries (%)	Average Recovery (%) ²
Bulbs	0.01	28857V0.01R7	MV	0.00969	0.00949	97	95±6
		28857V0.01R8	MV	0.00906		91	
		28857V0.01R9	MV	0.00918		92	
		28853C0.01R1	CR	0.00966		97	
		28241C0.01R2	CR	0.00985		98	
		28861C0.01R3	CR	0.00860		86	
		29148C0.01R4	CR	0.01040		104	
	0.1	28857V0.1R4	MV	0.0906	0.0924	91	92±2
		28857V0.1R5	MV	0.0943		94	
		28857V0.1R6	MV	0.0924		92	
	1.0	28857V1.0R4	MV	0.929	0.978	93	98±8
		28857V1.0R5	MV	0.922		92	
		28857V1.0R6	MV	0.949		95	
		28853C1.0R1	CR	0.985		99	
		28241C1.0R2	CR	0.908		91	
		28861C1.0R3	CR	1.03		103	
		29148C1.0R4	CR	1.12		112	
Plants	0.01	28849V0.01R1	MV	0.0104	0.00940	104	94±6
		28849V0.01R2	MV	0.00891		89	
		28849V0.01R3	MV	0.00971		97	
		28265C0.01R1	CR	0.00874		87	
		28265C0.01R2	CR	0.00915		91	
		28225C0.01R3	CR	0.00949		95	
	0.1	28849V0.1R1	MV	0.0900	0.0894	90	89±2
		28849V0.1R2	MV	0.0861		86	
		28849V0.1R3	MV	0.0913		91	
		28225C0.10R1	CR	0.0902		90	
	1.0	28849V1.0R1	MV	0.948	0.959	95	96±1
		28849V1.0R2	MV	0.971		97	
		28849V1.0R3	MV	0.959		96	
	2.0	28849V2.0R1	MVE	2.13	2.10	107	105±2
		28849V2.0R2	MVE	2.05		103	
		28849V2.0R3	MVE	2.13		106	

¹MV=Method Validation, CR=Concurrent Recovery, MVE=Method Validation Extension²Average % recovery and standard deviation calculations are based on rounded % recoveries.

Table VII.1.4: Summary of Recoveries, TFNG

Crop Fraction	Spike Level ppm	Lab Sample ID	Type of Recovery ¹	TFNG Found ppm	Average ppm	Recoveries (%)	Average Recovery (%) ²
Bulbs	0.01	28857V0.01R7	MV	0.0103	0.00995	103	100±3
		28857V0.01R8	MV	0.0100		100	
		28857V0.01R9	MV	0.00956		96	
		28853C0.01R1	CR	0.00951		95	
		28241C0.01R2	CR	0.0101		101	
		28861C0.01R3	CR	0.00991		99	
		29148C0.01R4	CR	0.0103		103	
	0.1	28857V0.1R4	MV	0.102	0.0997	102	100±2
		28857V0.1R5	MV	0.0984		98	
		28857V0.1R6	MV	0.0986		99	
	1.0	28857V1.0R4	MV	1.06	1.09	106	109±6
		28857V1.0R5	MV	1.10		110	
		28857V1.0R6	MV	1.09		109	
		28853C1.0R1	CR	1.05		105	
		28241C1.0R2	CR	0.995		100	
		28861C1.0R3	CR	1.18		118	
		29148C1.0R4	CR	1.13		113	
Plants	0.01	28849V0.01R1	MV	0.00922	0.00929	92	93±5
		28849V0.01R2	MV	0.00917		92	
		28849V0.01R3	MV	0.00851		85	
		28265C0.01R1	CR	0.0100		100	
		28265C0.01R2	CR	0.00904		90	
		28225C0.01R3	CR	0.00982		98	
	0.1	28849V0.1R1	MV	0.0982	0.100	98	100±3
		28849V0.1R2	MV	0.0983		98	
		28849V0.1R3	MV	0.102		102	
		28225C0.10R1	CR	0.103		103	
	1.0	28849V1.0R1	MV	1.04	1.05	104	105±2
		28849V1.0R2	MV	1.05		105	
		28849V1.0R3	MV	1.07		107	
	2.0	28849V2.0R1	MVE	2.35	2.33	117	116±2
		28849V2.0R2	MVE	2.27		114	
		28849V2.0R3	MVE	2.37		118	

¹MV=Method Validation, CR=Concurrent Recovery, MVE=Method Validation Extension²Average % recovery and standard deviation calculations are based on rounded % recoveries.

Table VII.2: Residue Data Results

Trial ID	Crop Fraction	Field Sample ID	Lab Sample ID	Sampling Date	Extraction Date	Analysis Date	Storage Interval ¹ (Days)	Residue Results (ppm)				
								Flonicamid	TFNA-AM ppmfound/ parent equivalent ²	TFNA ppm found/ parent equivalent ²	TFNG ppmfound/ parent equivalent ²	Total Flonicamid ³
CA*19	Bulbs	DBA	28989	12/03/20	05/06/21	05/06/21		<0.01	<0.01	<0.01	<0.01	
		DBB	28990	12/03/20	N/A	NA		---	---	---	---	
		DBC	28991	12/03/20	05/06/21	05/06/21	154	0.011	<0.01	0.013/0.016	0.01/0.0092	0.046
		DBD	28992	12/03/20	05/06/21	05/06/21	154	0.012	<0.01	0.015/0.018	0.01/0.0092	0.050
CA*20	Plants	GA	28849	08/31/20	05/14/21	05/14/21		<0.01	<0.01	<0.01	<0.01	
					05/26/21	05/26/21		<0.01	<0.01	<0.01	<0.01	
		GB	28850	08/31/20	N/A	NA		---	---	---	---	
		GC	28851	08/31/20	05/24/21	05/24/21	266	0.86	0.015/0.018	<0.01	0.013/0.012	0.90
		GD	28852	08/31/20	05/24/21	05/24/21	266	1.1	0.022/0.027	<0.01	0.020/0.018	1.2
WA*403	Bulbs	DBA	28861	10/02/20	05/25/21	05/25/21		<0.01	<0.01	<0.01	<0.01	
		DBB	28862	10/02/20	N/A	NA		---	---	---	---	
		DBC	28863	10/02/20	05/25/21	05/25/21	235	<0.01	<0.01	<0.01	<0.01	0.040
		DBD	28864	10/02/20	05/25/21	05/25/21	235	<0.01	<0.01	<0.01	<0.01	0.040
TX380	Bulbs	DBA	29148	06/07/21	07/20/21	07/20/21		<0.01	<0.01	<0.01	<0.01	
		DBB	29149	06/07/21	N/A	NA		---	---	---	---	
		DBC	29150	06/07/21	07/20/21	07/20/21	43	0.012	<0.01	<0.01	<0.01	0.042
		DBD	29151	06/07/21	07/20/21	07/20/21	43	0.019	<0.01	<0.01	<0.01	0.049
CA16	Bulbs	DBA	28241	06/30/20	05/10/21	05/10/21		<0.01	<0.01	<0.01	<0.01	
		DBB	28242	06/30/20	N/A	NA		---	---	---	---	
		DBC	28243	06/30/20	05/10/21	05/10/21	314	0.029	<0.01	<0.01	0.011/0.010	0.059
		DBD	28244	06/30/20	05/10/21	05/10/21	314	0.021	<0.01	<0.01	0.010/0.0092	0.050
CA17	Bulbs	DBA	28304	08/07/20	05/10/21	05/10/21		<0.01	<0.01	<0.01	<0.01	
		DBB	28305	08/07/20	N/A	NA		---	---	---	---	
		DBC	28306	08/07/20	05/10/21	05/10/21	276	0.026	<0.01	<0.01	<0.01	0.056
		DBD	28307	08/07/20	05/10/21	05/10/21	276	0.031	<0.01	<0.01	<0.01	0.061
CA18	Plants	GA	28225	05/11/20	05/24/21	05/24/21		<0.01	<0.01	<0.01	<0.01	
		GB	28226	05/11/20	N/A	NA		---	---	---	---	
		GC	28227	05/11/20	05/24/21	05/24/21	378	1.3	0.043/0.052	0.030/0.036	0.046/0.042	1.4
		GD	28228	05/11/20	05/24/21	05/24/21	378	1.4	0.050/0.060	0.038/0.046	0.054/0.050	1.6
ID181	Bulbs	DBA	28857	09/14/20	05/04/21	05/04/21		<0.01	<0.01	<0.01	<0.01	
		DBB	28858	09/14/20	N/A	NA		---	---	---	---	
		DBC	28859	09/14/20	05/10/21	05/10/21	238	<0.01	<0.01	<0.01	<0.01	0.040
		DBD	28860	09/14/20	05/10/21	05/10/21	238	0.010	<0.01	<0.01	<0.01	0.040

Table VII.2: Residue Data Results (cont.)

Trial ID	Crop Fraction	Field Sample ID	Lab Sample ID	Sampling Date	Extraction Date	Analysis Date	Storage Interval ¹ (Days)	Residue Results (ppm)				
								Flonicamid	TFNA-AM ppmfound/ parent equivalent ²	TFNA ppm found/ parent equivalent ²	TFNG ppmfound/ parent equivalent ²	Total Flonicamid ³
WA404	Bulbs	DBA	28654	09/08/20	05/25/21	05/25/21		<0.01	<0.01	<0.01	<0.01	
		DBB	28655	09/08/20	N/A	NA		---	---	---	---	
		DBC	28656	09/08/20	05/25/21	05/25/21	259	0.011	<0.01	<0.01	<0.01	0.041
		DBD	28657	09/08/20	05/25/21	05/25/21	259	<0.01	<0.01	<0.01	<0.01	0.040
OR405	Plants	GA	28265	06/11/20	05/19/21	05/19/21		<0.01	<0.01	<0.01	<0.01	
		GB	28266	06/11/20	N/A	NA		---	---	---	---	
		GC	28267	06/11/20	05/19/21	05/19/21	342	0.68	0.015/0.018	0.013/0.016	<0.01	0.72
		GD	28268	06/11/20	05/19/21	05/19/21	342	0.71	0.012/0.014	0.014/0.017	<0.01	0.75
CO461	Bulbs	DBA	28853	08/23/20	05/06/21	05/06/21		<0.01	<0.01	<0.01	<0.01	
		DBB	28854	08/23/20	N/A	NA		---	---	---	---	
		DBC	28855	08/23/20	05/06/21	05/06/21	256	0.13	<0.01	<0.01	<0.01	0.16
		DBD	28856	08/23/20	05/06/21	05/06/21	256	0.11	<0.01	<0.01	<0.01	0.14

N/A=Not Analyzed

¹Storage Interval calculated from sampling to extraction of treated samples²Parent equivalent shown for treated samples with residues $\geq$ LLMV.³Total=flonicamid + TFNA-AM + TFNA + TFNG (metabolites in parent equivalents); for residues <LLMV a value of 0.01 is used to calculate the total

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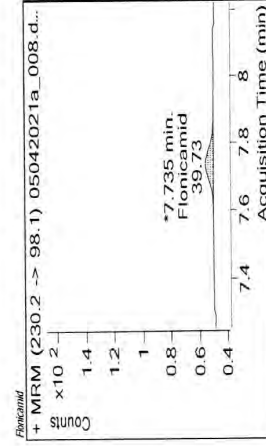
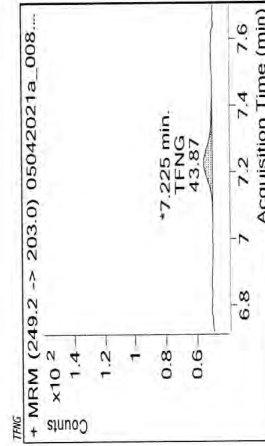
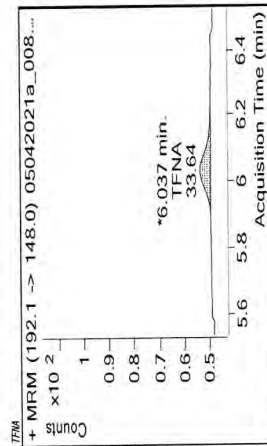
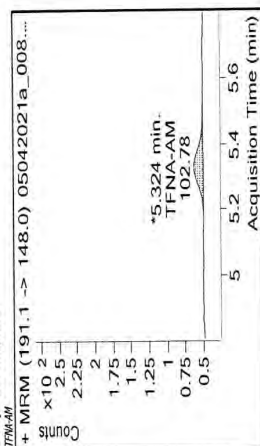
Each chromatogram represents a 10.0 µL injection.

An asterisk (*) next to the retention time on a chromatogram indicates that the peak was manually integrated.

05042021a_008.d
687-2M10 0.05 pg/ μ L
Calibration

Acq Time 5/4/21 18:03
Acq Method File 08550_EST_POS.m
Sample Info Vial 5
Position
Compound Graphics

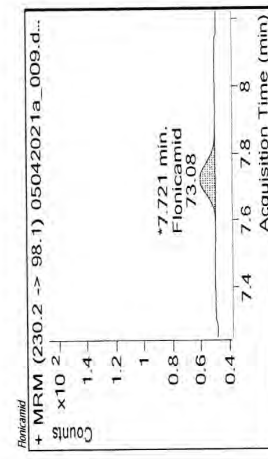
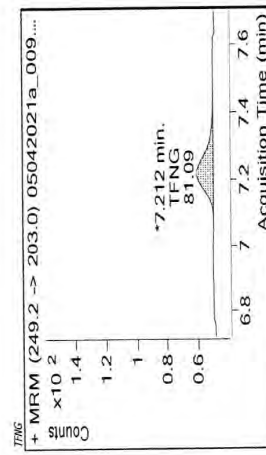
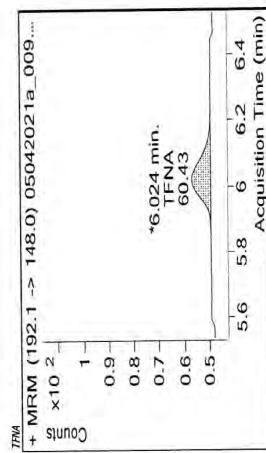
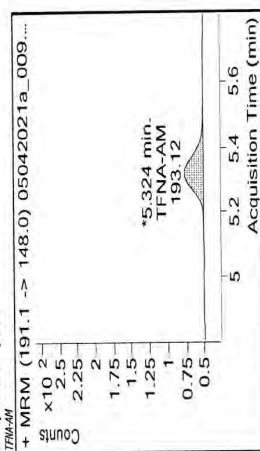
TFNA-AM



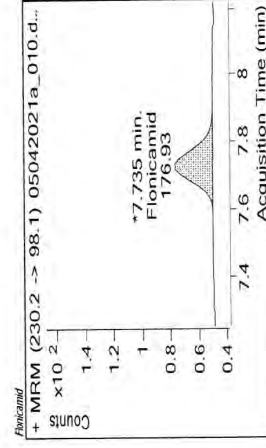
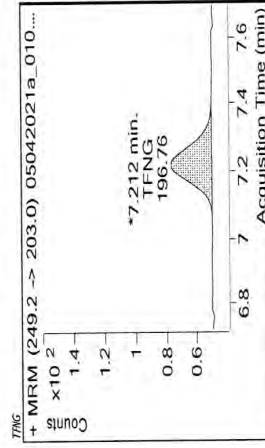
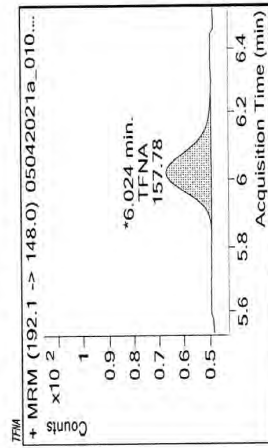
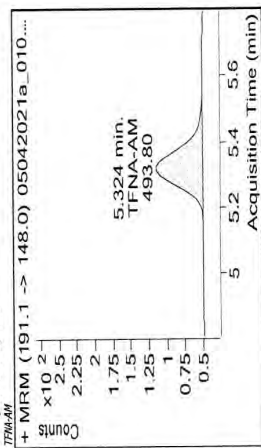
05042021a_009.d
6872M9 0.10 pg/μL
Calibration

Acq Time 5/4/21 18:22
Acq Method File 08550_ESI_POS.m
Sample Info Val 4
Position
Compound Graphics

TFNA-AM



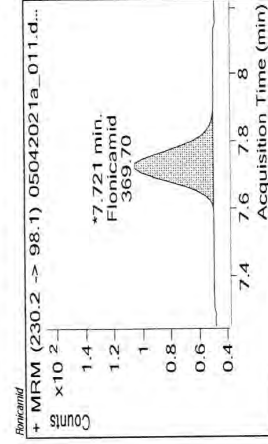
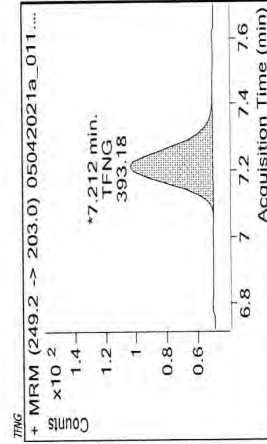
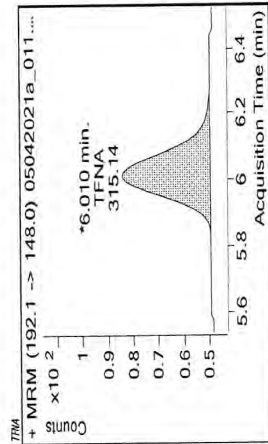
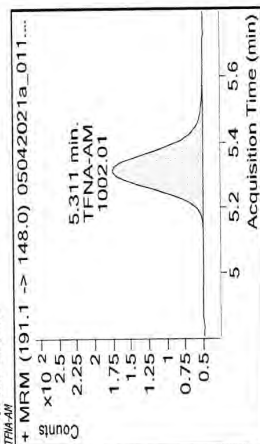
Acq Time 5/4/21 18:41 Data File 05042021a_010.d
 Acq Method File 08550_ESI_POS.m Sample Name
 Sample Info 687-208 0.25 µl
 Position Vial 3 Sample Type Calibration
 Compound Graphics



05042021a_011.d
687-2M7 0.50 µg/µL
Calibration

Data File
Sample Name
Sample Type

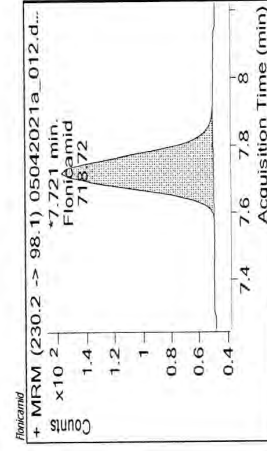
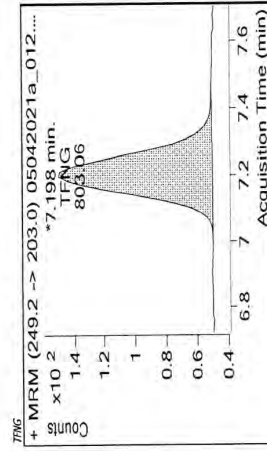
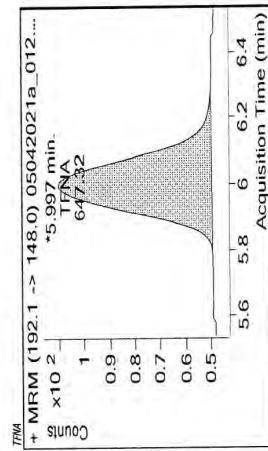
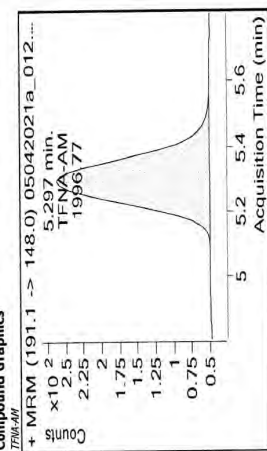
Acq Time 5/4/21 19:00
Acq Method File 08550_EST_POS.m
Sample Info Vial 2
Position
Compound Graphics



05042021a_012.d
687-2M6.1.0 pg/ul
Calibration

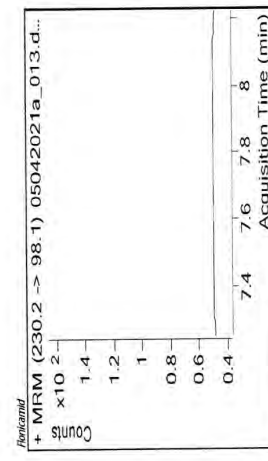
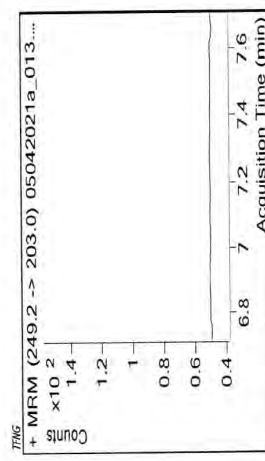
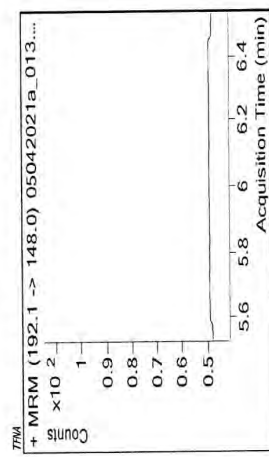
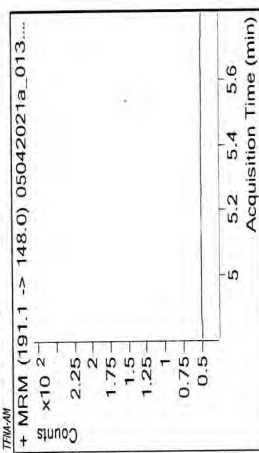
Data File
Sample Name
Sample Type

Acq Time 5/4/21 19:19
Acq Method File 08550_ESI_POS.m
Sample Info Vial 1
Position
Compound Graphics

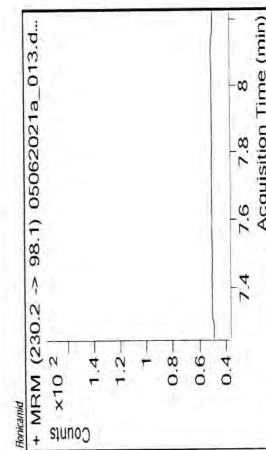
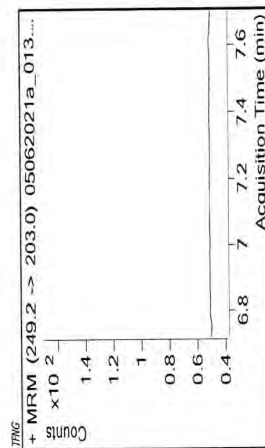
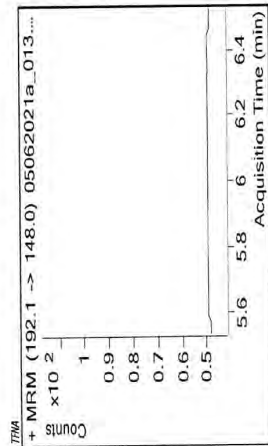
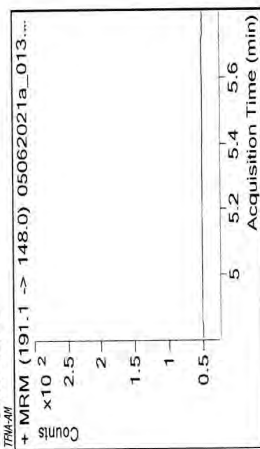


Acq Time 5/4/21 19:38 Data File 05042021a_013.d
 Acq Method File 0850_FSI_POS.m Sample Name 28857
 Sample Info D040181 Sample Type Sample
 Position PI-A1

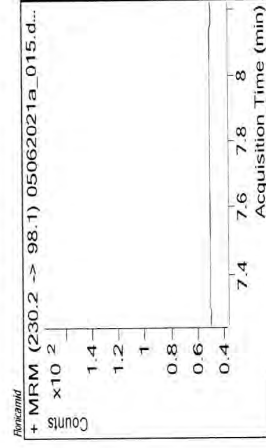
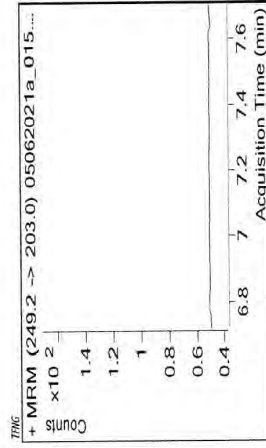
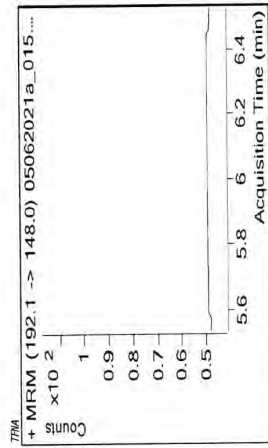
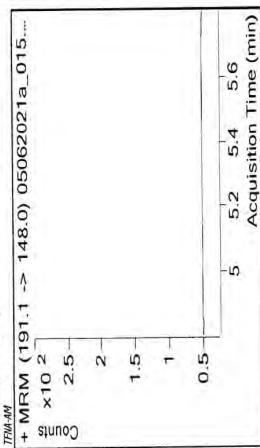
Compound Graphics



Acq Time 5/6/21 23:18 Data File 05062021a_013.d
Acq Method File 08550_ESL_POS.m Sample Name 28853
Sample Info DBA-CO-61 Sample Type Sample
Position P1-A1
Compound Graphics



Acq Time 5/6/21 23:56 Data File 05062021a_015.d
 Acq Method File 08550_EST_POS.m Sample Name 39999
 Sample Info 08A-CK-19 Sample Type Sample
 Position PLAL
 Compound Graphics



05192021a_013.d
28265
Sample

Data File
Sample Name
Sample Type

5/19/21 20:28
08550 ESI_POS.m
GA-OR405
P1-A1

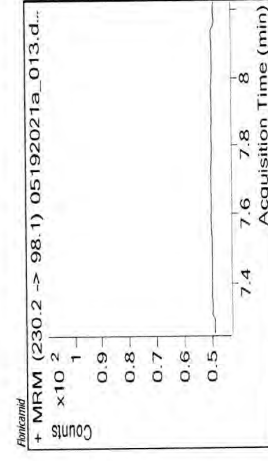
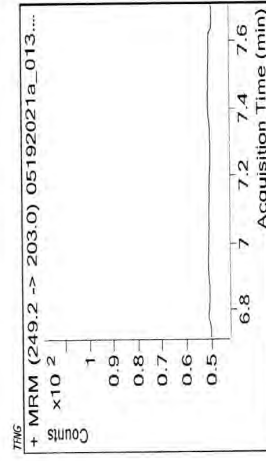
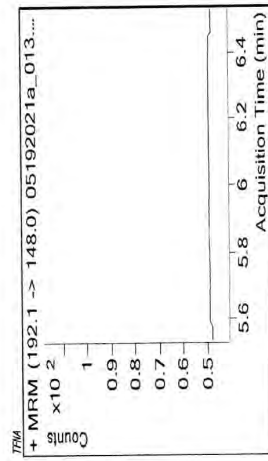
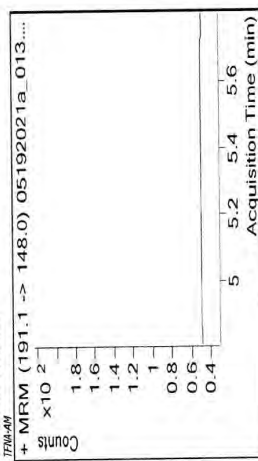
Acq Time

Acq Method File

Sample Info

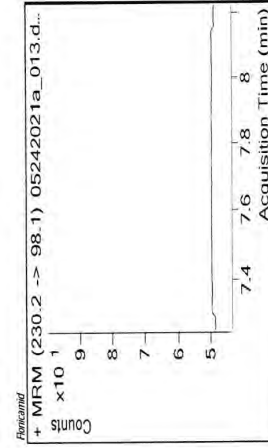
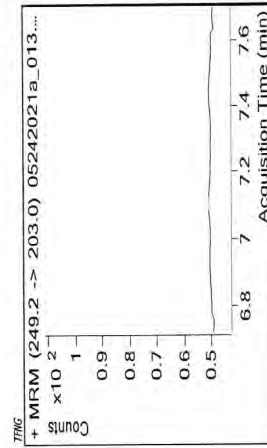
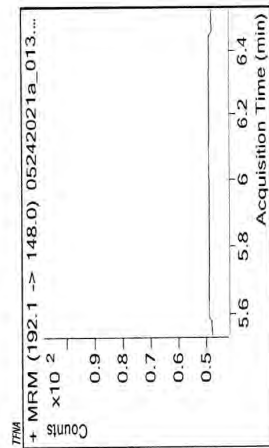
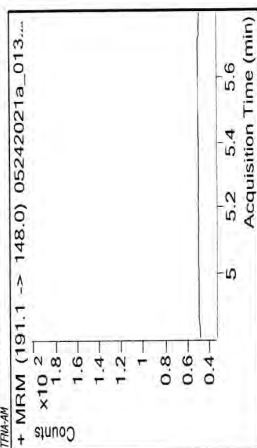
Position

Compound Graphics



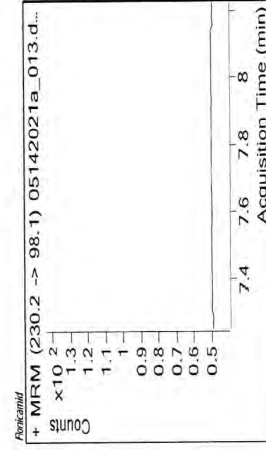
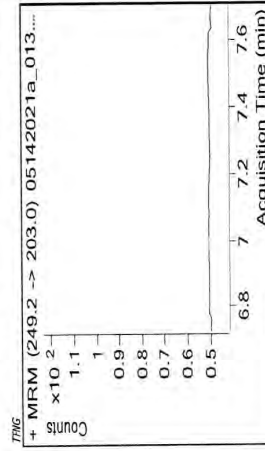
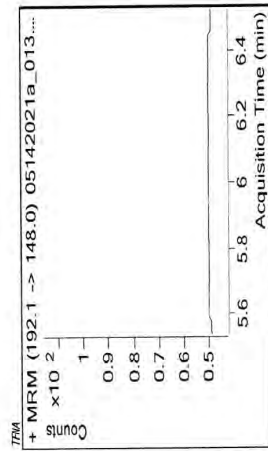
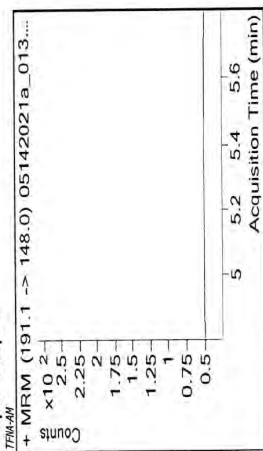
Acq Time 5/24/21 19:18 Data File 05242021a_013.d
Acq Method File 8531ESI_POS.m Sample Name 28225
Sample Info 05242021a_013 Sample Type Sample
Position PL-A1

Compound Graphics



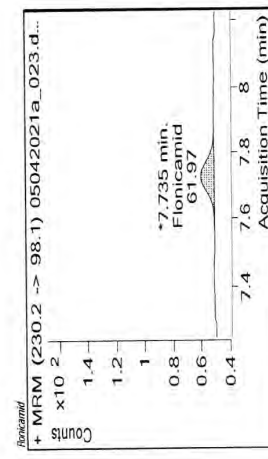
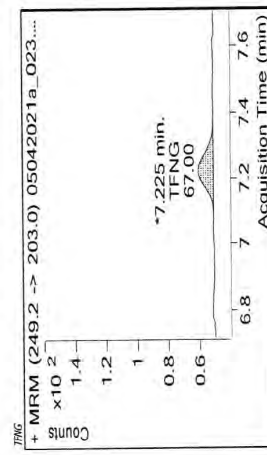
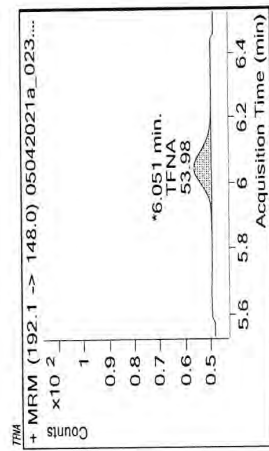
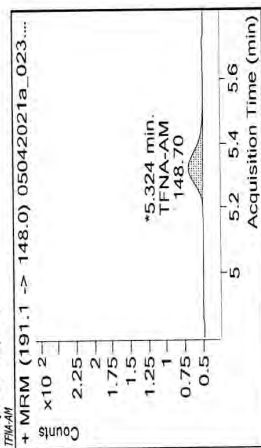
Acq Time 5/14/21 19:46 Data File 05142021a_013.d
Acq Method File 08550_EST_POS.m Sample Name 28849
Sample Info GA-CA*20 Sample Type Sample
Position P1-A1

Compound Graphics



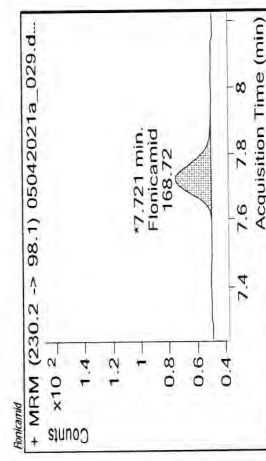
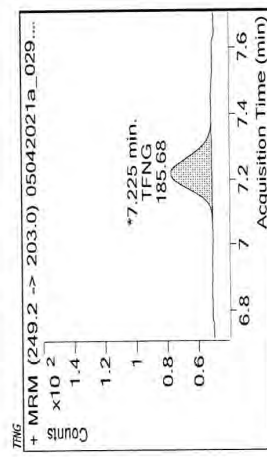
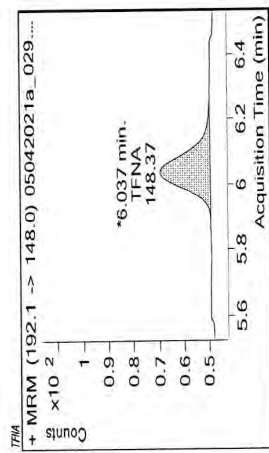
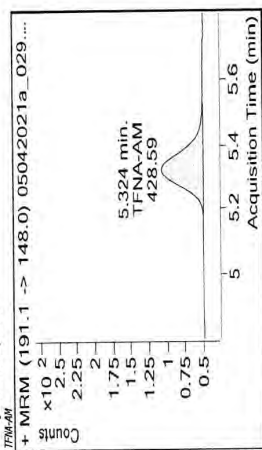
Acq Time 5/4/21 22:48
Acq Method File 08550_ESI_POS.m
Sample Info DBA-ID181
Position PI-A5
Data File 05042021a_023.d
Sample Name 28857VO.01R9
Sample Type Sample

Compound Graphics



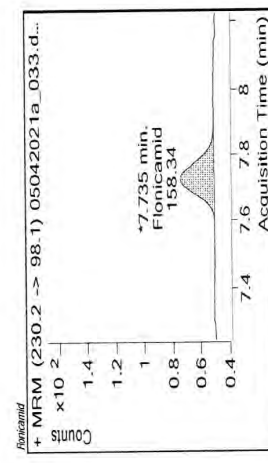
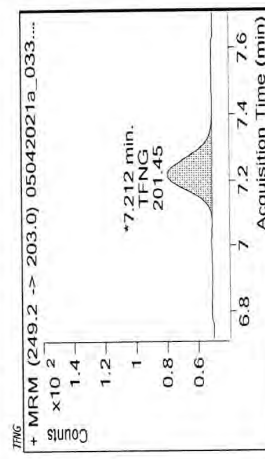
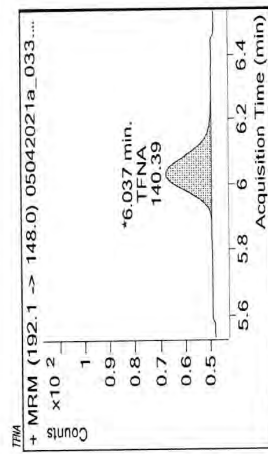
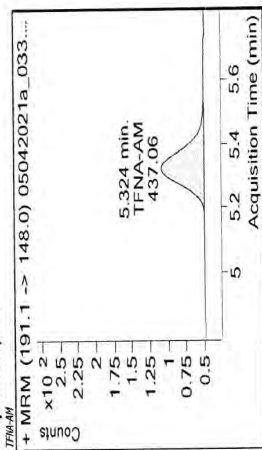
Acq Time 5/5/21 0:42 Data File 05042021a_029.d
Acq Method File 08550_ESI_POS.m Sample Name 28857V0.1R5
Sample Info DBA:ID:181 Sample Type Sample
Position P1-A7

Compound Graphics

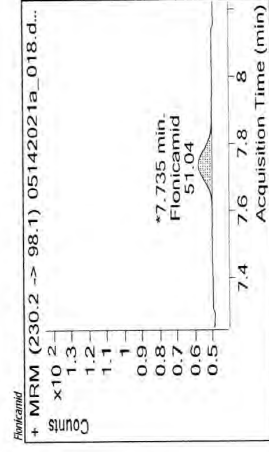
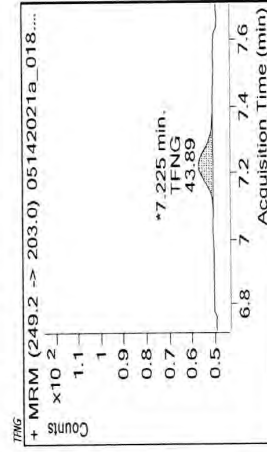
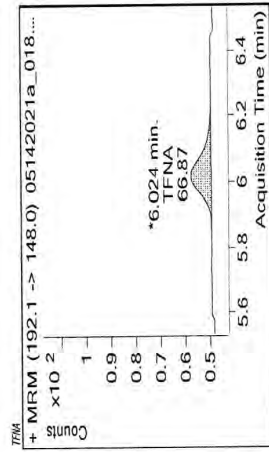
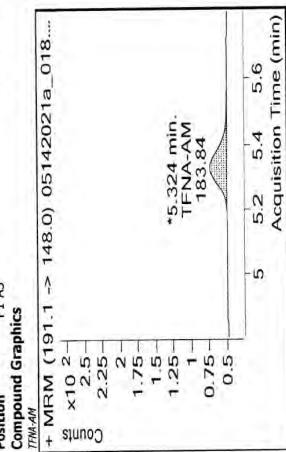


Acq Time 5/5/21 1:58 Data File 05042021a_033.d
Acq Method File 08550_ESI_POS.m Sample Name 28857V1.0K4
Sample Info DBA-ID181 Sample Type Sample
Position P1-A9

Compound Graphics



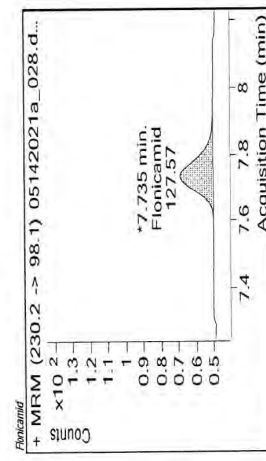
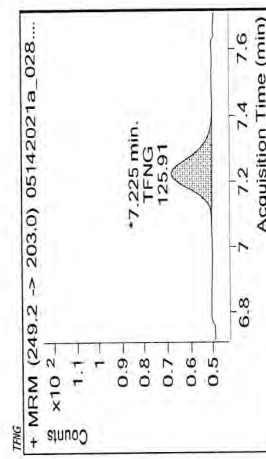
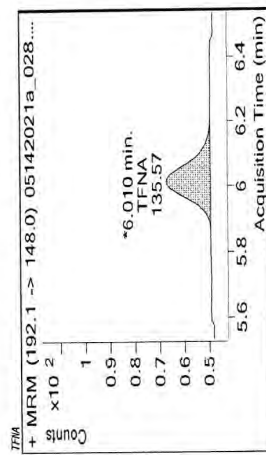
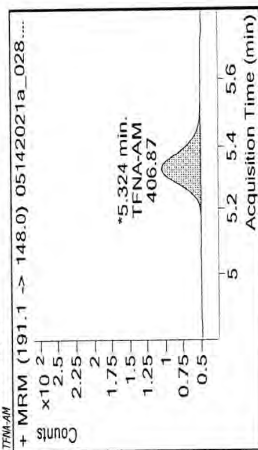
Acq Time 5/14/21 21:21 Data File 05142021a_018.d
Acq Method File 06550_ESL_POS.m Sample Name 28849V0.01R1
Sample Info GA-CA*20 Sample Type Sample
Position P1-A3
Compound Graphics



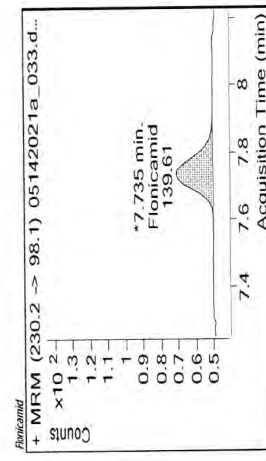
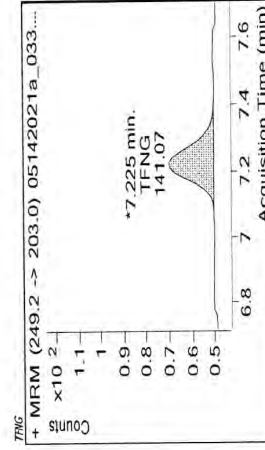
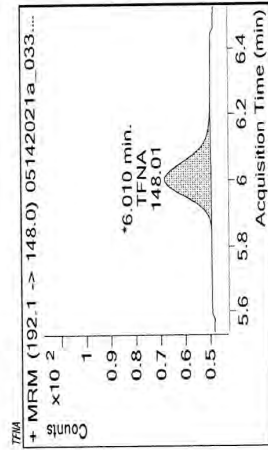
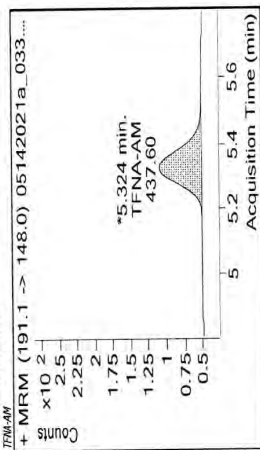
05142021a_028.d
28849V01R2
Sample

5/15/21 0:31
06550 ESI_POS.m
GA-CX-20
PI-A7
Sample Name
Sample Type

Compound Graphics

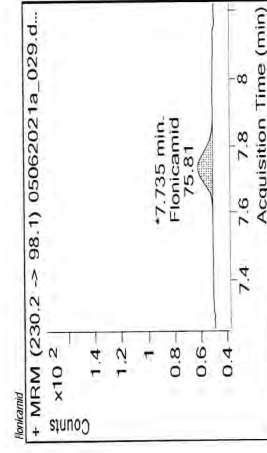
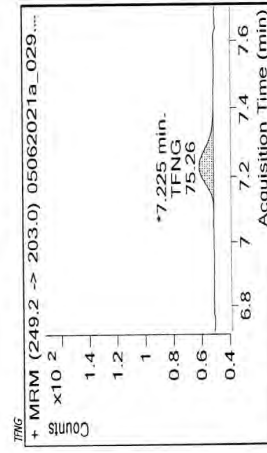
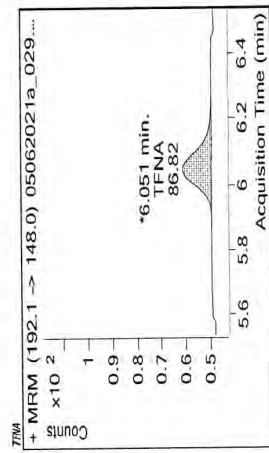
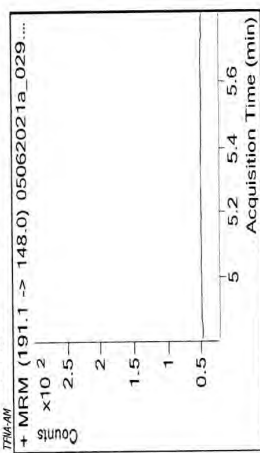


Acq Time 5/15/21 2:06 Data File 05142021a_033.d
Acq Method File 08550_ESI_POS.m Sample Name 28849V1.0R1
Sample Info GA-CA*20 Sample Type Sample
Position P1-A9
Compound Graphics

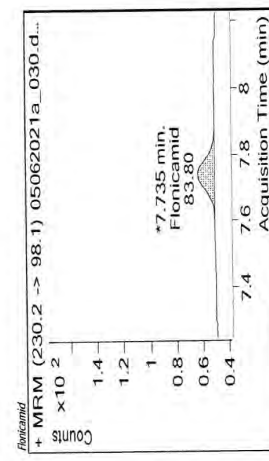
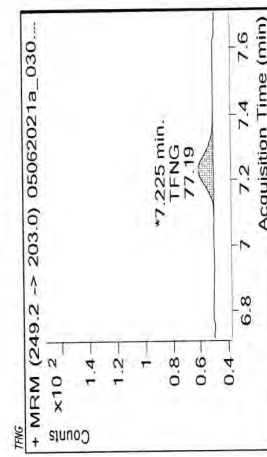
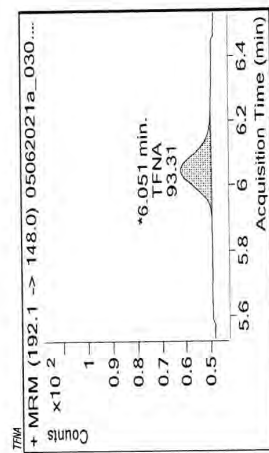
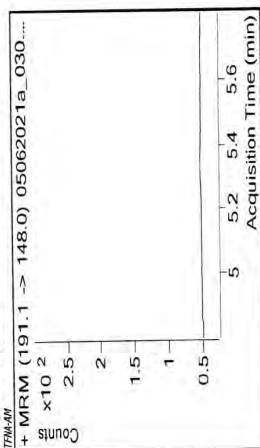


Acq Time 5/7/21 4:22 Data File 05062021a_029.d
Acq Method File 08550_ESI_POS.m Sample Name 28891
Sample Info D8C-CA*19 Sample Type Sample
Position P1-A7

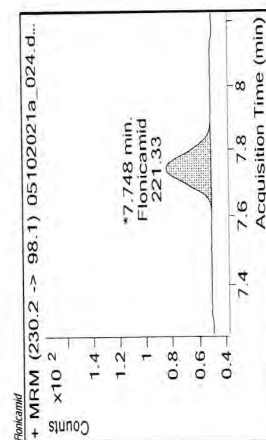
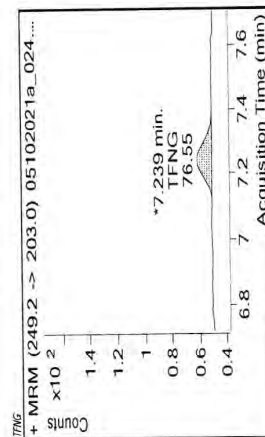
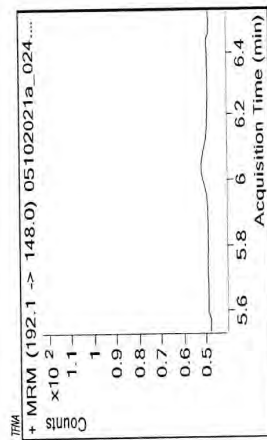
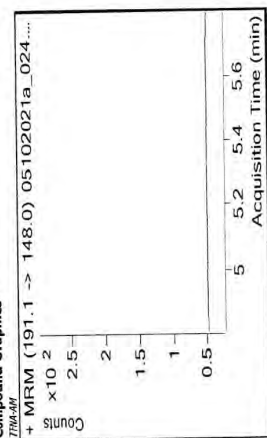
Compound Graphics



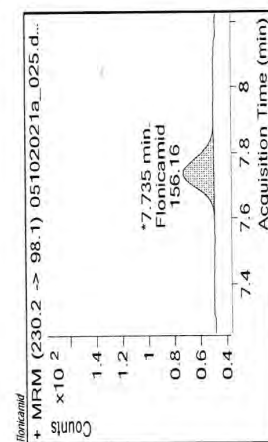
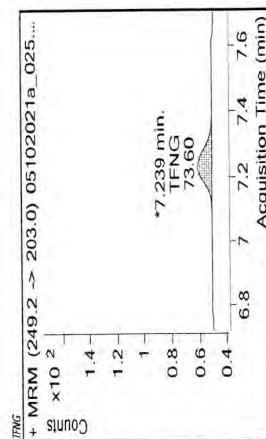
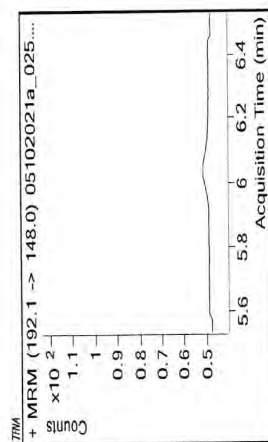
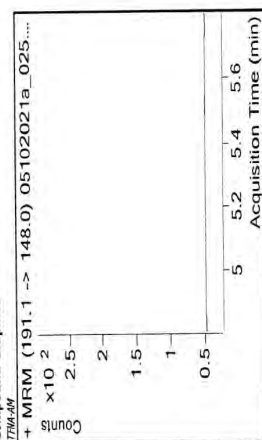
Acq Time 5/7/21 4:41 Data File 05062021a_030.d
Acq Method File 185501_ESI_POS.m Sample Name 28992
Sample Info DDD-CA*19 Sample Type Sample
Position PL-A8
Compound Graphics



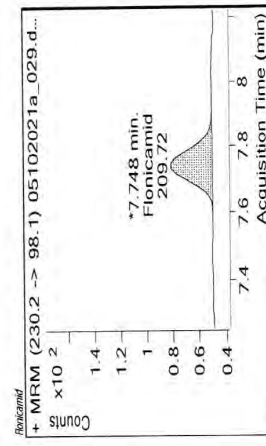
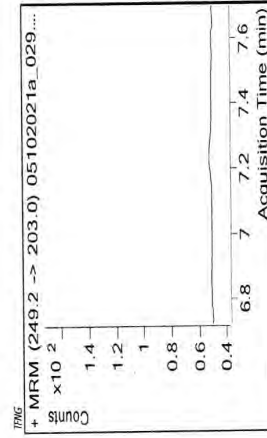
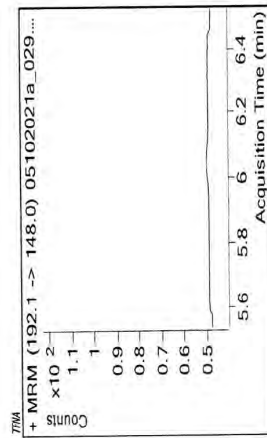
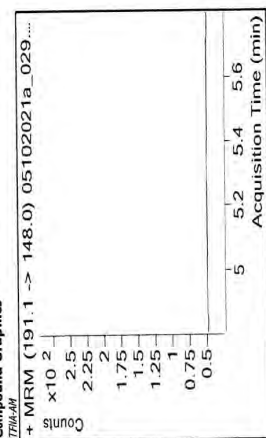
Acq Time 5/11/21 1:44 Data File 05102021a_024.d
Acq Method File 08550_ESI_POS.m Sample Name 28243
Sample Info DBC-CA16 Sample Type Sample
Position P1-A5
Compound Graphics



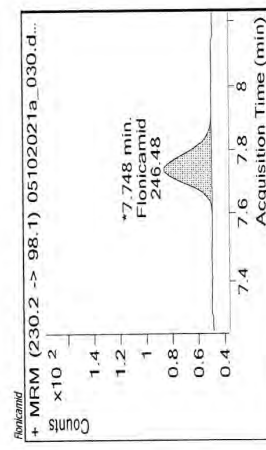
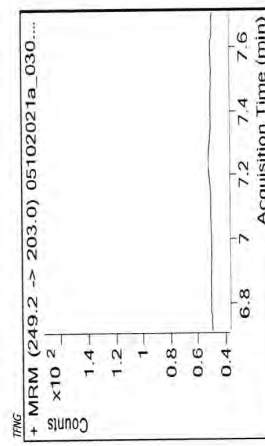
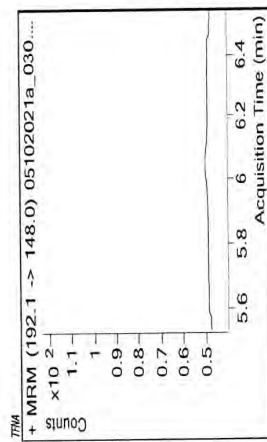
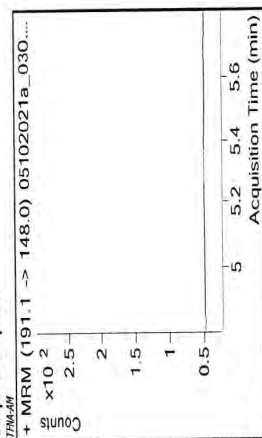
Acq Time 5/11/21 2:03 Data File 05102021a_025.d
Acq Method File 08550_ESI_POS.m Sample Name 28244
Sample Info DBD-CA16 Sample Type Sample
Position P1-A6
Compound Graphics



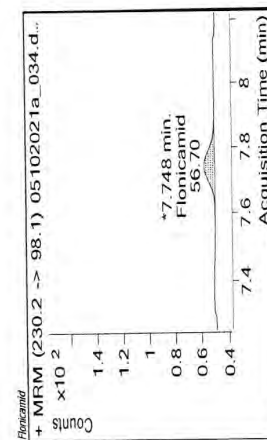
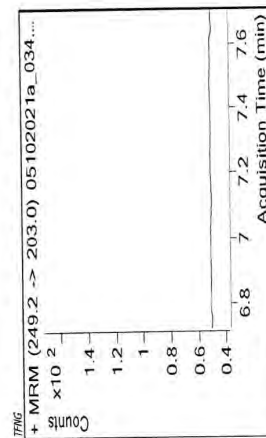
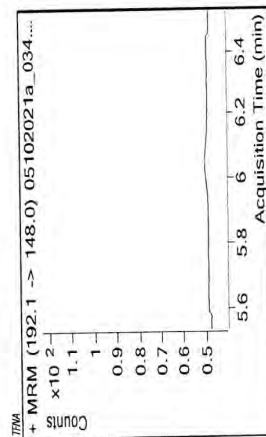
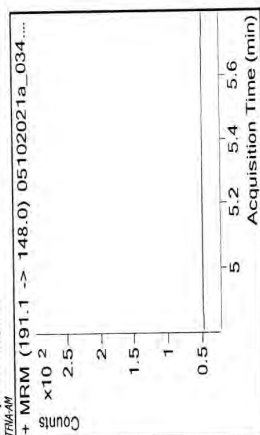
Acq Time 5/11/21 3:19 Data File 05102021a_029.d
Acq Method File 08550_ESI_POS.m Sample Name 28306
Sample Info DEC-CA17 Sample Type Sample
Position P1-A7
Compound Graphics



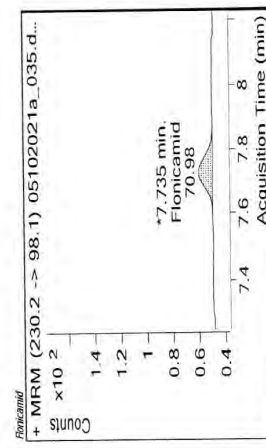
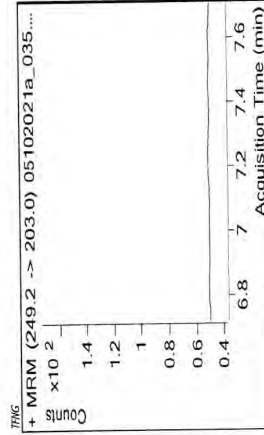
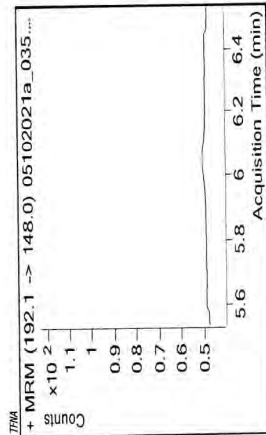
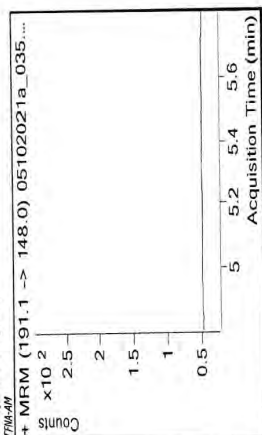
Acq Time 5/11/21 3:38 Data File 05102021a_030.d
Acq Method File 08550_ESI_POS.m Sample Name 28307
Sample Info DBD-CA17 Sample Type Sample
Position P1-A8
Compound Graphics



Acq Time 5/11/21 4:54 Data File 05102021a_034.d
Acq Method File 08550_ESI_POS.m Sample Name 28859
Sample Info DEC10181 Sample Type Sample
Position PL1A9
Compound Graphics



Acq Time 5/11/21 5:13 Data File 05102021a_035.d
 Acq Method File 08550_ESI_POS.m Sample Name 28860
 Sample Info DBQ-ID181 Sample Type Sample
 Position P1-B1
 Compound Graphics



05/25/2021a_029.d
28556
Sample

5/26/21 3:12
08556 ES1 POS.m
DOCW/404
PLA7

Acq Time

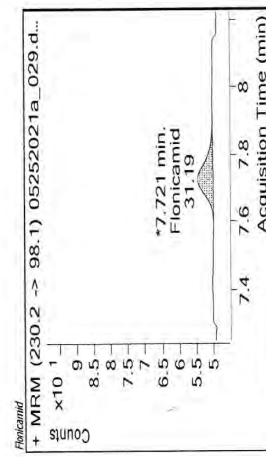
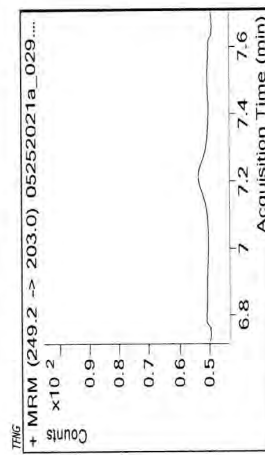
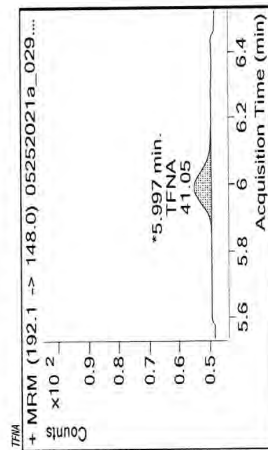
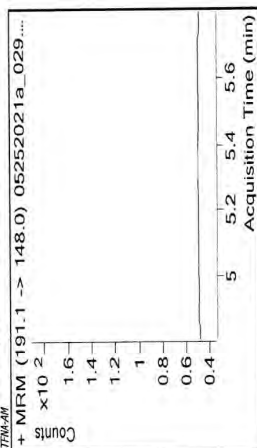
Acq Method File

Sample Info

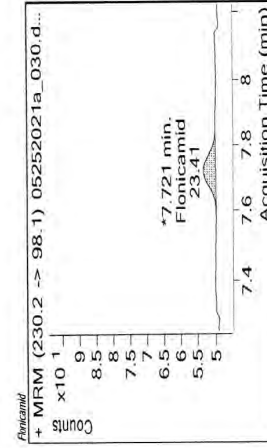
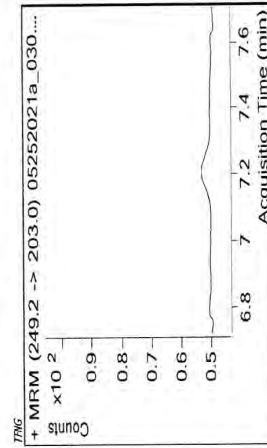
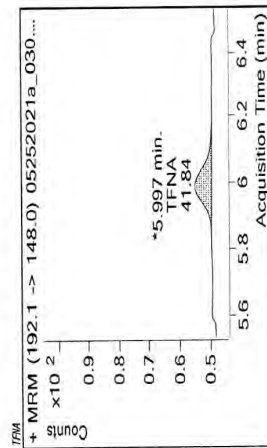
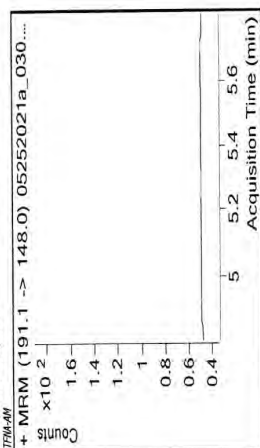
Position

Compound Graphics

Data File
Sample Name
Sample Type



Acq Time 5/26/21 3:32 Data File 05252021a_030.d
Acq Method File 05252021a_030.m Sample Name 28657
Sample Info DBQ-WA04 Sample Type Sample
Position P1-A8
Compound Graphics
TFNA



05192021a_026.d
28267 (Flonicamid)
Sample

5/20/21 0:35
08550_ESL_POS.m
GC-OK405
P1-A6

Acq Time

Acq Method File

Sample Info

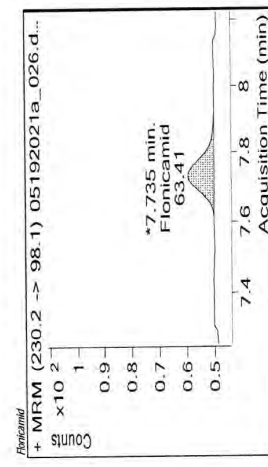
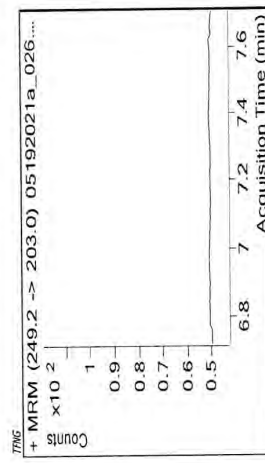
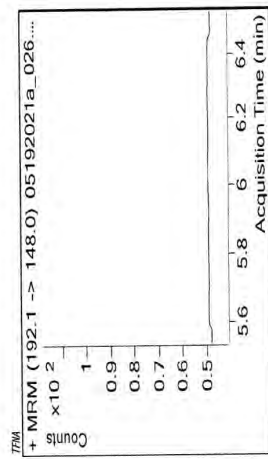
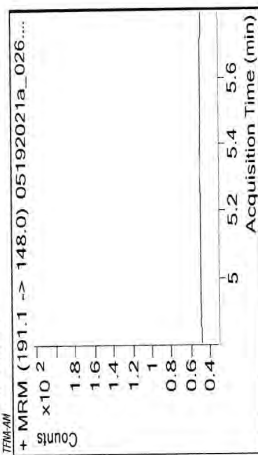
Position

Compound Graphics

Data File

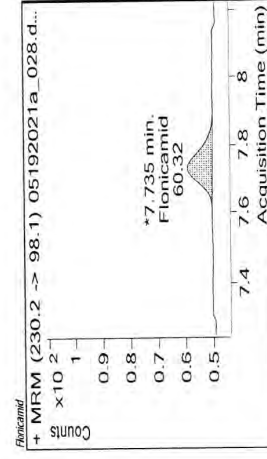
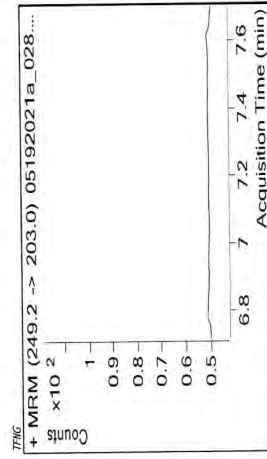
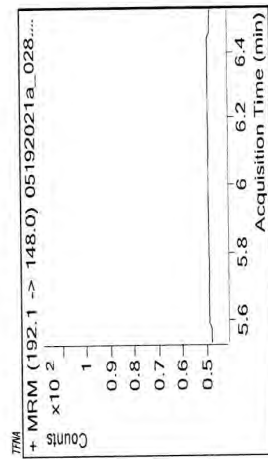
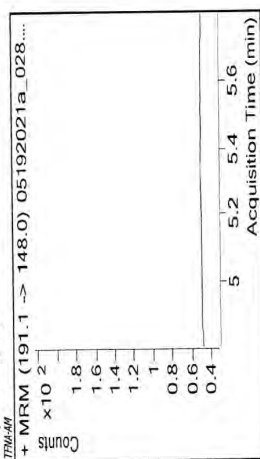
Sample Name

Sample Type



Acq Time 5/20/21 1:13
Acq Method File 08550_ESI_POS.m
Sample Info 05192021a_028.d
Position 28268 (Fonicamid)
Compound Graphics P1-A7

Data File 05192021a_028.d
Sample Name 28268 (Fonicamid)
Sample Type Sample



5/19/21 23:00
08550_ESI_POS.m
GC-08405
PL-44

Acq Time
Acq Method File
Sample Info
Position

Compound Graphics

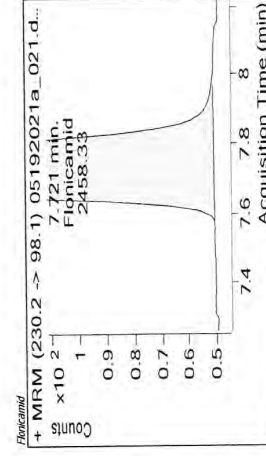
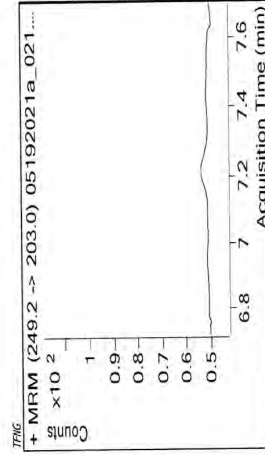
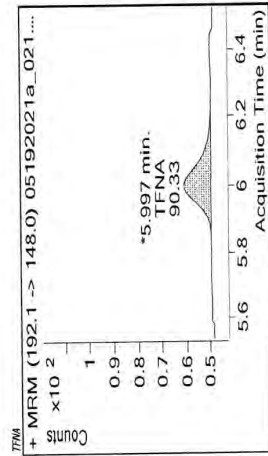
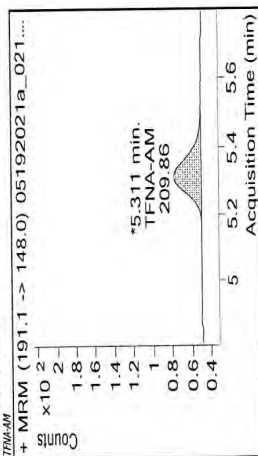
Counts
x10²

2
1.8
1.6
1.4
1.2
1
0.8
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0.4

5 5.2 5.4 5.6
Acquisition Time (min)

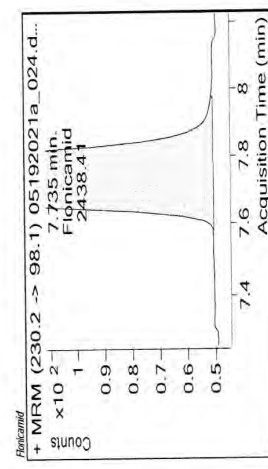
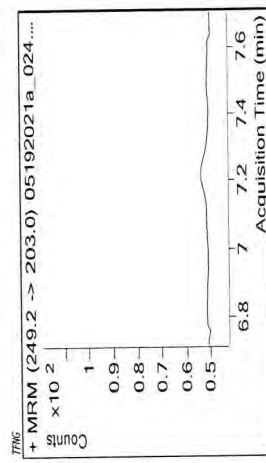
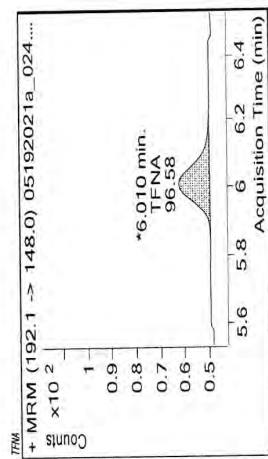
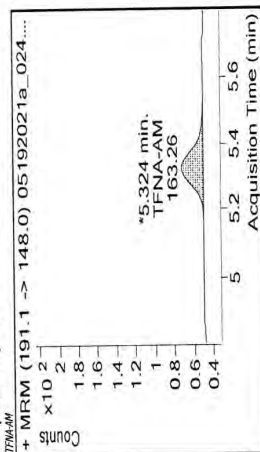
5/19/2021a_021.d
38367
Sample

Data File
Sample Name
Sample Type

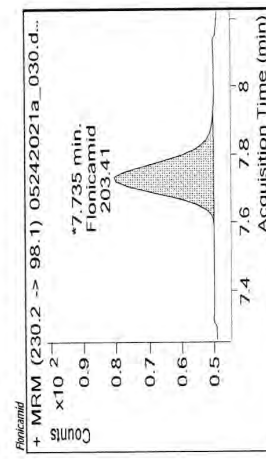
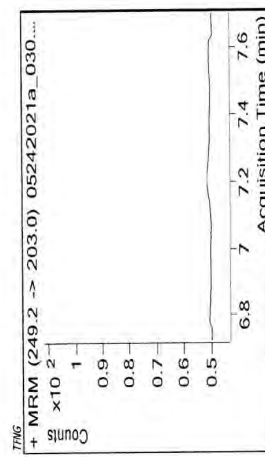
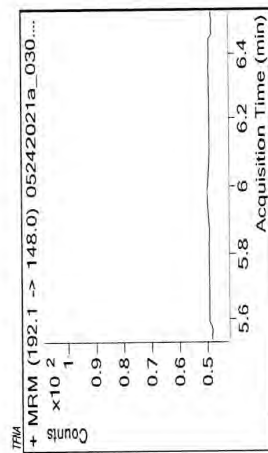
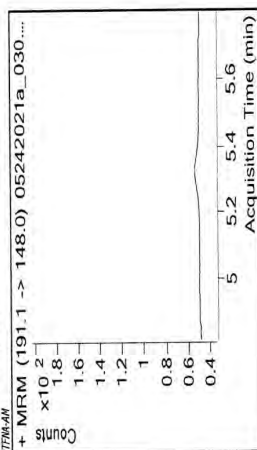


Acq Time 5/19/21 23:57 Data File 05192021a_024.d
Acq Method File 08550_EST_POS.m Sample Name 28368
Sample Info 00000000000000000000 Sample Type Sample
Position PLAS

Compound Graphics

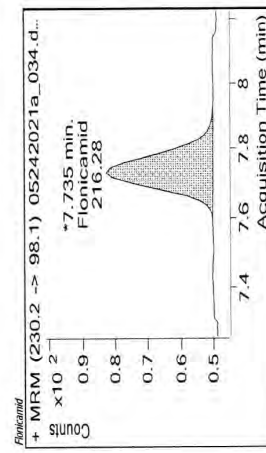
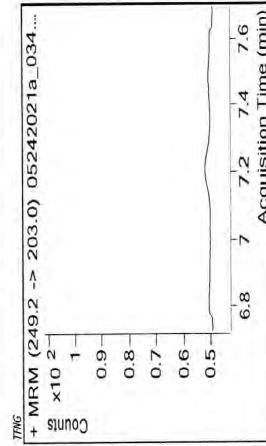
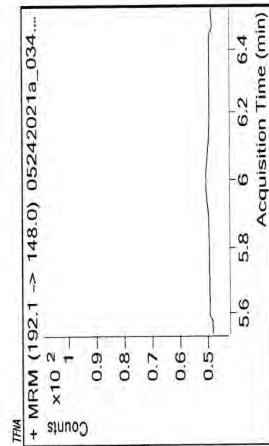
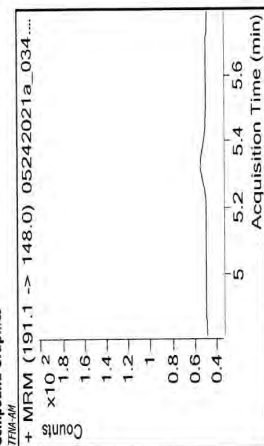


Acq Time 5/25/21 0:41 Data File 05242021a_030.d
Acq Method File 08550_Test_POS.m Sample Name 28277 (Fionicamid)
Sample Info P1-A8 Sample Type Sample
Position P1-A8
Compound Graphics



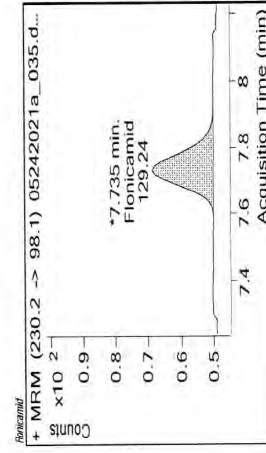
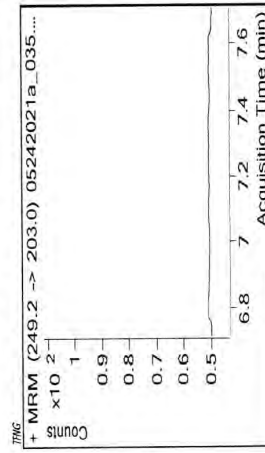
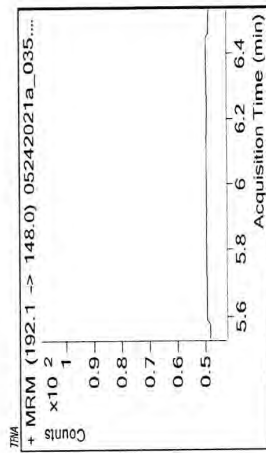
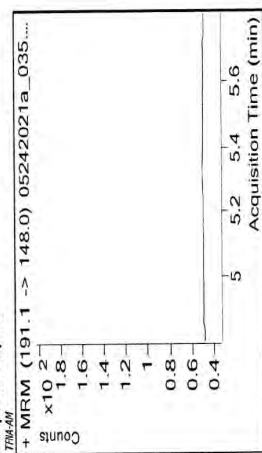
Acq Time 5/25/21 1:57
Acq Method File 08550_ESI_POS.m
Sample Info GD-CA18
Position P1-A9
Compound Graphics

Data File 05242021a_034.d
Sample Name 28228 (Flonicamid)
Sample Type Sample



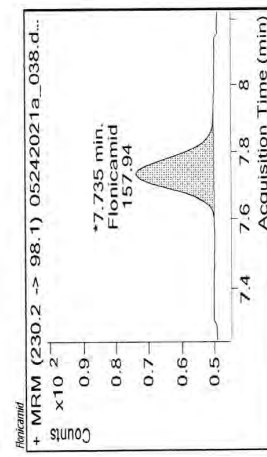
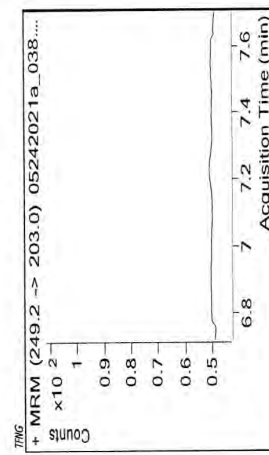
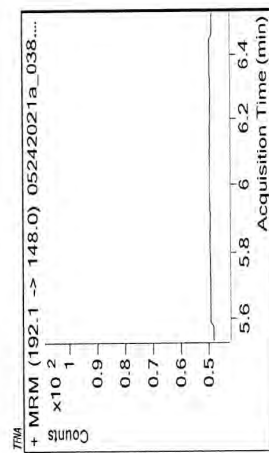
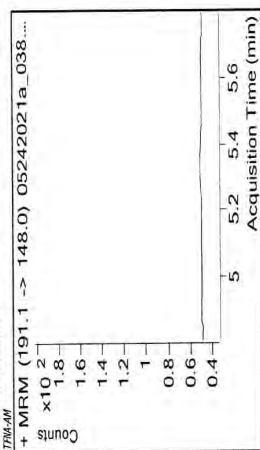
Acq Time 5/25/21 2:16 Data File 05242021a_035.d
Acq Method File 08550_EST_POS.m Sample Name 28851 (Flonicamid)
Sample Info GC-CA20 Sample Type Sample
Position P1-B1

Compound Graphics



Acq Time 5/25/21 3:13 Data File 05242021a_038.d
Acq Method File 08550_ESI_POS.m Sample Name 28852 (Fonicamid)
Sample Info GP-CA 20 Sample Type Sample
Position P1-B2

Compound Graphics



5/24/21 21:50
05242021a_021.d
Sample Name
28227
Sample

05242021a_021.d
Sample Name
28227
Sample

05242021a_021.d
Sample Name
28227
Sample

05242021a_021.d
Sample Name
28227
Sample

05242021a_021.d
Sample Name
28227
Sample

05242021a_021.d
Sample Name
28227
Sample

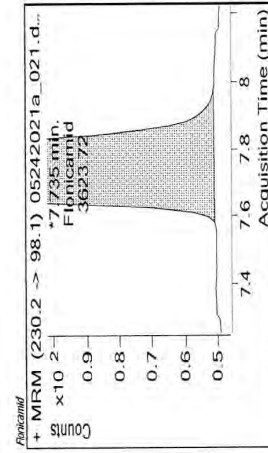
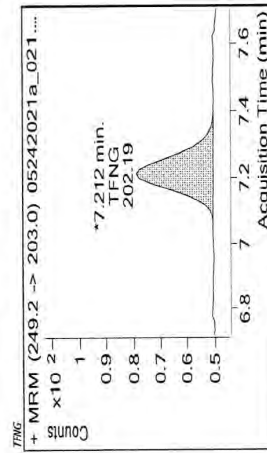
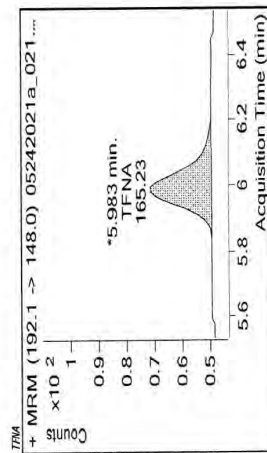
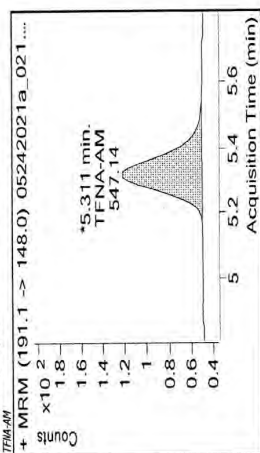
05242021a_021.d
Sample Name
28227
Sample

05242021a_021.d
Sample Name
28227
Sample

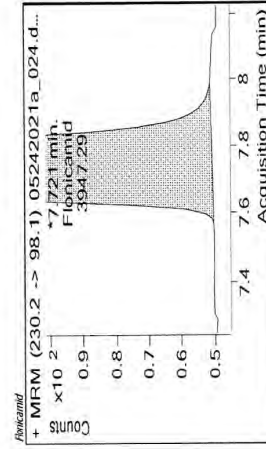
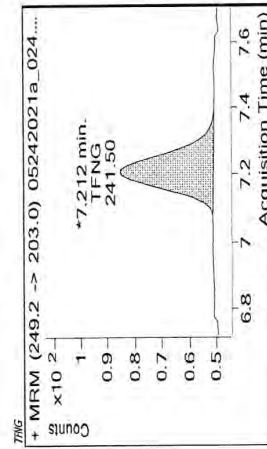
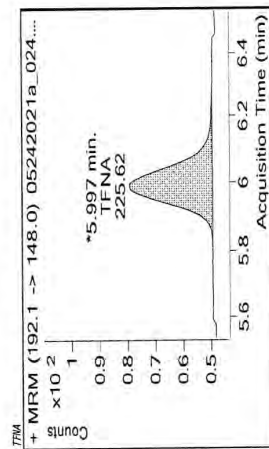
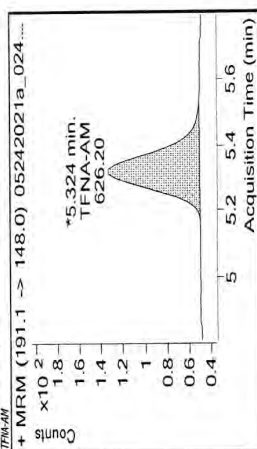
05242021a_021.d
Sample Name
28227
Sample

05242021a_021.d
Sample Name
28227
Sample

05242021a_021.d
Sample Name
28227
Sample

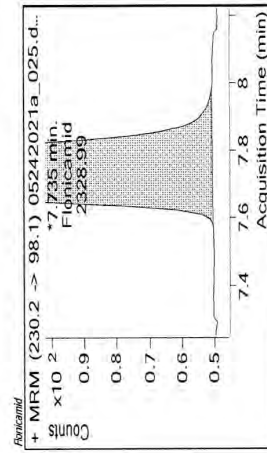
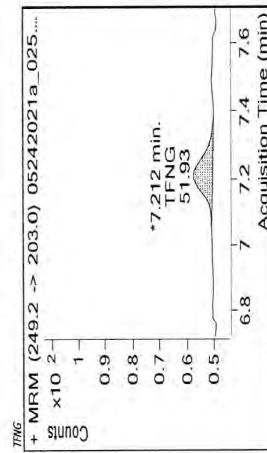
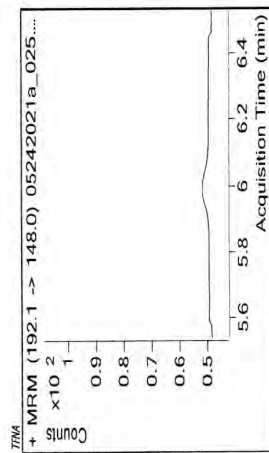
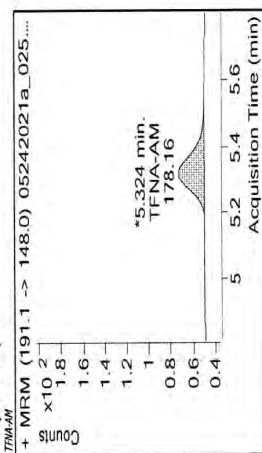


Acq Time 5/24/21 22:47 Data File 05242021a_024.d
Acq Method File 08501_TSI_POS.m Sample Name
Sample Info 00-AQ18 Sample
Position P1A5
Compound Graphics



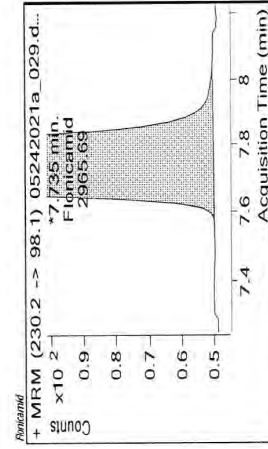
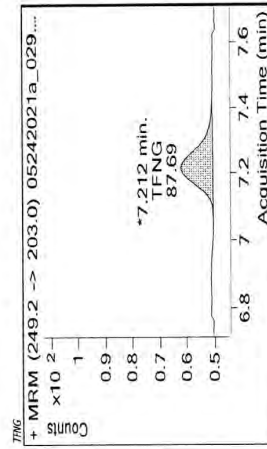
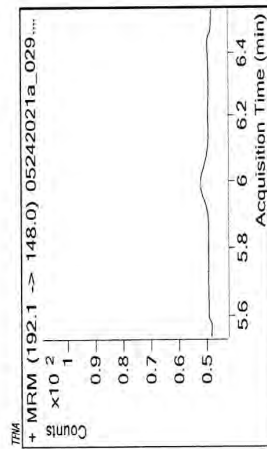
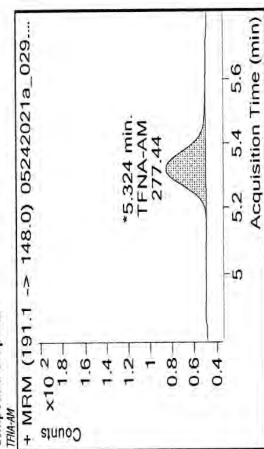
Acq Time 5/24/21 23:06 Data File 05242021a_025.d
Acq Method File 08550_ESI_POS.m Sample Name 28851
Sample Info GC-CA*20 Sample Type Sample
Position P1-A6

Compound Graphics



Acq Time 5/25/21 0:22 Data File 05242021a_029.d
Acq Method File 08550_ESL_POS.m Sample Name 28852
Sample Info GI CA*20 Sample Type Sample
Position PI-A7

Compound Graphics



ATTACHMENT B: DATA SUMMARIES AND STANDARD CURVES

Compound
Fonicamid
08550 - Fonicamid/Onion - Method Validation - Dry Bulb - Extracted 5/4/2021 by ASM

Injection Date
5/4/2021

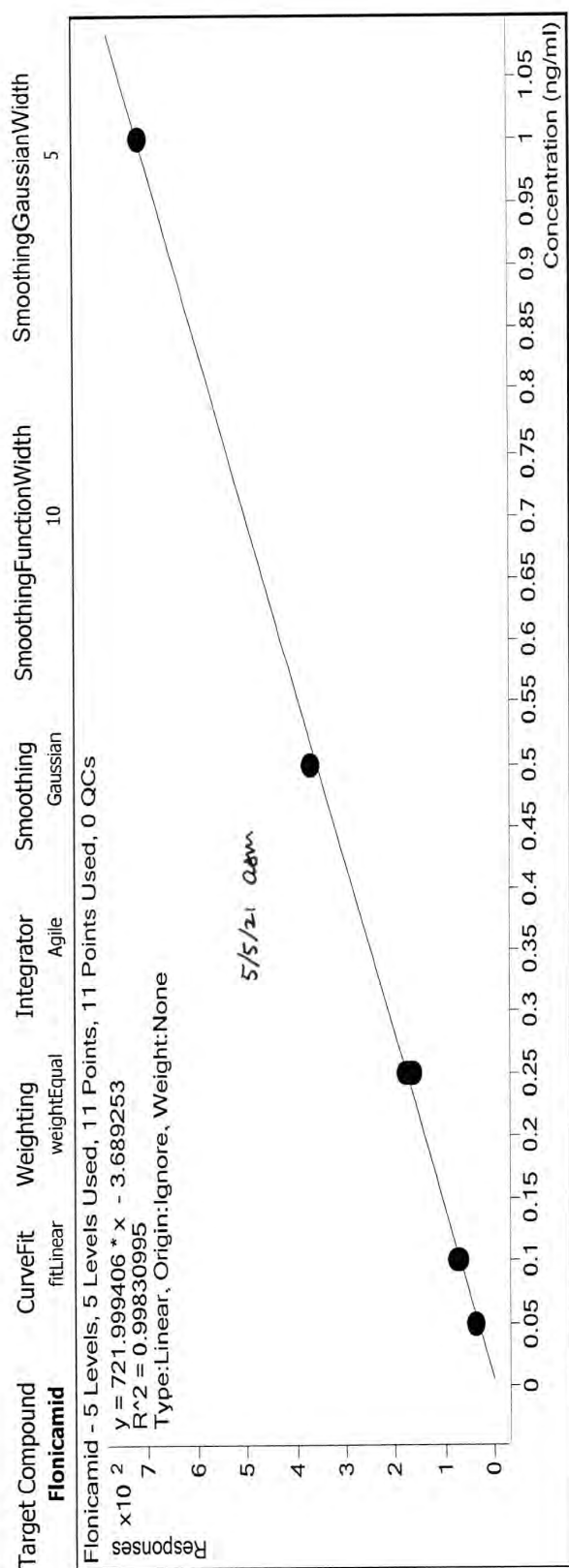
5/5/21 *asm*

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp (pg/µL)	Conc (pg/µL)	ppm	Ave ppm	% Rec	Ave % Rec	Std Dev
05042021a_001.d	7.830	Condition		Sample	P1-A3	10.0										
05042021a_002.d	7.721	Condition		Sample	P1-A3	10.0										
05042021a_003.d	7.721	Condition		Sample	P1-A3	10.0										
05042021a_004.d	7.721	Condition		Sample	P1-A3	10.0										
05042021a_005.d	7.721	Condition		Sample	P1-A3	10.0										
05042021a_006.d	7.721	Condition		Sample	P1-A3	10.0										
05042021a_007.d	7.735	Condition		Sample	P1-A3	10.0										
05042021a_008.d	7.735	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				39.73						
05042021a_009.d	7.721	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				73.08						
05042021a_010.d	7.735	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				176.93						
05042021a_011.d	7.721	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				369.70						
05042021a_012.d	7.721	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				718.72						
05042021a_013.d	-----	28857	DBA-ID181	Sample	P1-A1	10.0	0.100		0.1000		<0.10	<0.01				
05042021a_014.d	-----	28857	DBA-ID181	Sample	P1-A1	10.0	0.100		0.1000		<0.10	<0.01				
05042021a_015.d	-----	Reagent	Blank	Sample	P1-A2	10.0	0.100		0.1000		<0.10	<0.01				
05042021a_016.d	-----	Reagent	Blank	Sample	P1-A2	10.0	0.100		0.1000		<0.10	<0.01				
05042021a_017.d	7.721	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				72.91						
05042021a_018.d	7.735	28857V0.01R7	DBA-ID181	Sample	P1-A3	10.0	0.100		0.1000	68.09	0.0994	0.0099				
05042021a_019.d	7.735	28857V0.01R7	DBA-ID181	Sample	P1-A3	10.0	0.100		0.1000	61.45	0.0902	0.0090	0.00948	95%		
05042021a_020.d	7.735	28857V0.01R8	DBA-ID181	Sample	P1-A4	10.0	0.100		0.1000	57.64	0.0849	0.0085				
05042021a_021.d	7.721	28857V0.01R8	DBA-ID181	Sample	P1-A4	10.0	0.100		0.1000	59.81	0.0880	0.0088	0.00864	86%		
05042021a_022.d	7.735	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				169.93						
05042021a_023.d	7.735	28857V0.01R9	DBA-ID181	Sample	P1-A5	10.0	0.100		0.1000	61.97	0.0909	0.0091				
05042021a_024.d	7.735	28857V0.01R9	DBA-ID181	Sample	P1-A5	10.0	0.100		0.1000	69.03	0.1007	0.0101	0.00958	96%	92%	5%
05042021a_025.d	7.721	28857V0.1R4	DBA-ID181	Sample	P1-A6	10.0	0.100		0.0250	161.09	0.2282	0.0913				
05042021a_026.d	7.721	28857V0.1R4	DBA-ID181	Sample	P1-A6	10.0	0.100		0.0250	172.63	0.2442	0.0977	0.0945	94%		
05042021a_027.d	7.735	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				67.88						
05042021a_028.d	7.735	28857V0.1R5	DBA-ID181	Sample	P1-A7	10.0	0.100		0.0250	161.83	0.2293	0.0917				
05042021a_029.d	7.721	28857V0.1R5	DBA-ID181	Sample	P1-A7	10.0	0.100		0.0250	168.72	0.2388	0.0955	0.0936	94%		
05042021a_030.d	7.735	28857V0.1R6	DBA-ID181	Sample	P1-A8	10.0	0.100		0.0250	168.27	0.2382	0.0953				
05042021a_031.d	7.735	28857V0.1R6	DBA-ID181	Sample	P1-A8	10.0	0.100		0.0250	166.70	0.2360	0.0944	0.0948	95%	94%	1%
05042021a_032.d	7.735	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				163.41						
05042021a_033.d	7.735	28857V1.0R4	DBA-ID181	Sample	P1-A9	10.0	0.100		0.0025	158.34	0.2244	0.8976				
05042021a_034.d	7.735	28857V1.0R4	DBA-ID181	Sample	P1-A9	10.0	0.100		0.0025	182.75	0.2582	1.0329	0.965	97%		
05042021a_035.d	7.735	28857V1.0R5	DBA-ID181	Sample	P1-B1	10.0	0.100		0.0025	165.00	0.2336	0.9346				
05042021a_036.d	7.735	28857V1.0R5	DBA-ID181	Sample	P1-B1	10.0	0.100		0.0025	174.69	0.2471	0.9883	0.961	96%		
05042021a_037.d	7.735	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				73.12						
05042021a_038.d	7.735	28857V1.0R6	DBA-ID181	Sample	P1-B2	10.0	0.100		0.0025	163.35	0.2314	0.9254				
05042021a_039.d	7.735	28857V1.0R6	DBA-ID181	Sample	P1-B2	10.0	0.100		0.0025	179.23	0.2534	1.0134	0.969	97%	97%	0%
05042021a_040.d	7.735	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				163.89						

Calculated LOD at 0.045 pg/µL = 29

Calculated LOQ at 0.10 pg/µL = 69

"-----" denotes response below area threshold



Calibration STD

Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05042021\05042021a_012.d	1	☒	718.72	1.0000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05042021\05042021a_011.d	2	☒	369.70	0.5000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05042021\05042021a_010.d	3	☒	176.93	0.2500
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05042021\05042021a_022.d	3	☒	169.93	0.2500
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05042021\05042021a_032.d	3	☒	163.41	0.2500
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05042021\05042021a_040.d	3	☒	163.89	0.2500
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05042021\05042021a_009.d	4	☒	73.08	0.1000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05042021\05042021a_017.d	4	☒	72.91	0.1000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05042021\05042021a_027.d	4	☒	67.88	0.1000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05042021\05042021a_037.d	4	☒	73.12	0.1000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05042021\05042021a_008.d	5	☒	39.73	0.0500

Compound
TFNA-AM

Injection Date
5/4/2021

08550 - Flonicamid/Onion - Method Validation - Dry Bulb - Extracted 5/4/2021 by ASM

5/5/21 *agm*

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec	Ave % Rec	Std Dev
05042021a_001.d	5.406	Condition		Sample	P1-A3	10.0										
05042021a_002.d	5.311	Condition		Sample	P1-A3	10.0										
05042021a_003.d	5.324	Condition		Sample	P1-A3	10.0										
05042021a_004.d	5.324	Condition		Sample	P1-A3	10.0										
05042021a_005.d	5.324	Condition		Sample	P1-A3	10.0										
05042021a_006.d	5.324	Condition		Sample	P1-A3	10.0										
05042021a_007.d	5.324	Condition		Sample	P1-A3	10.0										
05042021a_008.d	5.324	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				102.78						
05042021a_009.d	5.324	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				193.12						
05042021a_010.d	5.324	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				493.80						
05042021a_011.d	5.311	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				1002.01						
05042021a_012.d	5.297	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				1996.77						
05042021a_013.d	-----	28857	DBA-ID181	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05042021a_014.d	-----	28857	DBA-ID181	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01			
05042021a_015.d	-----	Reagent	Blank	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05042021a_016.d	-----	Reagent	Blank	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05042021a_017.d	5.324	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				191.24						
05042021a_018.d	5.324	28857V0.01R7	DBA-ID181	Sample	P1-A3	10.0	0.100	10.0	0.1000	176.92	0.1055	0.0106				
05042021a_019.d	5.324	28857V0.01R7	DBA-ID181	Sample	P1-A3	10.0	0.100	10.0	0.1000	171.34	0.1028	0.0103	0.01041	104%		
05042021a_020.d	5.324	28857V0.01R8	DBA-ID181	Sample	P1-A4	10.0	0.100	10.0	0.1000	161.74	0.0980	0.0098				
05042021a_021.d	5.324	28857V0.01R8	DBA-ID181	Sample	P1-A4	10.0	0.100	10.0	0.1000	148.76	0.0916	0.0092	0.00948	95%		
05042021a_022.d	5.324	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				403.15						
05042021a_023.d	5.324	28857V0.01R9	DBA-ID181	Sample	P1-A5	10.0	0.100	10.0	0.1000	148.70	0.0916	0.0092				
05042021a_024.d	5.324	28857V0.01R9	DBA-ID181	Sample	P1-A5	10.0	0.100	10.0	0.1000	158.50	0.0964	0.0096	0.00940	94%	98%	6%
05042021a_025.d	5.324	28857V0.1R4	DBA-ID181	Sample	P1-A6	10.0	0.100	40.0	0.0250	423.98	0.2278	0.0911				
05042021a_026.d	5.324	28857V0.1R4	DBA-ID181	Sample	P1-A6	10.0	0.100	40.0	0.0250	413.55	0.2226	0.0890	0.0901	90%		
05042021a_027.d	5.324	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				157.93						
05042021a_028.d	5.324	28857V0.1R5	DBA-ID181	Sample	P1-A7	10.0	0.100	40.0	0.0250	414.24	0.2230	0.0892				
05042021a_029.d	5.324	28857V0.1R5	DBA-ID181	Sample	P1-A7	10.0	0.100	40.0	0.0250	428.59	0.2301	0.0920	0.0906	91%		
05042021a_030.d	5.324	28857V0.1R6	DBA-ID181	Sample	P1-A8	10.0	0.100	40.0	0.0250	418.60	0.2251	0.0900				
05042021a_031.d	5.324	28857V0.1R6	DBA-ID181	Sample	P1-A8	10.0	0.100	40.0	0.0250	422.43	0.2270	0.0908	0.0904	90%	90%	0%
05042021a_032.d	5.324	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				421.09						
05042021a_033.d	5.324	28857V1.0R4	DBA-ID181	Sample	P1-A9	10.0	0.100	400.0	0.0025	437.06	0.2342	0.9370				
05042021a_034.d	5.324	28857V1.0R4	DBA-ID181	Sample	P1-A9	10.0	0.100	400.0	0.0025	435.25	0.2334	0.9334	0.935	94%		
05042021a_035.d	5.324	28857V1.0R5	DBA-ID181	Sample	P1-B1	10.0	0.100	400.0	0.0025	431.43	0.2315	0.9259				
05042021a_036.d	5.324	28857V1.0R5	DBA-ID181	Sample	P1-B1	10.0	0.100	400.0	0.0025	449.39	0.2403	0.9614	0.944	94%		
05042021a_037.d	5.324	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				177.42						
05042021a_038.d	5.324	28857V1.0R6	DBA-ID181	Sample	P1-B2	10.0	0.100	400.0	0.0025	428.56	0.2300	0.9202				
05042021a_039.d	5.324	28857V1.0R6	DBA-ID181	Sample	P1-B2	10.0	0.100	400.0	0.0025	439.86	0.2356	0.9425	0.931	93%	94%	1%
05042021a_040.d	5.324	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				422.69						

Calculated LOD at 0.045 pg/µL = 55

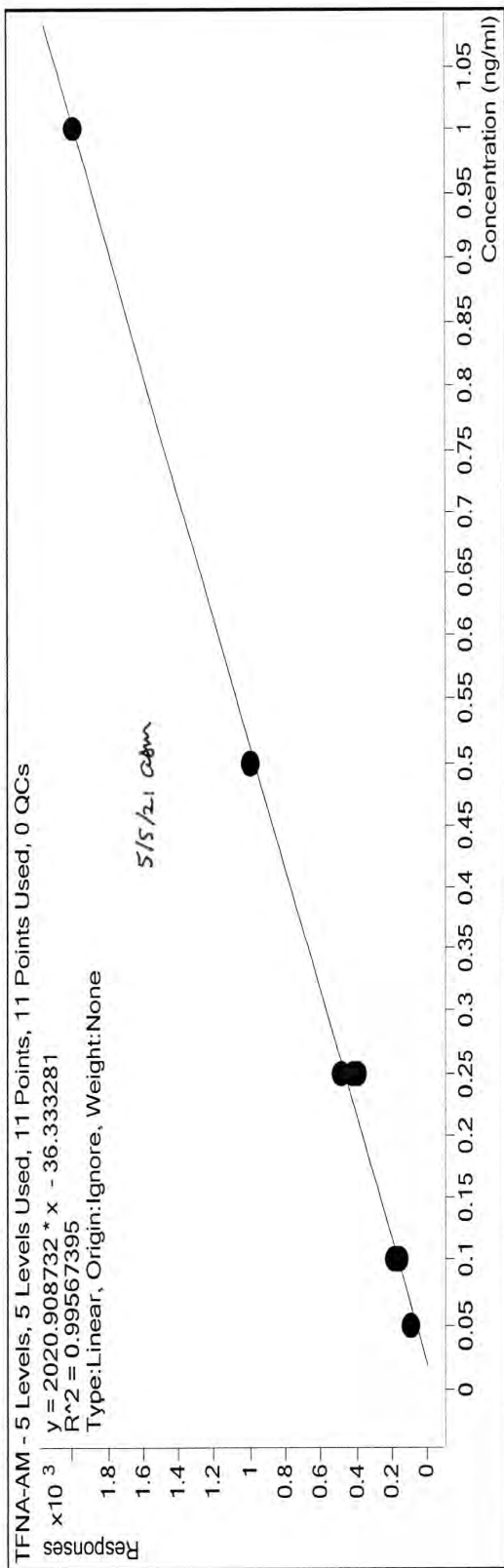
Calculated LOQ at 0.10 pg/µL = 166

"-----" denotes response below area threshold

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05042021\QuantResults\05042021a_batch.bin

Batch Data Path**Calibration Info**

Target Compound **TFNA-AM** CurveFit **fitLinear** Weighting **weightEqual** Integrator **Agile** Smoothing **Gaussian** SmoothingFunctionWidth **10** SmoothingGaussianWidth **5**

**Calibration STD**

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05042021\05042021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05042021\05042021a_011.d	Calibration	1	☒	1996.77	1.0000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05042021\05042021a_010.d	Calibration	2	☒	1002.01	0.5000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05042021\05042021a_022.d	Calibration	3	☒	493.80	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05042021\05042021a_032.d	Calibration	3	☒	403.15	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05042021\05042021a_040.d	Calibration	3	☒	421.09	0.2500
D:\MassHunter\Flonicamid\08550_Onion>Data\Results\05042021\05042021a_009.d	Calibration	3	☒	422.69	0.2500
D:\MassHunter\Flonicamid\08550_Onion>Data\Results\05042021\05042021a_017.d	Calibration	4	☒	193.12	0.1000
D:\MassHunter\Flonicamid\08550_Onion>Data\Results\05042021\05042021a_027.d	Calibration	4	☒	191.24	0.1000
D:\MassHunter\Flonicamid\08550_Onion>Data\Results\05042021\05042021a_037.d	Calibration	4	☒	157.93	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05042021\05042021a_008.d	Calibration	4	☒	177.42	0.1000
	Calibration	5	☒	102.78	0.0500

Compound Injection Date
TFNA 5/4/2021

08550 - Flonicamid/Onion - Method Validation - Dry Bulb - Extracted 5/4/2021 by ASM

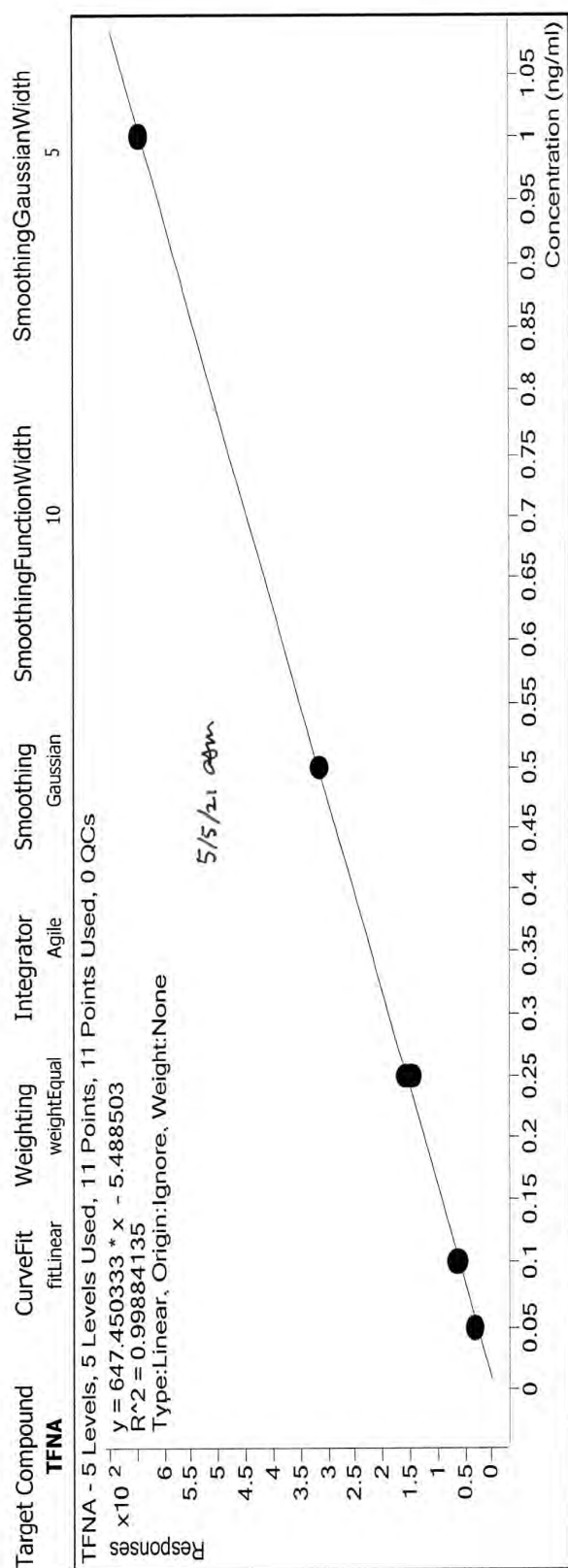
5/5/21 *asm*

Sample	Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	% Rec	Ave % Rec	Std Dev
Sample	Sample	P1-A3	10.0									
Sample	Sample	P1-A3	10.0									
Sample	Sample	P1-A3	10.0									
Sample	Sample	P1-A3	10.0									
Sample	Sample	P1-A3	10.0									
Sample	Sample	P1-A3	10.0									
Sample	Sample	P1-A3	10.0									
Calibration	Calibration	Vial 5	10.0				33.64					
Calibration	Calibration	Vial 4	10.0				60.43					
Calibration	Calibration	Vial 3	10.0				157.78					
Calibration	Calibration	Vial 2	10.0				315.14					
Calibration	Calibration	Vial 1	10.0				647.32					
Sample	DBA-ID181	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01			
Sample	DBA-ID181	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01			
Sample	Blank	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01			
Sample	Blank	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01			
Calibration	Calibration	Vial 4	10.0				67.13					
Sample	DBA-ID181	P1-A3	10.0	0.100	10.0	0.1000	57.43	0.0972	0.0097			
Sample	DBA-ID181	P1-A3	10.0	0.100	10.0	0.1000	57.02	0.0965	0.0097	97%		
Sample	DBA-ID181	P1-A4	10.0	0.100	10.0	0.1000	58.69	0.0991	0.0099			
Sample	DBA-ID181	P1-A4	10.0	0.100	10.0	0.1000	47.60	0.0820	0.00906	91%		
Calibration	Calibration	Vial 3	10.0				144.49					
Sample	DBA-ID181	P1-A5	10.0	0.100	10.0	0.1000	53.98	0.0919	0.0092			
Sample	DBA-ID181	P1-A5	10.0	0.100	10.0	0.1000	53.89	0.0917	0.0092	92%	93%	3%
Sample	DBA-ID181	P1-A6	10.0	0.100	40.0	0.0250	146.29	0.2344	0.0938			
Sample	DBA-ID181	P1-A6	10.0	0.100	40.0	0.0250	135.94	0.2184	0.0874	91%		
Calibration	Calibration	Vial 4	10.0				59.56					
Sample	DBA-ID181	P1-A7	10.0	0.100	40.0	0.0250	146.07	0.2341	0.0936			
Sample	DBA-ID181	P1-A7	10.0	0.100	40.0	0.0250	148.37	0.2376	0.0951	94%		
Sample	DBA-ID181	P1-A8	10.0	0.100	40.0	0.0250	146.86	0.2353	0.0941			
Sample	DBA-ID181	P1-A8	10.0	0.100	40.0	0.0250	141.37	0.2268	0.0907	92%	92%	2%
Calibration	Calibration	Vial 3	10.0				151.58					
Sample	DBA-ID181	P1-A9	10.0	0.100	400.0	0.0025	140.39	0.2253	0.9012			
Sample	DBA-ID181	P1-A9	10.0	0.100	400.0	0.0025	149.30	0.2391	0.9563	93%		
Sample	DBA-ID181	P1-B1	10.0	0.100	400.0	0.0025	152.62	0.2442	0.9768			
Sample	DBA-ID181	P1-B1	10.0	0.100	400.0	0.0025	135.01	0.2170	0.8680	92%		
Calibration	Calibration	Vial 4	10.0				62.55					
Sample	DBA-ID181	P1-B2	10.0	0.100	400.0	0.0025	147.36	0.2361	0.9443			
Sample	DBA-ID181	P1-B2	10.0	0.100	400.0	0.0025	148.72	0.2382	0.9527	95%	93%	1%
Calibration	Calibration	Vial 3	10.0				149.97					

Calculated LOD at 0.045 pg/µL = 24

Calculated LOQ at 0.10 pg/µL = 59

"----" denotes response below area threshold



Calibration STD

D:\MassHunter\Fionicaid\08550_Onion\Data\Results\05042021\05042021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Fionicaid\08550_Onion\Data\Results\05042021\05042021a_011.d	Calibration	1	☒	647.32	1.0000
D:\MassHunter\Fionicaid\08550_Onion\Data\Results\05042021\05042021a_010.d	Calibration	2	☒	315.14	0.5000
D:\MassHunter\Fionicaid\08550_Onion\Data\Results\05042021\05042021a_010.d	Calibration	3	☒	157.78	0.2500
D:\MassHunter\Fionicaid\08550_Onion\Data\Results\05042021\05042021a_022.d	Calibration	3	☒	144.49	0.2500
D:\MassHunter\Fionicaid\08550_Onion\Data\Results\05042021\05042021a_032.d	Calibration	3	☒	151.58	0.2500
D:\MassHunter\Fionicaid\08550_Onion\Data\Results\05042021\05042021a_040.d	Calibration	3	☒	149.97	0.2500
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D:\MassHunter\Fionicaid\08550_Onion\Data\Results\05042021\05042021a_027.d	Calibration	4	☒	59.56	0.1000
D:\MassHunter\Fionicaid\08550_Onion\Data\Results\05042021\05042021a_037.d	Calibration	4	☒	62.55	0.1000
D:\MassHunter\Fionicaid\08550_Onion\Data\Results\05042021\05042021a_008.d	Calibration	5	☒	33.64	0.0500

Compound	Injection Date
TFNG	5/4/2021

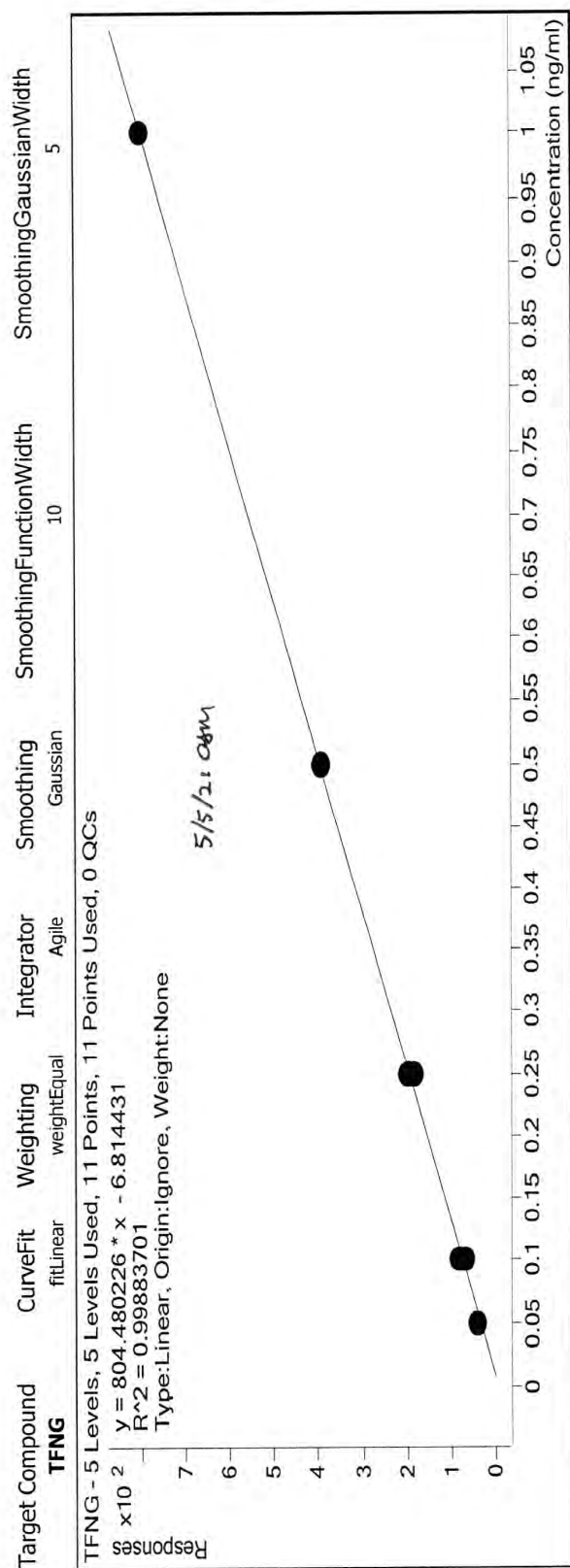
TFNG 5/4/2021

08550 - Flonicamid/Onion - Method Validation - Dry Bulb - Extracted 5/4/2021 by ASM

5/5/21 am

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec	Ave % Rec	Std Dev
05042021a_001.d	7.321	Condition		Sample	P1-A3	10.0										
05042021a_002.d	7.212	Condition		Sample	P1-A3	10.0										
05042021a_003.d	7.212	Condition		Sample	P1-A3	10.0										
05042021a_004.d	7.225	Condition		Sample	P1-A3	10.0										
05042021a_005.d	7.212	Condition		Sample	P1-A3	10.0										
05042021a_006.d	7.212	Condition		Sample	P1-A3	10.0										
05042021a_007.d	7.225	Condition		Sample	P1-A3	10.0										
05042021a_008.d	7.225	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				43.87						
05042021a_009.d	7.212	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				81.09						
05042021a_010.d	7.212	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				196.76						
05042021a_011.d	7.212	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				393.18						
05042021a_012.d	7.198	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				803.06						
05042021a_013.d	-----	28857	DBA-ID181	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05042021a_014.d	-----	28857	DBA-ID181	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01			
05042021a_015.d	-----	Reagent	Blank	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05042021a_016.d	-----	Reagent	Blank	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05042021a_017.d	7.225	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				78.98						
05042021a_018.d	7.212	28857V0.01R7	DBA-ID181	Sample	P1-A3	10.0	0.100	10.0	0.1000	79.87	0.1078	0.0108				
05042021a_019.d	7.225	28857V0.01R7	DBA-ID181	Sample	P1-A3	10.0	0.100	10.0	0.1000	71.60	0.0975	0.0097	0.0103	103%		
05042021a_020.d	7.212	28857V0.01R8	DBA-ID181	Sample	P1-A4	10.0	0.100	10.0	0.1000	74.74	0.1014	0.0101				
05042021a_021.d	7.212	28857V0.01R8	DBA-ID181	Sample	P1-A4	10.0	0.100	10.0	0.1000	72.86	0.0990	0.0099	0.0100	100%		
05042021a_022.d	7.212	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				181.79						
05042021a_023.d	7.225	28857V0.01R9	DBA-ID181	Sample	P1-A5	10.0	0.100	10.0	0.1000	67.00	0.0918	0.0092				
05042021a_024.d	7.225	28857V0.01R9	DBA-ID181	Sample	P1-A5	10.0	0.100	10.0	0.1000	73.14	0.0994	0.0099	0.00956	96%	99%	4%
05042021a_025.d	7.212	28857V0.1R4	DBA-ID181	Sample	P1-A6	10.0	0.100	40.0	0.0250	194.35	0.2501	0.1000				
05042021a_026.d	7.225	28857V0.1R4	DBA-ID181	Sample	P1-A6	10.0	0.100	40.0	0.0250	201.38	0.2588	0.1035	0.102	102%		
05042021a_027.d	7.225	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				68.14						
05042021a_028.d	7.225	28857V0.1R5	DBA-ID181	Sample	P1-A7	10.0	0.100	40.0	0.0250	196.69	0.2530	0.1012				
05042021a_029.d	7.225	28857V0.1R5	DBA-ID181	Sample	P1-A7	10.0	0.100	40.0	0.0250	185.68	0.2393	0.0957	0.0984	98%		
05042021a_030.d	7.225	28857V0.1R6	DBA-ID181	Sample	P1-A8	10.0	0.100	40.0	0.0250	199.05	0.2559	0.1024				
05042021a_031.d	7.225	28857V0.1R6	DBA-ID181	Sample	P1-A8	10.0	0.100	40.0	0.0250	183.92	0.2371	0.0948	0.0986	95%	100%	2%
05042021a_032.d	7.212	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				182.45						
05042021a_033.d	7.212	28857V1.0R4	DBA-ID181	Sample	P1-A9	10.0	0.100	400.0	0.0025	201.45	0.2589	1.0355				
05042021a_034.d	7.225	28857V1.0R4	DBA-ID181	Sample	P1-A9	10.0	0.100	400.0	0.0025	210.21	0.2698	1.0791	1.06	106%		
05042021a_035.d	7.212	28857V1.0R5	DBA-ID181	Sample	P1-B1	10.0	0.100	400.0	0.0025	218.04	0.2795	1.1180				
05042021a_036.d	7.225	28857V1.0R5	DBA-ID181	Sample	P1-B1	10.0	0.100	400.0	0.0025	209.30	0.2686	1.0745	1.10	110%		
05042021a_037.d	7.212	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				75.62						
05042021a_038.d	7.212	28857V1.0R6	DBA-ID181	Sample	P1-B2	10.0	0.100	400.0	0.0025	205.87	0.2644	1.0575				
05042021a_039.d	7.225	28857V1.0R6	DBA-ID181	Sample	P1-B2	10.0	0.100	400.0	0.0025	217.83	0.2792	1.1170	1.09	109%	108%	2%
05042021a_040.d	7.225	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				193.30						
Calculated LOD at 0.045 pg/µL = 29																
Calculated LOQ at 0.10 pg/µL = 74																
-----" denotes response below area threshold																

Printed at: 9:43 AM on: 5/5/2021

**Calibration STD**

D:\MassHunter\FI\Fluorimetry\08550_012.d	Calibration	Level 1	Enabled ☒	Response 803.06	Exp Conc 1.0000
D:\MassHunter\FI\Fluorimetry\08550_011.d	Calibration	Level 2	Enabled ☒	Response 393.18	Exp Conc 0.5000
D:\MassHunter\FI\Fluorimetry\08550_010.d	Calibration	Level 3	Enabled ☒	Response 196.76	Exp Conc 0.2500
D:\MassHunter\FI\Fluorimetry\08550_022.d	Calibration	Level 3	Enabled ☒	Response 181.79	Exp Conc 0.2500
D:\MassHunter\FI\Fluorimetry\08550_032.d	Calibration	Level 3	Enabled ☒	Response 182.45	Exp Conc 0.2500
D:\MassHunter\FI\Fluorimetry\08550_040.d	Calibration	Level 3	Enabled ☒	Response 193.30	Exp Conc 0.2500
D:\MassHunter\FI\Fluorimetry\08550_009.d	Calibration	Level 4	Enabled ☒	Response 81.09	Exp Conc 0.1000
D:\MassHunter\FI\Fluorimetry\08550_017.d	Calibration	Level 4	Enabled ☒	Response 78.98	Exp Conc 0.1000
D:\MassHunter\FI\Fluorimetry\08550_027.d	Calibration	Level 4	Enabled ☒	Response 68.14	Exp Conc 0.1000
D:\MassHunter\FI\Fluorimetry\08550_037.d	Calibration	Level 4	Enabled ☒	Response 75.62	Exp Conc 0.1000
D:\MassHunter\FI\Fluorimetry\08550_008.d	Calibration	Level 5	Enabled ☒	Response 43.87	Exp Conc 0.0500

Compound
08550 - Flonicamid/Onion - Method Validation (Green Onion) - Extracted 5/14/21 by ASM

5/15/21 *asm*

Injection Date
5/14/2021

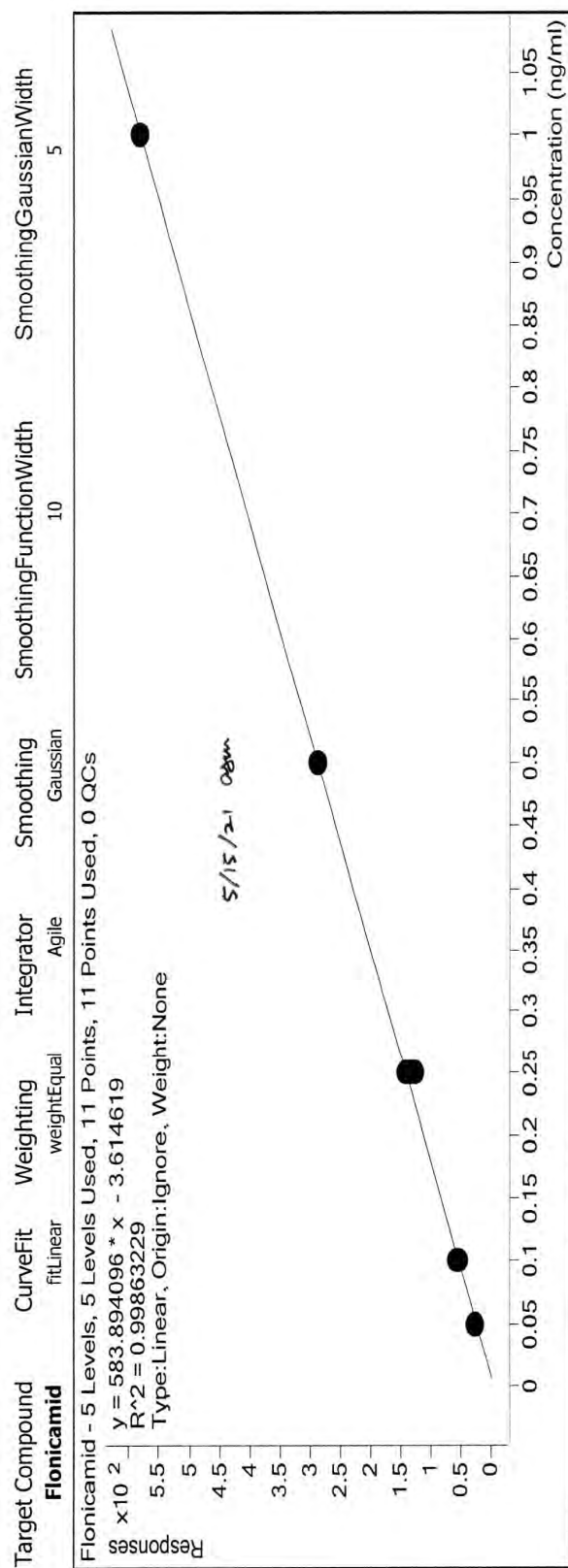
08550 - Flonicamid/Onion - Method Validation (Green Onion) - Extracted 5/14/21 by ASM

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec	Ave % Rec	Std Dev
05142021a_001.d	7.721	Condition		Sample	P1-A3	10.0										
05142021a_002.d	7.735	Condition		Sample	P1-A3	10.0										
05142021a_003.d	7.735	Condition		Sample	P1-A3	10.0										
05142021a_004.d	7.735	Condition		Sample	P1-A3	10.0										
05142021a_005.d	7.721	Condition		Sample	P1-A3	10.0										
05142021a_006.d	7.721	Condition		Sample	P1-A3	10.0										
05142021a_007.d	7.721	Condition		Sample	P1-A3	10.0										
05142021a_008.d	7.721	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				29.83						
05142021a_009.d	7.721	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				57.95						
05142021a_010.d	7.735	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				144.28						
05142021a_011.d	7.721	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				287.04						
05142021a_012.d	7.721	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				584.34						
05142021a_013.d	7.735	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05142021a_014.d	7.735	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05142021a_015.d	7.735	Reagent	Blank	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05142021a_016.d	7.735	Reagent	Blank	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05142021a_017.d	7.735	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				59.27						
05142021a_018.d	7.735	28849V0.01R1	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	51.04	0.0936	0.0094				
05142021a_019.d	7.735	28849V0.01R1	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	53.86	0.0984	0.0098	96%			
05142021a_020.d	7.735	28849V0.01R2	GA-CA*20	Sample	P1-A4	10.0	0.100	10.0	0.1000	50.05	0.0919	0.0092				
05142021a_021.d	7.735	28849V0.01R2	GA-CA*20	Sample	P1-A4	10.0	0.100	10.0	0.1000	50.56	0.0928	0.0093	92%			
05142021a_022.d	7.721	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				127.88						
05142021a_023.d	7.735	28849V0.01R3	GA-CA*20	Sample	P1-A5	10.0	0.100	10.0	0.1000	53.57	0.0979	0.0098				
05142021a_024.d	7.735	28849V0.01R3	GA-CA*20	Sample	P1-A5	10.0	0.100	10.0	0.1000	45.38	0.0839	0.0084	91%	93%		3%
05142021a_025.d	7.735	28849V0.1R1	GA-CA*20	Sample	P1-A6	10.0	0.100	40.0	0.0250	130.61	0.2299	0.0919				
05142021a_026.d	7.748	28849V0.1R1	GA-CA*20	Sample	P1-A6	10.0	0.100	40.0	0.0250	122.12	0.2153	0.0861				
05142021a_027.d	7.735	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				57.98						
05142021a_028.d	7.735	28849V0.1R2	GA-CA*20	Sample	P1-A7	10.0	0.100	40.0	0.0250	127.57	0.2247	0.0899				
05142021a_029.d	7.735	28849V0.1R2	GA-CA*20	Sample	P1-A7	10.0	0.100	40.0	0.0250	126.32	0.2225	0.0890				
05142021a_030.d	7.735	28849V0.1R3	GA-CA*20	Sample	P1-A8	10.0	0.100	40.0	0.0250	130.96	0.2305	0.0922				
05142021a_031.d	7.735	28849V0.1R3	GA-CA*20	Sample	P1-A8	10.0	0.100	40.0	0.0250	131.94	0.2322	0.0929	93%	90%		2%
05142021a_032.d	7.721	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				134.77						
05142021a_033.d	7.735	28849V1.0R1	GA-CA*20	Sample	P1-A9	10.0	0.100	400.0	0.0025	139.61	0.2453	0.9812				
05142021a_034.d	7.735	28849V1.0R1	GA-CA*20	Sample	P1-A9	10.0	0.100	400.0	0.0025	142.23	0.2498	0.9991				
05142021a_035.d	7.735	28849V1.0R2	GA-CA*20	Sample	P1-B1	10.0	0.100	400.0	0.0025	145.06	0.2546	1.0185				
05142021a_036.d	7.748	28849V1.0R2	GA-CA*20	Sample	P1-B1	10.0	0.100	400.0	0.0025	134.03	0.2357	0.9429				
05142021a_037.d	7.735	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				56.39						
05142021a_038.d	7.735	28849V1.0R3	GA-CA*20	Sample	P1-B2	10.0	0.100	400.0	0.0025	142.57	0.2504	1.0014				
05142021a_039.d	7.735	28849V1.0R3	GA-CA*20	Sample	P1-B2	10.0	0.100	400.0	0.0025	138.84	0.2440	0.9759				
05142021a_040.d	7.735	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				143.01						

Calculated LOD at 0.045 pg/µL =

Calculated LOQ at 0.10 pg/µL =

*----- denotes response below area threshold



Calibration STD

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_011.d	Calibration	1	☒	584.34	1.0000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_010.d	Calibration	2	☒	287.04	0.5000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_022.d	Calibration	3	☒	144.28	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_032.d	Calibration	3	☒	127.88	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_040.d	Calibration	3	☒	134.77	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_009.d	Calibration	4	☒	143.01	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_017.d	Calibration	4	☒	57.95	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_027.d	Calibration	4	☒	59.27	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_037.d	Calibration	4	☒	57.98	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_008.d	Calibration	5	☒	56.39	0.1000
				29.83	0.0500

Compound Injection Date

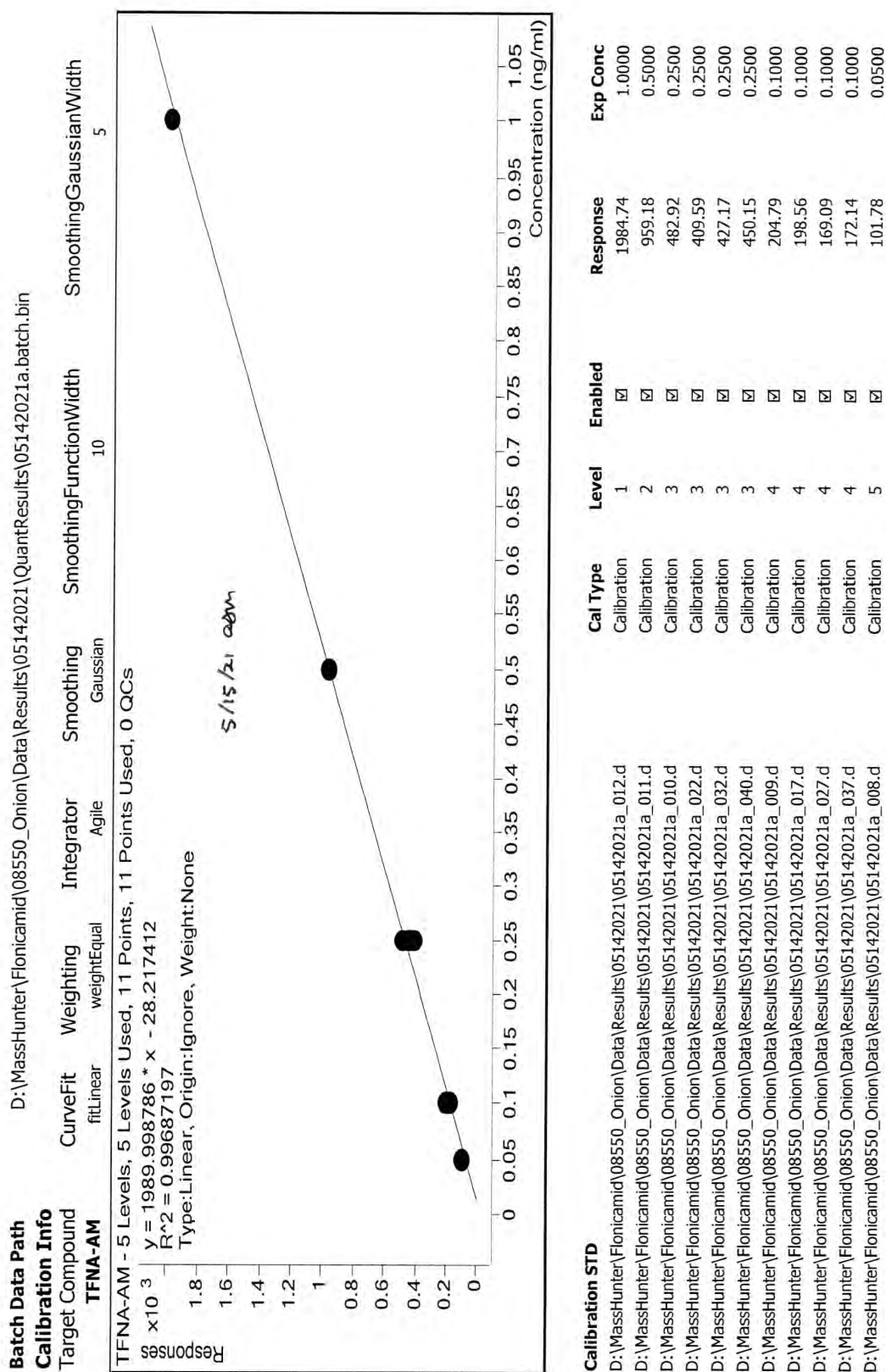
TFNA-AM 5/14/2021

08550 - Fonicamid/Onion - Method Validation (Green Onion) - Extracted 5/14/21 by ASM

5/15/21 asm

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec	Ave % Rec	Std Dev
05142021a_001.d	-----	Condition		Sample	P1-A3	10.0										
05142021a_002.d	5.311	Condition		Sample	P1-A3	10.0										
05142021a_003.d	5.324	Condition		Sample	P1-A3	10.0										
05142021a_004.d	5.324	Condition		Sample	P1-A3	10.0										
05142021a_005.d	5.324	Condition		Sample	P1-A3	10.0										
05142021a_006.d	5.311	Condition		Sample	P1-A3	10.0										
05142021a_007.d	5.311	Condition		Sample	P1-A3	10.0										
05142021a_008.d	5.311	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				101.78						
05142021a_009.d	5.311	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				204.79						
05142021a_010.d	5.324	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				482.92						
05142021a_011.d	5.311	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				959.18						
05142021a_012.d	5.297	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				1984.74						
05142021a_013.d	-----	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05142021a_014.d	-----	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05142021a_015.d	-----	Reagent	Blank	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05142021a_016.d	-----	Reagent	Blank	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05142021a_017.d	5.324	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				198.56						
05142021a_018.d	5.324	28849V0.01R1	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	183.84	0.1066	0.0107				
05142021a_019.d	5.311	28849V0.01R1	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	180.06	0.1047	0.0105	0.0106	106%		
05142021a_020.d	5.324	28849V0.01R2	GA-CA*20	Sample	P1-A4	10.0	0.100	10.0	0.1000	171.81	0.1005	0.0101				
05142021a_021.d	5.324	28849V0.01R2	GA-CA*20	Sample	P1-A4	10.0	0.100	10.0	0.1000	155.72	0.0924	0.0092	0.00965	96%		
05142021a_022.d	5.311	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				409.59						
05142021a_023.d	5.324	28849V0.01R3	GA-CA*20	Sample	P1-A5	10.0	0.100	10.0	0.1000	149.21	0.0892	0.0089				
05142021a_024.d	5.324	28849V0.01R3	GA-CA*20	Sample	P1-A5	10.0	0.100	10.0	0.1000	154.41	0.0918	0.0092	0.00905	90%	98%	8%
05142021a_025.d	5.324	28849V0.1R1	GA-CA*20	Sample	P1-A6	10.0	0.100	40.0	0.0250	389.92	0.2101	0.0840				
05142021a_026.d	5.338	28849V0.1R1	GA-CA*20	Sample	P1-A6	10.0	0.100	40.0	0.0250	387.79	0.2090	0.0836	0.0838	84%		
05142021a_027.d	5.324	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				169.09						
05142021a_028.d	5.324	28849V0.1R2	GA-CA*20	Sample	P1-A7	10.0	0.100	40.0	0.0250	406.87	0.2186	0.0875				
05142021a_029.d	5.324	28849V0.1R2	GA-CA*20	Sample	P1-A7	10.0	0.100	40.0	0.0250	402.63	0.2165	0.0866	0.0870	87%		
05142021a_030.d	5.324	28849V0.1R3	GA-CA*20	Sample	P1-A8	10.0	0.100	40.0	0.0250	403.71	0.2171	0.0868				
05142021a_031.d	5.324	28849V0.1R3	GA-CA*20	Sample	P1-A8	10.0	0.100	40.0	0.0250	427.48	0.2290	0.0916	0.0892	89%	87%	3%
05142021a_032.d	5.311	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				427.17						
05142021a_033.d	5.324	28849V1.0R1	GA-CA*20	Sample	P1-A9	10.0	0.100	400.0	0.0025	437.60	0.2341	0.9363				
05142021a_034.d	5.324	28849V1.0R1	GA-CA*20	Sample	P1-A9	10.0	0.100	400.0	0.0025	434.80	0.2327	0.9307	0.934	93%		
05142021a_035.d	5.324	28849V1.0R2	GA-CA*20	Sample	P1-B1	10.0	0.100	400.0	0.0025	455.33	0.2430	0.9719				
05142021a_036.d	5.338	28849V1.0R2	GA-CA*20	Sample	P1-B1	10.0	0.100	400.0	0.0025	441.68	0.2361	0.9445	0.958	96%		
05142021a_037.d	5.324	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				172.14						
05142021a_038.d	5.338	28849V1.0R3	GA-CA*20	Sample	P1-B2	10.0	0.100	400.0	0.0025	455.17	0.2429	0.9716				
05142021a_039.d	5.324	28849V1.0R3	GA-CA*20	Sample	P1-B2	10.0	0.100	400.0	0.0025	436.71	0.2336	0.9345	0.953	95%	95%	1%
05142021a_040.d	5.324	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				450.15						
Calculated LOD at 0.045 pg/µL = 61																
Calculated LOQ at 0.10 pg/µL = 171																
"-----" denotes response below area threshold																

Printed at: 1:11 PM on: 5/15/2021



Compound Injection Date
TFNA 5/14/2021

08550 - Flonicamid/Onion - Method Validation (Green Onion) - Extracted 5/14/21 by ASM

5/15/21 *asm*

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp (pg/µL)	Conc (pg/µL)	ppm	Ave ppm	% Rec	Ave % Rec	Std Dev
05142021a_001.d	-----	Condition		Sample	P1-A3	10.0										
05142021a_002.d	6.010	Condition		Sample	P1-A3	10.0										
05142021a_003.d	6.010	Condition		Sample	P1-A3	10.0										
05142021a_004.d	6.024	Condition		Sample	P1-A3	10.0										
05142021a_005.d	6.010	Condition		Sample	P1-A3	10.0										
05142021a_006.d	5.997	Condition		Sample	P1-A3	10.0										
05142021a_007.d	6.010	Condition		Sample	P1-A3	10.0										
05142021a_008.d	5.983	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				33.20						
05142021a_009.d	5.983	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				61.67						
05142021a_010.d	5.997	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				166.08						
05142021a_011.d	5.983	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				319.08						
05142021a_012.d	5.969	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				637.36						
05142021a_013.d	-----	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01				
05142021a_014.d	-----	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01			
05142021a_015.d	-----	Reagent	Blank	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01				
05142021a_016.d	-----	Reagent	Blank	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01				
05142021a_017.d	5.997	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				64.06						
05142021a_018.d	6.024	28849V0.01R1	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	66.87	0.1095	0.0110				
05142021a_019.d	6.010	28849V0.01R1	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	59.48	0.0980	0.0098	0.0104	104%		
05142021a_020.d	6.010	28849V0.01R2	GA-CA*20	Sample	P1-A4	10.0	0.100	10.0	0.1000	53.66	0.0889	0.0089				
05142021a_021.d	6.024	28849V0.01R2	GA-CA*20	Sample	P1-A4	10.0	0.100	10.0	0.1000	53.99	0.0894	0.0089	0.00891	89%		
05142021a_022.d	5.983	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				147.46						
05142021a_023.d	6.024	28849V0.01R3	GA-CA*20	Sample	P1-A5	10.0	0.100	10.0	0.1000	59.19	0.0975	0.0098				
05142021a_024.d	6.010	28849V0.01R3	GA-CA*20	Sample	P1-A5	10.0	0.100	10.0	0.1000	58.69	0.0967	0.0097	0.00971	97%	97%	7%
05142021a_025.d	6.010	28849V0.1R1	GA-CA*20	Sample	P1-A6	10.0	0.100	40.0	0.0250	136.17	0.2177	0.0871				
05142021a_026.d	6.024	28849V0.1R1	GA-CA*20	Sample	P1-A6	10.0	0.100	40.0	0.0250	145.42	0.2322	0.0929	0.0900	90%		
05142021a_027.d	5.997	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				54.48						
05142021a_028.d	6.010	28849V0.1R2	GA-CA*20	Sample	P1-A7	10.0	0.100	40.0	0.0250	135.57	0.2168	0.0867				
05142021a_029.d	6.010	28849V0.1R2	GA-CA*20	Sample	P1-A7	10.0	0.100	40.0	0.0250	133.61	0.2137	0.0855	0.0861	86%		
05142021a_030.d	6.010	28849V0.1R3	GA-CA*20	Sample	P1-A8	10.0	0.100	40.0	0.0250	137.95	0.2205	0.0882				
05142021a_031.d	6.024	28849V0.1R3	GA-CA*20	Sample	P1-A8	10.0	0.100	40.0	0.0250	147.76	0.2359	0.0943	0.0913	91%	89%	3%
05142021a_032.d	5.983	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				158.79						
05142021a_033.d	6.010	28849V1.0R1	GA-CA*20	Sample	P1-A9	10.0	0.100	400.0	0.0025	148.01	0.2362	0.9450				
05142021a_034.d	5.997	28849V1.0R1	GA-CA*20	Sample	P1-A9	10.0	0.100	400.0	0.0025	149.00	0.2378	0.9512	0.948	95%		
05142021a_035.d	6.010	28849V1.0R2	GA-CA*20	Sample	P1-B1	10.0	0.100	400.0	0.0025	157.93	0.2517	1.0070				
05142021a_036.d	6.010	28849V1.0R2	GA-CA*20	Sample	P1-B1	10.0	0.100	400.0	0.0025	146.28	0.2335	0.9341	0.971	97%		
05142021a_037.d	5.997	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				62.24						
05142021a_038.d	6.010	28849V1.0R3	GA-CA*20	Sample	P1-B2	10.0	0.100	400.0	0.0025	150.39	0.2400	0.9598				
05142021a_039.d	6.010	28849V1.0R3	GA-CA*20	Sample	P1-B2	10.0	0.100	400.0	0.0025	150.04	0.2394	0.9576	0.959	96%	96%	1%
05142021a_040.d	5.997	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				148.66						

26

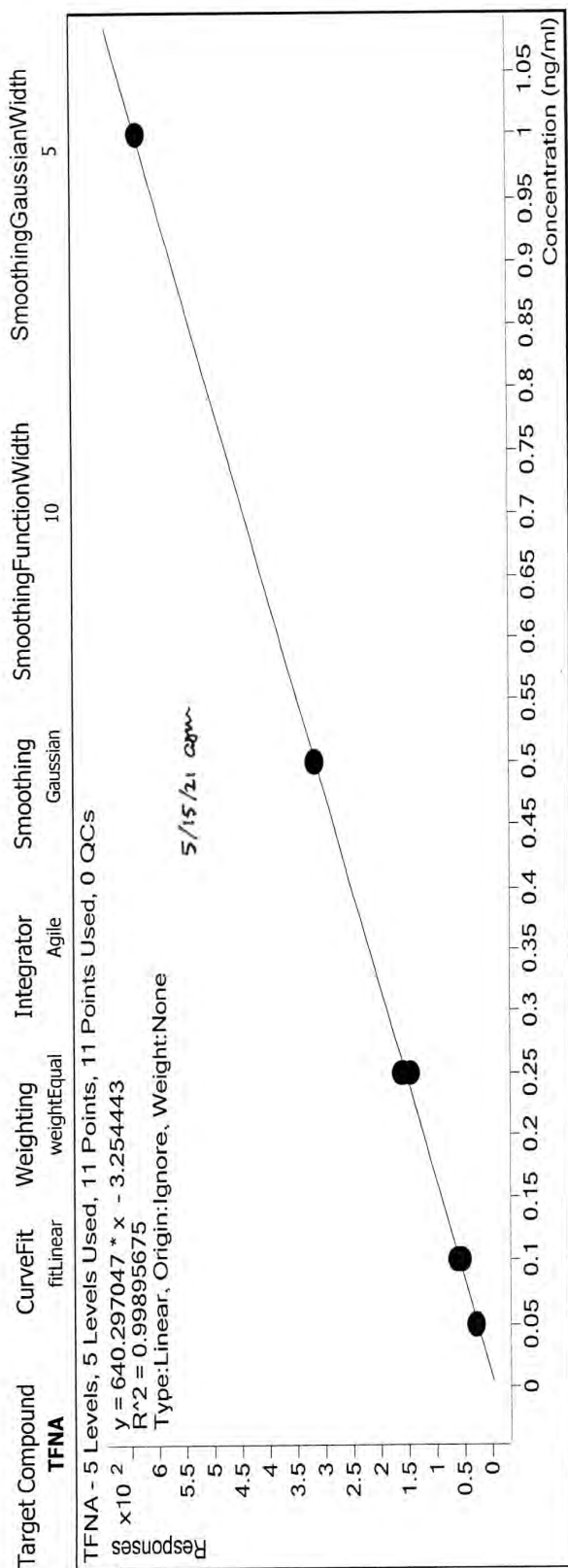
61

Calculated LOD at 0.045 pg/µL =

Calculated LOQ at 0.10 pg/µL =

"-----" denotes response below area threshold

Printed at: 1:11 PM on: 5/15/2021



Calibration STD

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_011.d	Calibration	1	☒	637.36	1.0000
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D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_010.d	Calibration	3	☒	166.08	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_022.d	Calibration	3	☒	147.46	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_032.d	Calibration	3	☒	158.79	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_040.d	Calibration	3	☒	148.66	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_009.d	Calibration	4	☒	61.67	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_017.d	Calibration	4	☒	64.06	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_027.d	Calibration	4	☒	54.48	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_037.d	Calibration	4	☒	62.24	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_008.d	Calibration	5	☒	33.20	0.0500

Compound Injection Date
TFNG 5/14/2021

08550 - Flonicamid/Onion - Method Validation (Green Onion) - Extracted 5/14/21 by ASM

5/15/21 *asm*

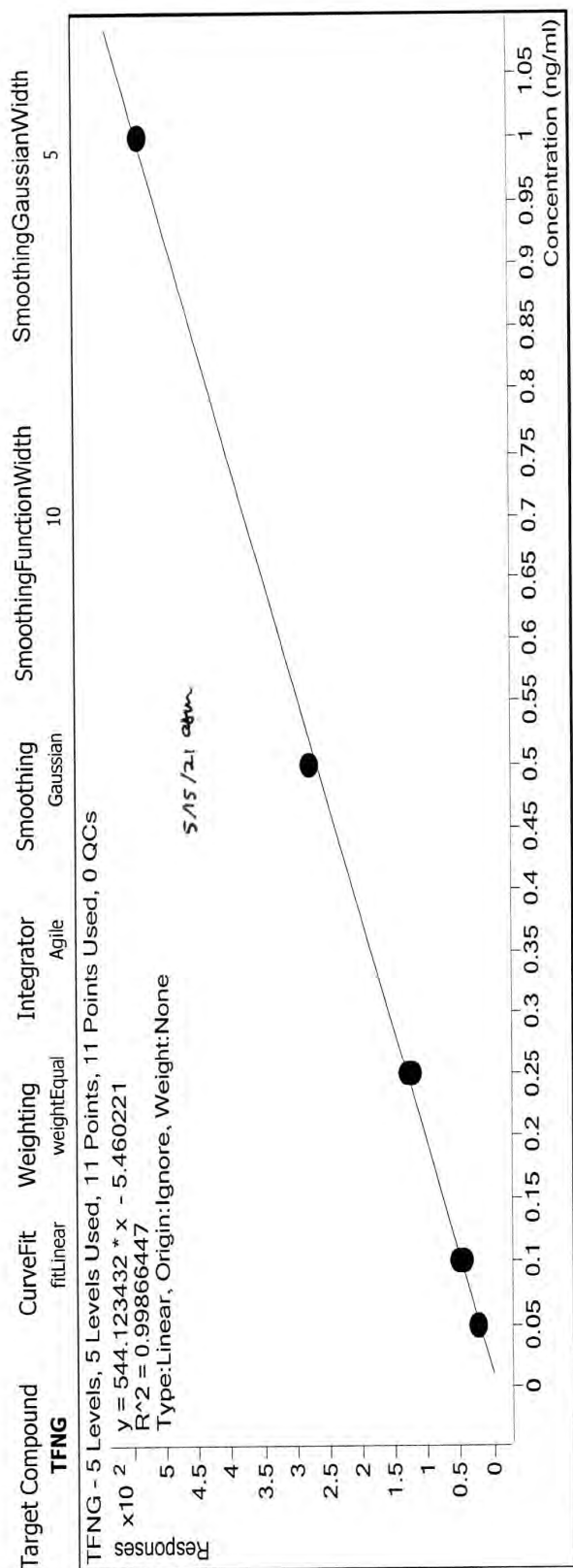
Sample	Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp (pg/µL)	Conc (pg/µL)	ppm	Ave ppm	% Rec	Ave % Rec	Std Dev
Sample	P1-A3	10.0	10.0										
Sample	P1-A3	10.0	10.0										
Sample	P1-A3	10.0	10.0										
Sample	P1-A3	10.0	10.0										
Sample	P1-A3	10.0	10.0										
Sample	P1-A3	10.0	10.0										
Sample	P1-A3	10.0	10.0										
Calibration	Vial 5	10.0	10.0				25.24						
Calibration	Vial 4	10.0	10.0				52.01						
Calibration	Vial 3	10.0	10.0				124.67						
Calibration	Vial 2	10.0	10.0				277.14						
Calibration	Vial 1	10.0	10.0				537.20						
Sample	P1-A1	10.0	10.0	0.100	10.0	0.1000		<0.10	<0.01				
Sample	P1-A1	10.0	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01			
Sample	P1-A2	10.0	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01			
Sample	P1-A2	10.0	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01			
Calibration	Vial 4	10.0	10.0	0.100	10.0	0.1000	53.00						
Sample	P1-A3	10.0	10.0	0.100	10.0	0.1000	43.89	0.0907	0.0091				
Sample	P1-A3	10.0	10.0	0.100	10.0	0.1000	45.47	0.0936	0.0094	0.00922	92%		
Sample	P1-A4	10.0	10.0	0.100	10.0	0.1000	43.64	0.0902	0.0090				
Sample	P1-A4	10.0	10.0	0.100	10.0	0.1000	45.19	0.0931	0.0093	0.00917	92%		
Calibration	Vial 3	10.0	10.0				122.76						
Sample	P1-A5	10.0	10.0	0.100	10.0	0.1000	39.48	0.0826	0.0083				
Sample	P1-A5	10.0	10.0	0.100	10.0	0.1000	42.25	0.0877	0.0088	0.00851	85%	90%	4%
Sample	P1-A6	10.0	10.0	0.100	40.0	0.0250	126.20	0.2420	0.0968				
Sample	P1-A6	10.0	10.0	0.100	40.0	0.0250	129.99	0.2489	0.0996	0.0982	98%		
Calibration	Vial 4	10.0	10.0				52.60						
Sample	P1-A7	10.0	10.0	0.100	40.0	0.0250	125.91	0.2414	0.0966				
Sample	P1-A7	10.0	10.0	0.100	40.0	0.0250	130.60	0.2501	0.1000	0.0983	98%		
Sample	P1-A8	10.0	10.0	0.100	40.0	0.0250	136.77	0.2614	0.1046				
Sample	P1-A8	10.0	10.0	0.100	40.0	0.0250	130.64	0.2501	0.1001	0.102	102%	100%	2%
Calibration	Vial 3	10.0	10.0				127.87						
Sample	P1-A9	10.0	10.0	0.100	400.0	0.0025	141.07	0.2693	1.0771				
Sample	P1-A9	10.0	10.0	0.100	400.0	0.0025	132.10	0.2528	1.0112	1.04	104%		
Sample	P1-B1	10.0	10.0	0.100	400.0	0.0025	140.56	0.2684	1.0734				
Sample	P1-B1	10.0	10.0	0.100	400.0	0.0025	135.44	0.2589	1.0358	1.05	105%		
Calibration	Vial 4	10.0	10.0				43.77						
Sample	P1-B2	10.0	10.0	0.100	400.0	0.0025	138.65	0.2649	1.0594				
Sample	P1-B2	10.0	10.0	0.100	400.0	0.0025	141.30	0.2697	1.0789	1.07	107%	106%	1%
Calibration	Vial 3	10.0	10.0				128.84						

Calculated LOD at 0.045 pg/µL = 19

Calculated LOQ at 0.10 pg/µL = 49

"-----" denotes response below area threshold

Printed at: 1:11 PM on: 5/15/2021

**Calibration STD**

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_011.d	Calibration	1	☒	537.20	1.0000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_010.d	Calibration	2	☒	277.14	0.5000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_009.d	Calibration	3	☒	124.67	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_008.d	Calibration	3	☒	122.76	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_007.d	Calibration	3	☒	127.87	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_006.d	Calibration	3	☒	128.84	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_005.d	Calibration	4	☒	52.01	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_004.d	Calibration	4	☒	53.00	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_003.d	Calibration	4	☒	52.60	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_002.d	Calibration	4	☒	43.77	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05142021\05142021a_001.d	Calibration	5	☒	25.24	0.0500

Compound Injection Date
Flonicamid 5/6/2021

08550 - Flonicamid/Onion - Sample Analysis - Field Trials CO461, CA*19 - Extracted 5/6/2021 by ASM

5/7/21 *asm*

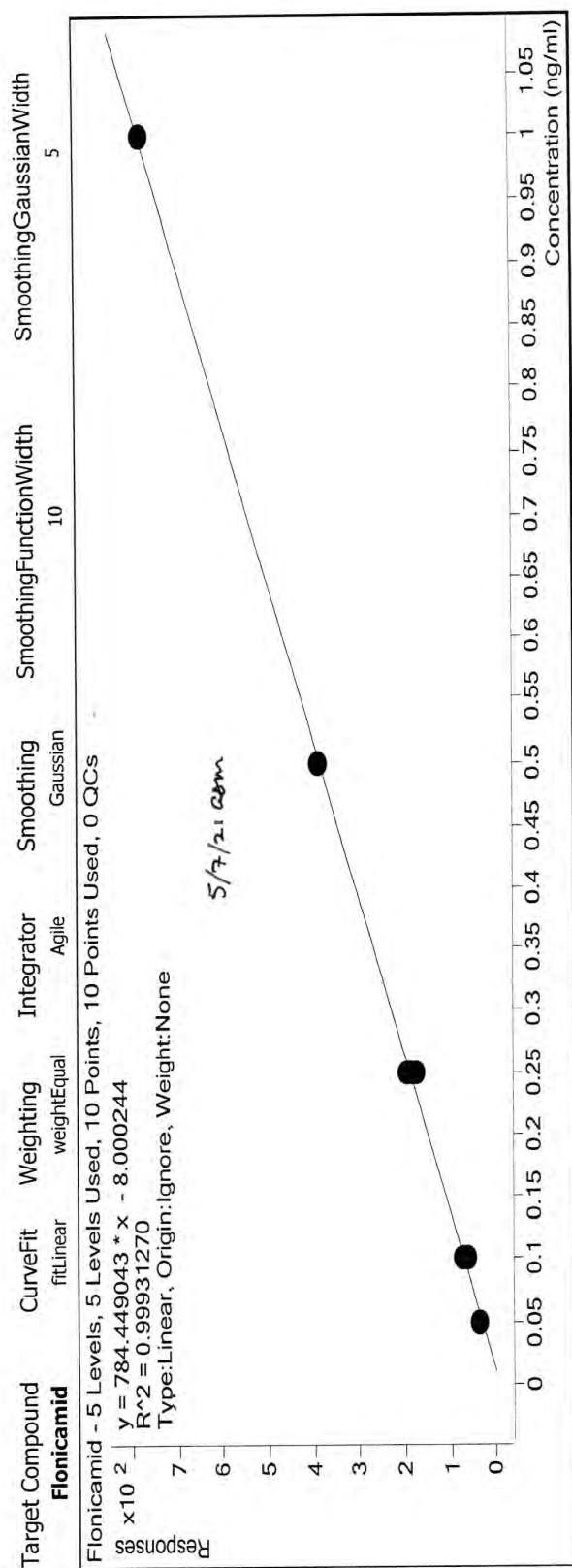
Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05062021a_001.d	-----	Condition		Sample	P1-A3	10.0								
05062021a_002.d	7.721	Condition		Sample	P1-A3	10.0								
05062021a_003.d	7.735	Condition		Sample	P1-A3	10.0								
05062021a_004.d	7.721	Condition		Sample	P1-A3	10.0								
05062021a_005.d	7.721	Condition		Sample	P1-A3	10.0								
05062021a_006.d	7.735	Condition		Sample	P1-A3	10.0								
05062021a_007.d	7.735	Condition		Sample	P1-A3	10.0								
05062021a_008.d	7.721	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				39.46				
05062021a_009.d	7.735	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				73.00				
05062021a_010.d	7.721	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				187.94				
05062021a_011.d	7.721	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				388.39				
05062021a_012.d	7.721	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				776.78				
05062021a_013.d	-----	28853	DBA-CO461	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05062021a_014.d	-----	28853	DBA-CO461	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05062021a_015.d	-----	28989	DBA-CA*19	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05062021a_016.d	-----	28989	DBA-CA*19	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05062021a_017.d	7.735	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				64.29				
05062021a_018.d	7.721	28853C0.01R1	DBA-CO461	Sample	P1-A3	10.0	0.100	10.0	0.1000	61.28	0.0883	0.0088		
05062021a_019.d	7.721	28853C0.01R1	DBA-CO461	Sample	P1-A3	10.0	0.100	10.0	0.1000	66.63	0.0951	0.0095	0.00917	92%
05062021a_020.d	7.721	28853C1.0R1	DBA-CO461	Sample	P1-A4	10.0	0.100	400.0	0.0025	180.66	0.2405	0.9620		
05062021a_021.d	7.735	28853C1.0R1	DBA-CO461	Sample	P1-A4	10.0	0.100	400.0	0.0025	190.53	0.2531	1.0123	0.987	99%
05062021a_022.d	7.721	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				176.02				
05062021a_023.d	7.735	28855	DBC-CO461	Sample	P1-A5	40.0	0.100	40.0	0.1000	872.42	1.1223	0.1122		
05062021a_024.d	7.721	28855	DBC-CO461	Sample	P1-A5	40.0	0.100	40.0	0.1000	880.47	1.1326	0.1133	0.11	
05062021a_025.d	7.735	28856	DBD-CO461	Sample	P1-A6	40.0	0.100	40.0	0.1000	741.68	0.9557	0.0956		
05062021a_026.d	7.735	28856	DBD-CO461	Sample	P1-A6	40.0	0.100	40.0	0.1000	747.23	0.9628	0.0963	0.10	
05062021a_027.d	7.721	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				71.64				
05062021a_028.d	7.735	28991	DBC-CA*19	Sample	P1-A7	10.0	0.100	10.0	0.1000	77.69	0.1092	0.0109		
05062021a_029.d	7.735	28991	DBC-CA*19	Sample	P1-A7	10.0	0.100	10.0	0.1000	75.81	0.1068	0.0107	0.011	
05062021a_030.d	7.735	28992	DBD-CA*19	Sample	P1-A8	10.0	0.100	10.0	0.1000	83.80	0.1170	0.0117		
05062021a_031.d	7.735	28992	DBD-CA*19	Sample	P1-A8	10.0	0.100	10.0	0.1000	81.27	0.1138	0.0114	0.012	
05062021a_032.d	7.735	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				192.66				
05062021a_033.d	7.735	28855 (Flonicamid)	DBC-CO461	Sample	P1-A9	10.0	0.100	40.0	0.0250	237.54	0.3130	0.1252		
05062021a_034.d	7.748	28855 (Flonicamid)	DBC-CO461	Sample	P1-A9	10.0	0.100	40.0	0.0250	236.87	0.3122	0.1249	0.13	
05062021a_035.d	7.735	28856 (Flonicamid)	DBD-CO461	Sample	P1-B1	10.0	0.100	40.0	0.0250	207.03	0.2741	0.1096		
05062021a_036.d	7.748	28856 (Flonicamid)	DBD-CO461	Sample	P1-B1	10.0	0.100	40.0	0.0250	211.59	0.2799	0.1120	0.11	
05062021a_037.d	7.735	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				185.50				

Calculated LOD at 0.045 pg/µL = 27

Calculated LOQ at 0.10 pg/µL = 70

"-----" denotes response below area threshold

Strikethrough denotes data not used, dilutions used for CO461 flonicamid samples only

**Calibration STD**

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_012.d	Calibration	Level	1	Enabled	☒	Response	776.78	Exp Conc	1.0000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_011.d	Calibration	Level	2	Enabled	☒	Response	388.39	Exp Conc	0.5000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_010.d	Calibration	Level	3	Enabled	☒	Response	187.94	Exp Conc	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_022.d	Calibration	Level	3	Enabled	☒	Response	176.02	Exp Conc	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_032.d	Calibration	Level	3	Enabled	☒	Response	192.66	Exp Conc	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_037.d	Calibration	Level	3	Enabled	☒	Response	185.50	Exp Conc	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_009.d	Calibration	Level	4	Enabled	☒	Response	73.00	Exp Conc	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_017.d	Calibration	Level	4	Enabled	☒	Response	64.29	Exp Conc	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_027.d	Calibration	Level	4	Enabled	☒	Response	71.64	Exp Conc	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_008.d	Calibration	Level	5	Enabled	☒	Response	39.46	Exp Conc	0.0500

Compound Injection Date
TFNA-AM 5/6/2021

5/7/21 agm

08550 - Flonicamid/Onion - Sample Analysis - Field Trials CO461, CA*19 - Extracted 5/6/2021 by ASM

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05062021a_001.d	-----	Condition		Sample	P1-A3	10.0								
05062021a_002.d	5.311	Condition		Sample	P1-A3	10.0								
05062021a_003.d	5.311	Condition		Sample	P1-A3	10.0								
05062021a_004.d	5.324	Condition		Sample	P1-A3	10.0								
05062021a_005.d	5.311	Condition		Sample	P1-A3	10.0								
05062021a_006.d	5.311	Condition		Sample	P1-A3	10.0								
05062021a_007.d	5.324	Condition		Sample	P1-A3	10.0								
05062021a_008.d	5.324	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				103.75				
05062021a_009.d	5.324	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				217.34				
05062021a_010.d	5.311	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				527.23				
05062021a_011.d	5.311	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				1071.15				
05062021a_012.d	5.297	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				2150.88				
05062021a_013.d	-----	28853	DBA-CO461	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05062021a_014.d	-----	28853	DBA-CO461	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05062021a_015.d	-----	28989	DBA-CA*19	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05062021a_016.d	-----	28989	DBA-CA*19	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05062021a_017.d	5.324	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				172.29				
05062021a_018.d	5.311	28853CO.01R1	DBA-CO461	Sample	P1-A3	10.0	0.100	10.0	0.1000	165.24	0.0922	0.0092		
05062021a_019.d	5.324	28853CO.01R1	DBA-CO461	Sample	P1-A3	10.0	0.100	10.0	0.1000	171.74	0.0952	0.0095	0.00937	94%
05062021a_020.d	5.324	28853C1.0R1	DBA-CO461	Sample	P1-A4	10.0	0.100	400.0	0.0025	474.88	0.2345	0.9378		
05062021a_021.d	5.324	28853C1.0R1	DBA-CO461	Sample	P1-A4	10.0	0.100	400.0	0.0025	470.94	0.2326	0.9306	0.934	93%
05062021a_022.d	5.311	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				461.18				
05062021a_023.d	-----	28855	DBC-CO461	Sample	P1-A5	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05062021a_024.d	-----	28855	DBC-CO461	Sample	P1-A5	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05062021a_025.d	-----	28856	DBD-CO461	Sample	P1-A6	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05062021a_026.d	-----	28856	DBD-CO461	Sample	P1-A6	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05062021a_027.d	5.324	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				197.86				
05062021a_028.d	-----	28991	DBC-CA*19	Sample	P1-A7	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05062021a_029.d	-----	28991	DBC-CA*19	Sample	P1-A7	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05062021a_030.d	-----	28992	DBD-CA*19	Sample	P1-A8	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05062021a_031.d	-----	28992	DBD-CA*19	Sample	P1-A8	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05062021a_032.d	5.311	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				470.09				
05062021a_033.d	-----	28855-(Flonicamid)	DBC-CO461	Sample	P1-A9	10.0	0.100	40.0	0.0250		<0.10	<0.01		
05062021a_034.d	-----	28855-(Flonicamid)	DBC-CO461	Sample	P1-A9	10.0	0.100	40.0	0.0250		<0.10	<0.01		
05062021a_035.d	-----	28856-(Flonicamid)	DBD-CO461	Sample	P1-B1	10.0	0.100	40.0	0.0250		<0.10	<0.01		
05062021a_036.d	-----	28856-(Flonicamid)	DBD-CO461	Sample	P1-B1	10.0	0.100	40.0	0.0250		<0.10	<0.01		
05062021a_037.d	5.311	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				476.88				

Calculated LOD at 0.045 pg/µL = 63

Calculated LOQ at 0.10 pg/µL = 182

"-----" denotes response below area threshold

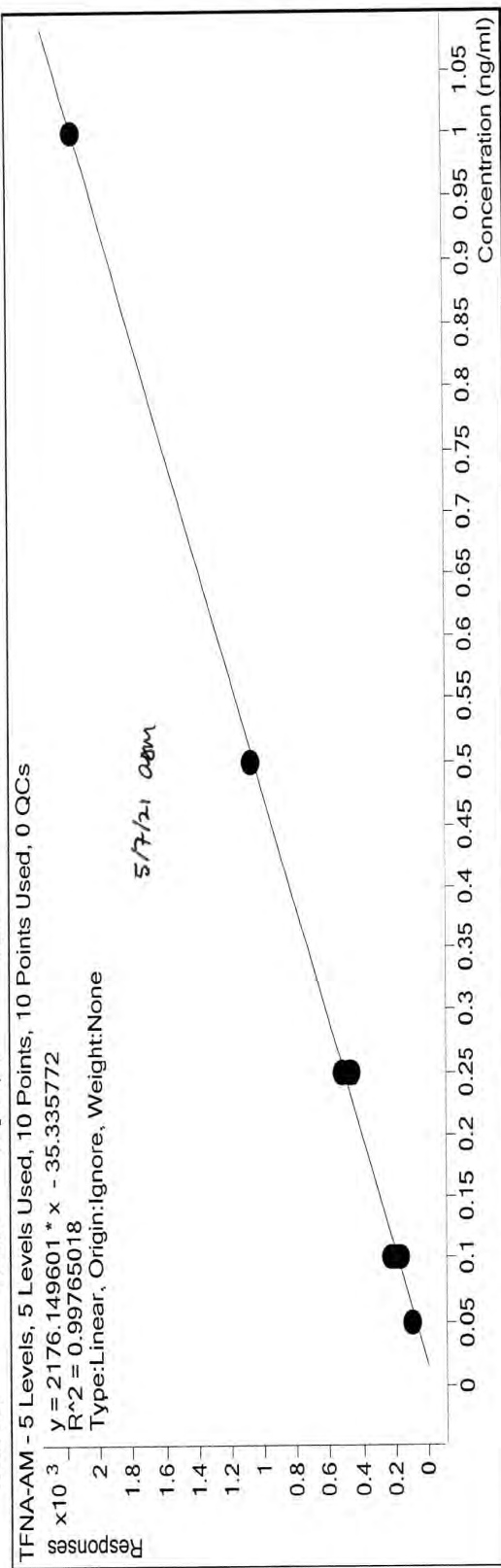
Strikethrough denotes data not used, dilutions used for CO461 flonicamid samples only

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\QuantResults\05062021a_batch.bin

Batch Data Path**Calibration Info**

Target Compound

TFNA-AM

CurveFit
fitLinearWeighting
weightEqualIntegrator
AgileSmoothing
GaussianSmoothingFunctionWidth
10SmoothingGaussianWidth
5**Calibration STD**

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 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_022.d
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 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_017.d
 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_027.d
 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05062021\05062021a_008.d

Cal Type	Level	Enabled	Response	Exp Conc
Calibration	1	☒	2150.88	1.0000
Calibration	2	☒	1071.15	0.5000
Calibration	3	☒	527.23	0.2500
Calibration	3	☒	461.18	0.2500
Calibration	3	☒	470.09	0.2500
Calibration	3	☒	476.88	0.2500
Calibration	4	☒	217.34	0.1000
Calibration	4	☒	172.29	0.1000
Calibration	4	☒	197.86	0.1000
Calibration	5	☒	103.75	0.0500

Compound Injection Date
TFNA 5/6/2021

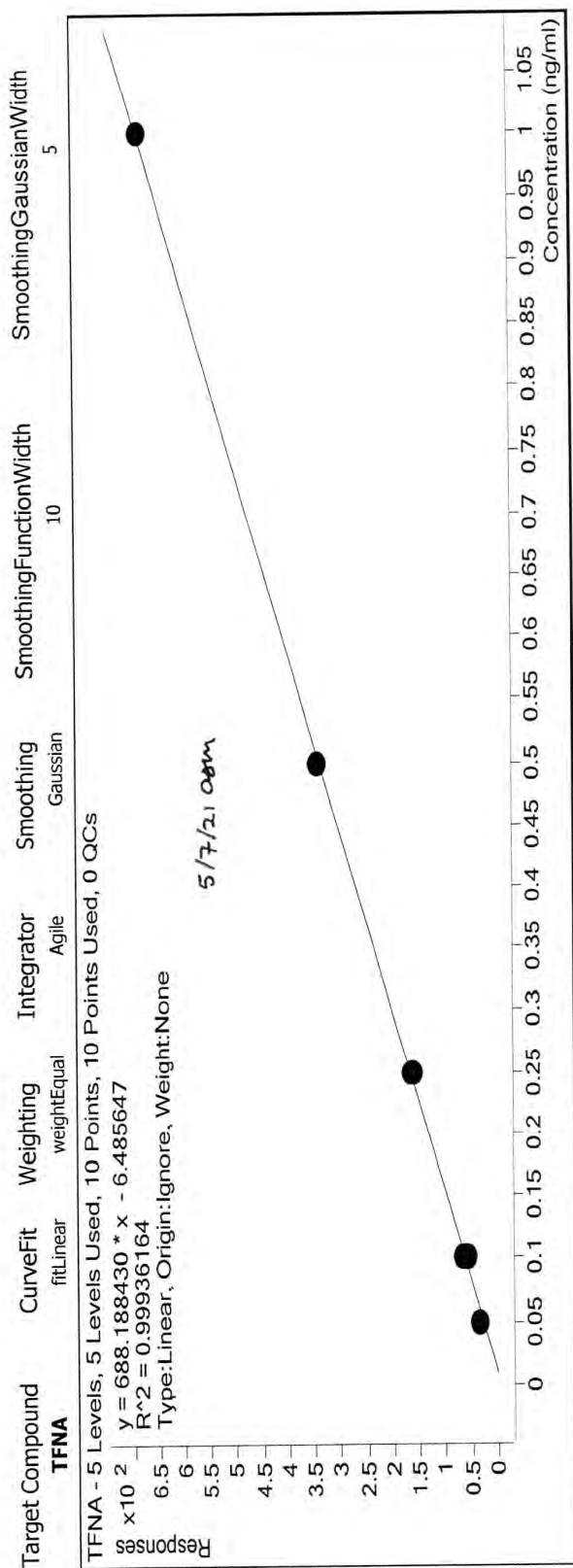
08550 - Fonicamid/Onion - Sample Analysis - Field Trials CO461, CA*19 - Extracted 5/6/2021 by ASM

5/7/21 *adm*

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp (pg/µL)	Conc (pg/µL)	ppm	Ave ppm	% Rec
05062021a_001.d	-----	Condition		Sample	P1-A3	10.0								
05062021a_002.d	6.037	Condition		Sample	P1-A3	10.0								
05062021a_003.d	6.037	Condition		Sample	P1-A3	10.0								
05062021a_004.d	6.037	Condition		Sample	P1-A3	10.0								
05062021a_005.d	6.037	Condition		Sample	P1-A3	10.0								
05062021a_006.d	6.037	Condition		Sample	P1-A3	10.0								
05062021a_007.d	6.037	Condition		Sample	P1-A3	10.0								
05062021a_008.d	6.024	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				36.96				
05062021a_009.d	6.010	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				67.44				
05062021a_010.d	6.010	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				160.66				
05062021a_011.d	6.010	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				341.56				
05062021a_012.d	5.997	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				682.21				
05062021a_013.d	-----	28853	DBA-CO461	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05062021a_014.d	-----	28853	DBA-CO461	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05062021a_015.d	-----	28989	DBA-CA*19	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05062021a_016.d	-----	28989	DBA-CA*19	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05062021a_017.d	6.024	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				55.04				
05062021a_018.d	6.037	28853C0.01R1	DBA-CO461	Sample	P1-A3	10.0	0.100	10.0	0.1000	60.45	0.0973	0.0097		
05062021a_019.d	6.037	28853C0.01R1	DBA-CO461	Sample	P1-A3	10.0	0.100	10.0	0.1000	59.49	0.0959	0.0096	0.00966	97%
05062021a_020.d	6.024	28853C1.0R1	DBA-CO461	Sample	P1-A4	10.0	0.100	400.0	0.0025	164.06	0.2478	0.9913		
05062021a_021.d	6.024	28853C1.0R1	DBA-CO461	Sample	P1-A4	10.0	0.100	400.0	0.0025	162.06	0.2449	0.9796	0.985	99%
05062021a_022.d	6.010	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				162.73				
05062021a_023.d	-----	28855	DBC-CO461	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05062021a_024.d	-----	28855	DBC-CO461	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05062021a_025.d	-----	28856	DBD-CO461	Sample	P1-A6	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05062021a_026.d	-----	28856	DBD-CO461	Sample	P1-A6	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05062021a_027.d	6.010	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				61.58				
05062021a_028.d	6.051	28991	DBC-CA*19	Sample	P1-A7	10.0	0.100	10.0	0.1000	83.79	0.1312	0.0131		
05062021a_029.d	6.051	28991	DBC-CA*19	Sample	P1-A7	10.0	0.100	10.0	0.1000	86.82	0.1356	0.0136	0.013	
05062021a_030.d	6.051	28992	DBD-CA*19	Sample	P1-A8	10.0	0.100	10.0	0.1000	93.31	0.1450	0.0145		
05062021a_031.d	6.051	28992	DBD-CA*19	Sample	P1-A8	10.0	0.100	10.0	0.1000	97.61	0.1513	0.0151	0.015	
05062021a_032.d	6.010	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				162.63				
05062021a_033.d	-----	28855 (Fonicamid)	DBC-CO461	Sample	P1-A9	10.0	0.100	40.0	0.0250	-----	<0.10	<0.01	<0.01	
05062021a_034.d	-----	28855 (Fonicamid)	DBC-CO461	Sample	P1-A9	10.0	0.100	40.0	0.0250	-----	<0.10	<0.01	<0.01	
05062021a_035.d	-----	28856 (Fonicamid)	DBD-CO461	Sample	P1-B1	10.0	0.100	40.0	0.0250	-----	<0.10	<0.01	<0.01	
05062021a_036.d	-----	28856 (Fonicamid)	DBD-CO461	Sample	P1-B1	10.0	0.100	40.0	0.0250	-----	<0.10	<0.01	<0.01	
05062021a_037.d	6.010	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				165.67				
Calculated LOD at 0.045 pg/µL =	24													
Calculated LOQ at 0.10 pg/µL =	62													

"-----" denotes response below area threshold

Strikethrough denotes data not used, dilutions used for CO461 fonicamid samples only

**Calibration STD**

D:\MassHunter\F\lonicamid\08550_Orion\Data\Results\05062021\05062021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\F\lonicamid\08550_Orion\Data\Results\05062021\05062021a_011.d	Calibration	1	☒	682.21	1.0000
D:\MassHunter\F\lonicamid\08550_Orion\Data\Results\05062021\05062021a_010.d	Calibration	2	☒	341.56	0.5000
D:\MassHunter\F\lonicamid\08550_Orion\Data\Results\05062021\05062021a_010.d	Calibration	3	☒	160.66	0.2500
D:\MassHunter\F\lonicamid\08550_Orion\Data\Results\05062021\05062021a_022.d	Calibration	3	☒	162.73	0.2500
D:\MassHunter\F\lonicamid\08550_Orion\Data\Results\05062021\05062021a_032.d	Calibration	3	☒	162.63	0.2500
D:\MassHunter\F\lonicamid\08550_Orion\Data\Results\05062021\05062021a_037.d	Calibration	3	☒	165.67	0.2500
D:\MassHunter\F\lonicamid\08550_Orion\Data\Results\05062021\05062021a_009.d	Calibration	4	☒	67.44	0.1000
D:\MassHunter\F\lonicamid\08550_Orion\Data\Results\05062021\05062021a_017.d	Calibration	4	☒	55.04	0.1000
D:\MassHunter\F\lonicamid\08550_Orion\Data\Results\05062021\05062021a_027.d	Calibration	4	☒	61.58	0.1000
D:\MassHunter\F\lonicamid\08550_Orion\Data\Results\05062021\05062021a_008.d	Calibration	5	☒	36.96	0.0500

Compound Injection Date
TFNG 5/6/2021

08550 - Flonicamid/Onion - Sample Analysis - Field Trials CO461, CA*19 - Extracted 5/6/2021 by ASM

5/7/21 ASM

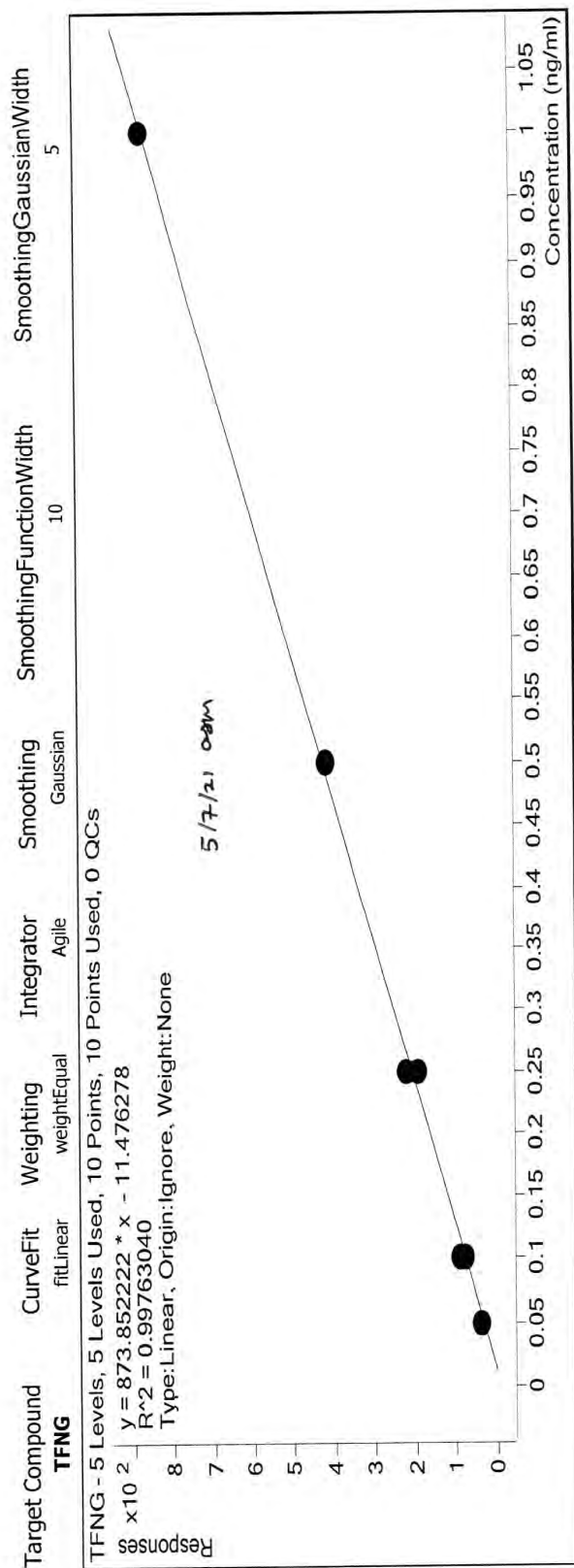
Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05062021a_001.d	-----	Condition		Sample	P1-A3	10.0								
05062021a_002.d	7.212	Condition		Sample	P1-A3	10.0								
05062021a_003.d	7.212	Condition		Sample	P1-A3	10.0								
05062021a_004.d	7.212	Condition		Sample	P1-A3	10.0								
05062021a_005.d	7.212	Condition		Sample	P1-A3	10.0								
05062021a_006.d	7.212	Condition		Sample	P1-A3	10.0								
05062021a_007.d	7.212	Condition		Sample	P1-A3	10.0								
05062021a_008.d	7.198	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				40.97				
05062021a_009.d	7.212	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				90.87				
05062021a_010.d	7.212	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				221.95				
05062021a_011.d	7.212	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				414.77				
05062021a_012.d	7.198	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				871.97				
05062021a_013.d	-----	28853	DBA-CO461	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05062021a_014.d	-----	28853	DBA-CO461	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05062021a_015.d	-----	28989	DBA-CA*19	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05062021a_016.d	-----	28989	DBA-CA*19	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05062021a_017.d	7.212	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				75.98				
05062021a_018.d	7.212	28853C0.01R1	DBA-CO461	Sample	P1-A3	10.0	0.100	10.0	0.1000	67.45	0.0903	0.0090		
05062021a_019.d	7.212	28853C0.01R1	DBA-CO461	Sample	P1-A3	10.0	0.100	10.0	0.1000	75.72	0.0998	0.0100	0.00951	95%
05062021a_020.d	7.212	28853C1.0R1	DBA-CO461	Sample	P1-A4	10.0	0.100	400.0	0.0025	210.84	0.2544	1.0176		
05062021a_021.d	7.212	28853C1.0R1	DBA-CO461	Sample	P1-A4	10.0	0.100	400.0	0.0025	226.61	0.2725	1.0898	1.05	105%
05062021a_022.d	7.212	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				192.76				
05062021a_023.d	7.225	28855	DBC-CO461	Sample	P1-A5	10.0	0.100	10.0	0.1000	49.46	<0.10	<0.01		
05062021a_024.d	7.212	28855	DBC-CO461	Sample	P1-A5	10.0	0.100	10.0	0.1000	50.68	<0.10	<0.01	<0.01	
05062021a_025.d	7.239	28856	DBD-CO461	Sample	P1-A6	10.0	0.100	10.0	0.1000	34.19	<0.10	<0.01		
05062021a_026.d	7.225	28856	DBD-CO461	Sample	P1-A6	10.0	0.100	10.0	0.1000	38.00	<0.10	<0.01	<0.01	
05062021a_027.d	7.212	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				79.35				
05062021a_028.d	7.225	28991	DBC-CA*19	Sample	P1-A7	10.0	0.100	10.0	0.1000	77.82	0.1022	0.0102		
05062021a_029.d	7.225	28991	DBC-CA*19	Sample	P1-A7	10.0	0.100	10.0	0.1000	75.26	0.0993	0.0099	0.010	
05062021a_030.d	7.225	28992	DBD-CA*19	Sample	P1-A8	10.0	0.100	10.0	0.1000	77.19	0.1015	0.0101		
05062021a_031.d	7.239	28992	DBD-CA*19	Sample	P1-A8	10.0	0.100	10.0	0.1000	80.14	0.1048	0.0105	0.010	
05062021a_032.d	7.212	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				192.95				
05062021a_033.d	-----	28855 (Flonicamid)	DBC-CO461	Sample	P1-A9	40.0	0.100	40.0	0.0250	-----	<0.10	<0.01	<0.01	
05062021a_034.d	-----	28855 (Flonicamid)	DBC-CO461	Sample	P1-A9	40.0	0.100	40.0	0.0250	-----	<0.10	<0.01	<0.01	
05062021a_035.d	-----	28856 (Flonicamid)	DBD-CO461	Sample	P1-B1	40.0	0.100	40.0	0.0250	-----	<0.10	<0.01	<0.01	
05062021a_036.d	-----	28856 (Flonicamid)	DBD-CO461	Sample	P1-B1	40.0	0.100	40.0	0.0250	-----	<0.10	<0.01	<0.01	
05062021a_037.d	7.212	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				194.15				

Calculated LOD at 0.045 pg/µL = 28

Calculated LOQ at 0.10 pg/µL = 76

"-----" denotes response below area threshold

Strikethrough denotes data not used, dilutions used for CO461 flonicamid samples only

**Calibration STD**

D:\MassHunter\FI\onid\08550_Onion\Data\Results\05062021\05062021a_012.d	Calibration	Level	1	Enabled	☒	Response	871.97	Exp Conc	1.0000
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D:\MassHunter\FI\onid\08550_Onion\Data\Results\05062021\05062021a_010.d	Calibration	Level	3	Enabled	☒	Response	221.95	Exp Conc	0.2500
D:\MassHunter\FI\onid\08550_Onion\Data\Results\05062021\05062021a_022.d	Calibration	Level	3	Enabled	☒	Response	192.76	Exp Conc	0.2500
D:\MassHunter\FI\onid\08550_Onion\Data\Results\05062021\05062021a_032.d	Calibration	Level	3	Enabled	☒	Response	192.95	Exp Conc	0.2500
D:\MassHunter\FI\onid\08550_Onion\Data\Results\05062021\05062021a_037.d	Calibration	Level	3	Enabled	☒	Response	194.15	Exp Conc	0.2500
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D:\MassHunter\FI\onid\08550_Onion\Data\Results\05062021\05062021a_017.d	Calibration	Level	4	Enabled	☒	Response	75.98	Exp Conc	0.1000
D:\MassHunter\FI\onid\08550_Onion\Data\Results\05062021\05062021a_027.d	Calibration	Level	4	Enabled	☒	Response	79.35	Exp Conc	0.1000
D:\MassHunter\FI\onid\08550_Onion\Data\Results\05062021\05062021a_008.d	Calibration	Level	5	Enabled	☒	Response	40.97	Exp Conc	0.0500

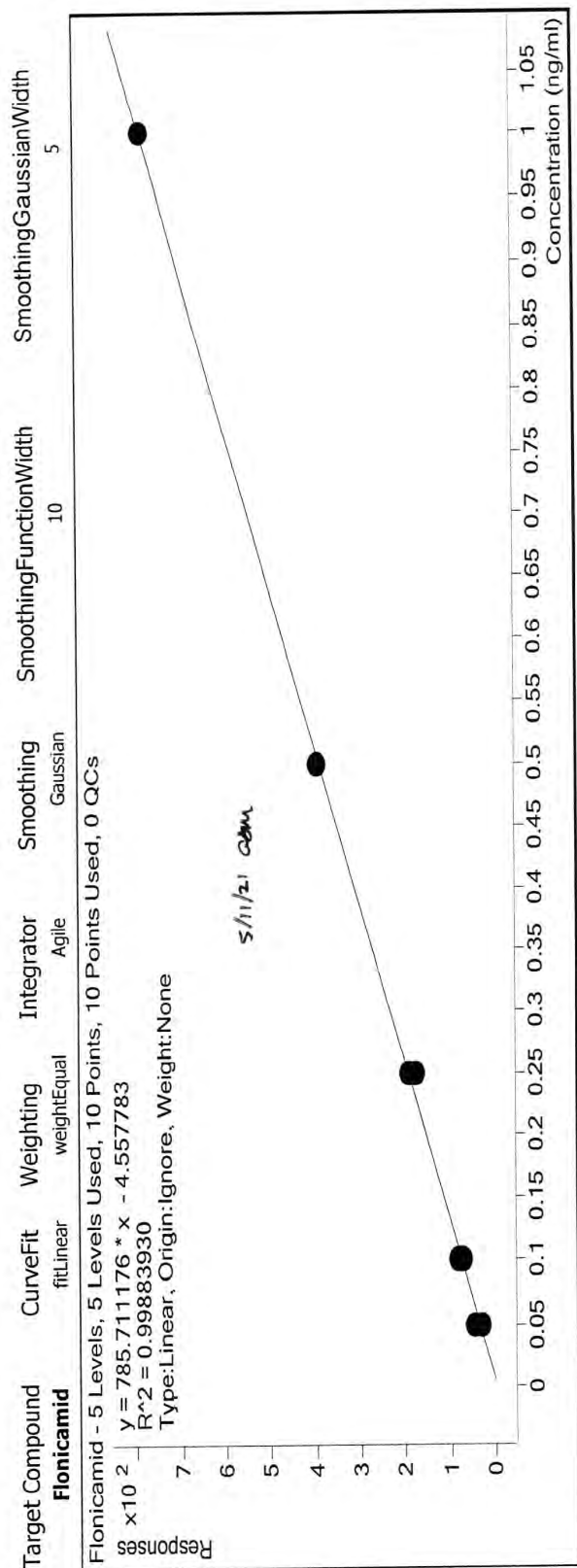
Compound
Fonicamid
08550 - Fonicamid/Onion - Sample Analysis - Field Trials CA16, CA17, ID181 - Extracted 5/10/2021 by ASM

Injection Date
5/10/2021

5/11/21 *QSM*

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05102021a_001.d	7.735	Condition		Sample	P1-A3	10.0								
05102021a_002.d	7.735	Condition		Sample	P1-A3	10.0								
05102021a_003.d	7.735	Condition		Sample	P1-A3	10.0								
05102021a_004.d	7.735	Condition		Sample	P1-A3	10.0								
05102021a_005.d	7.735	Condition		Sample	P1-A3	10.0								
05102021a_006.d	7.735	Condition		Sample	P1-A3	10.0								
05102021a_007.d	7.721	Condition		Sample	P1-A3	10.0								
05102021a_008.d	7.735	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				43.66				
05102021a_009.d	7.748	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				84.06				
05102021a_010.d	7.735	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				188.36				
05102021a_011.d	7.721	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				391.81				
05102021a_012.d	7.735	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				783.83				
05102021a_013.d	7.735	28241	DBA-CA16	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05102021a_014.d	7.748	28241	DBA-CA16	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05102021a_015.d	7.748	28304	DBA-CA17	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05102021a_016.d	7.735	28304	DBA-CA17	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05102021a_017.d	7.735	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				32.69				
05102021a_018.d	7.748	28241C0.01R2	DBA-CA16	Sample	P1-A3	10.0	0.100	10.0	0.1000	69.04	0.0937	0.0094		
05102021a_019.d	7.748	28241C0.01R2	DBA-CA16	Sample	P1-A3	10.0	0.100	10.0	0.1000	64.37	0.0877	0.0088		91%
05102021a_020.d	7.748	28241C1.0R2	DBA-CA16	Sample	P1-A4	10.0	0.100	400.0	0.0025	157.23	0.2059	0.8236		
05102021a_021.d	7.735	28241C1.0R2	DBA-CA16	Sample	P1-A4	10.0	0.100	400.0	0.0025	168.09	0.2197	0.8789		85%
05102021a_022.d	7.735	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				72.70				
05102021a_023.d	7.748	28243	DBC-CA16	Sample	P1-A5	10.0	0.100	10.0	0.1000	222.84	0.2894	0.0289		
05102021a_024.d	7.748	28243	DBC-CA16	Sample	P1-A5	10.0	0.100	10.0	0.1000	221.33	0.2875	0.0288		0.029
05102021a_025.d	7.735	28244	DBD-CA16	Sample	P1-A6	10.0	0.100	10.0	0.1000	156.16	0.2046	0.0205		
05102021a_026.d	7.735	28244	DBD-CA16	Sample	P1-A6	10.0	0.100	10.0	0.1000	157.91	0.2068	0.0207		0.021
05102021a_027.d	7.735	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				173.69				
05102021a_028.d	7.748	28306	DBC-CA17	Sample	P1-A7	10.0	0.100	10.0	0.1000	197.42	0.2571	0.0257		
05102021a_029.d	7.748	28306	DBC-CA17	Sample	P1-A7	10.0	0.100	10.0	0.1000	209.72	0.2727	0.0273		0.026
05102021a_030.d	7.748	28307	DBD-CA17	Sample	P1-A8	10.0	0.100	10.0	0.1000	246.48	0.3195	0.0320		
05102021a_031.d	7.735	28307	DBD-CA17	Sample	P1-A8	10.0	0.100	10.0	0.1000	239.30	0.3104	0.0310		0.031
05102021a_032.d	7.735	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				75.32				
05102021a_033.d	7.735	28859	DBC-ID181	Sample	P1-A9	10.0	0.100	10.0	0.1000	58.34	<0.10	<0.01		
05102021a_034.d	7.748	28859	DBC-ID181	Sample	P1-A9	10.0	0.100	10.0	0.1000	56.70	<0.10	<0.01		
05102021a_035.d	7.735	28860	DBD-ID181	Sample	P1-B1	10.0	0.100	10.0	0.1000	70.98	0.0961	0.0096		
05102021a_036.d	7.735	28860	DBD-ID181	Sample	P1-B1	10.0	0.100	10.0	0.1000	78.66	0.1059	0.0106		0.010
05102021a_037.d	7.748	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				190.44				
Calculated LOD at 0.045 pg/µL =		31												
Calculated LOQ at 0.10 pg/µL =		74												

"----" denotes response below area threshold

**Calibration STD**

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_011.d	Calibration	1	☒	783.83	1.0000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_010.d	Calibration	2	☒	391.81	0.5000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_010.d	Calibration	3	☒	188.36	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_027.d	Calibration	3	☒	173.69	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_037.d	Calibration	3	☒	190.44	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_009.d	Calibration	4	☒	84.06	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_022.d	Calibration	4	☒	72.70	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_032.d	Calibration	4	☒	75.32	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_008.d	Calibration	5	☒	43.66	0.0500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_017.d	Calibration	5	☒	32.69	0.0500

Compound
TFNA-AM

Injection Date
5/10/2021

5/11/21 *adm*

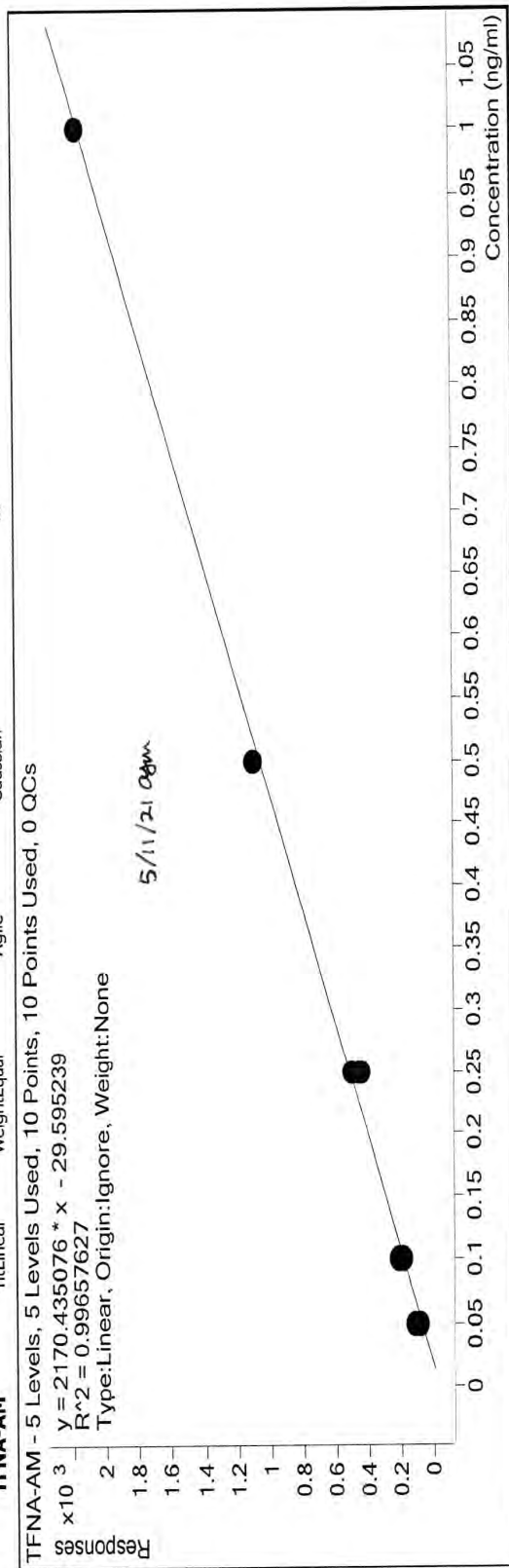
08550 - Flonicamid/Onion - Sample Analysis - Field Trials CA16, CA17, ID181 - Extracted 5/10/2021 by ASM

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp (pg/µL)	Conc (pg/µL)	ppm	Ave ppm	% Rec
05102021a_001.d	-----	Condition		Sample	P1-A3	10.0								
05102021a_002.d	5.324	Condition		Sample	P1-A3	10.0								
05102021a_003.d	5.324	Condition		Sample	P1-A3	10.0								
05102021a_004.d	5.324	Condition		Sample	P1-A3	10.0								
05102021a_005.d	5.338	Condition		Sample	P1-A3	10.0								
05102021a_006.d	5.324	Condition		Sample	P1-A3	10.0								
05102021a_007.d	5.324	Condition		Sample	P1-A3	10.0								
05102021a_008.d	5.338	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				112.56				
05102021a_009.d	5.338	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				211.10				
05102021a_010.d	5.338	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				495.13				
05102021a_011.d	5.311	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				1092.59				
05102021a_012.d	5.297	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				2151.22				
05102021a_013.d	-----	28241	DBA-CA16	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05102021a_014.d	-----	28241	DBA-CA16	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05102021a_015.d	-----	28304	DBA-CA17	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05102021a_016.d	-----	28304	DBA-CA17	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05102021a_017.d	5.338	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				97.26				
05102021a_018.d	5.338	28241C0.01R2	DBA-CA16	Sample	P1-A3	10.0	0.100	10.0	0.1000	153.58	0.0844	0.0084		
05102021a_019.d	5.338	28241C0.01R2	DBA-CA16	Sample	P1-A3	10.0	0.100	10.0	0.1000	162.53	0.0885	0.0089	0.00865	86%
05102021a_020.d	5.338	28241C1.0R2	DBA-CA16	Sample	P1-A4	10.0	0.100	400.0	0.0025	415.51	0.2051	0.8203		
05102021a_021.d	5.324	28241C1.0R2	DBA-CA16	Sample	P1-A4	10.0	0.100	400.0	0.0025	425.04	0.2095	0.8379	0.829	83%
05102021a_022.d	5.324	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				186.79				
05102021a_023.d	-----	28243	DBC-CA16	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05102021a_024.d	-----	28243	DBC-CA16	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05102021a_025.d	-----	28244	DBC-CA16	Sample	P1-A6	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05102021a_026.d	-----	28244	DBC-CA16	Sample	P1-A6	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05102021a_027.d	5.324	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				437.90				
05102021a_028.d	-----	28306	DBC-CA17	Sample	P1-A7	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05102021a_029.d	-----	28306	DBC-CA17	Sample	P1-A7	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05102021a_030.d	-----	28307	DBC-CA17	Sample	P1-A8	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05102021a_031.d	-----	28307	DBC-CA17	Sample	P1-A8	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05102021a_032.d	5.324	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				208.67				
05102021a_033.d	-----	28859	DBC-ID181	Sample	P1-A9	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05102021a_034.d	-----	28859	DBC-ID181	Sample	P1-A9	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05102021a_035.d	-----	28860	DBC-ID181	Sample	P1-B1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05102021a_036.d	-----	28860	DBC-ID181	Sample	P1-B1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05102021a_037.d	5.324	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				462.50				
Calculated LOD at 0.045 pg/µL =		68												
Calculated LOQ at 0.10 pg/µL =		187												
"-----" denotes response below area threshold														

Batch Data Path D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\QuantResults\05102021a.batch.bin

Calibration Info

Target Compound TFNA-AM CurveFit fitLinear Weighting weightEqual Integrator Agile Smoothing Gaussian SmoothingFunctionWidth 10 SmoothingGaussianWidth 5



Calibration STD

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 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_009.d
 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_022.d
 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_032.d
 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05102021\05102021a_008.d
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Cal Type	Level	Enabled	Response	Exp Conc
Calibration	1	☒	2151.22	1.0000
Calibration	2	☒	1092.59	0.5000
Calibration	3	☒	495.13	0.2500
Calibration	3	☒	437.90	0.2500
Calibration	3	☒	462.50	0.2500
Calibration	4	☒	211.10	0.1000
Calibration	4	☒	186.79	0.1000
Calibration	4	☒	208.67	0.1000
Calibration	5	☒	112.56	0.0500
Calibration	5	☒	97.26	0.0500

Compound Injection Date
TFNA 5/10/2021

08550 - Flonicamid/Onion - Sample Analysis - Field Trials CA16, CA17, ID181 - Extracted 5/10/2021 by ASM

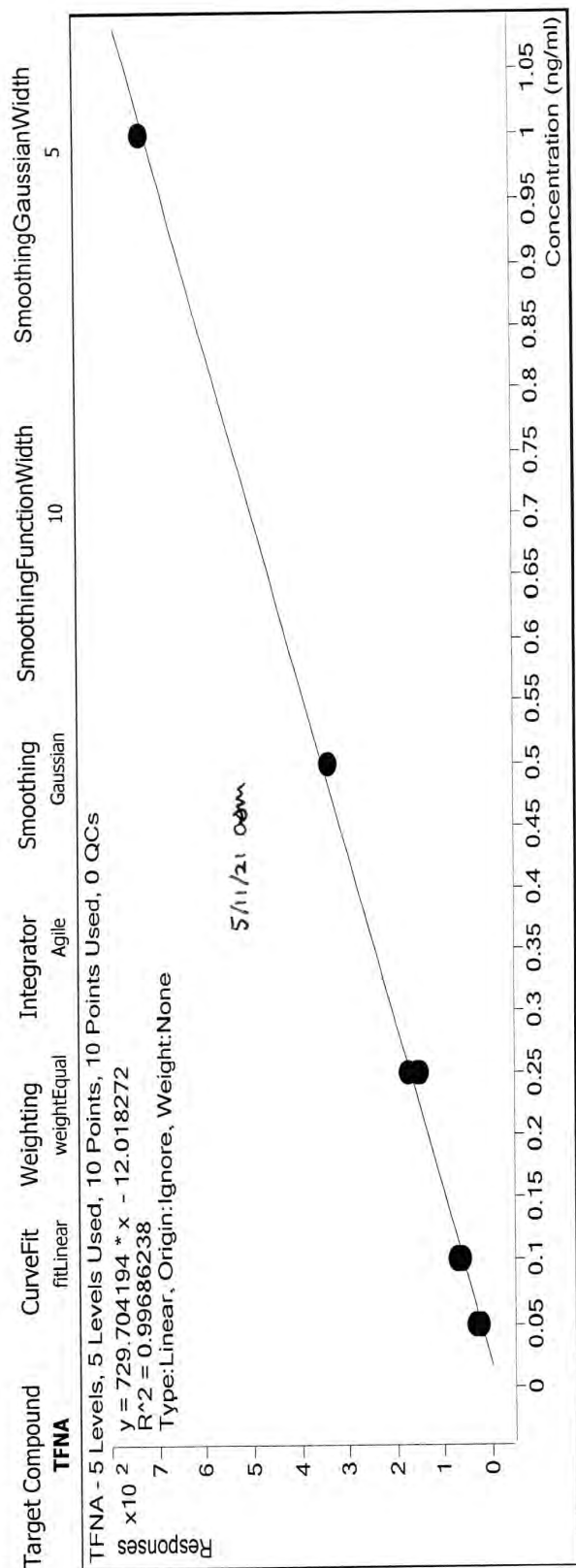
5/11/21 *agm*

RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
6.037	Condition		Sample	P1-A3	10.0								
6.037	Condition		Sample	P1-A3	10.0								
6.037	Condition		Sample	P1-A3	10.0								
6.037	Condition		Sample	P1-A3	10.0								
6.037	Condition		Sample	P1-A3	10.0								
6.037	Condition		Sample	P1-A3	10.0								
6.037	Condition		Sample	P1-A3	10.0								
6.024	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				34.87				
6.024	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				72.96				
6.024	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				172.88				
6.010	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				338.61				
5.997	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				730.20				
6.024	687-2M10 0.05 pg/µL	DBA-CA16	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.051	28241	DBA-CA16	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.051	28241	DBA-CA16	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.037	28304	DBA-CA16	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.024	28304	DBA-CA16	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.024	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				27.26				
6.051	28241C0.01R2	DBA-CA16	Sample	P1-A3	10.0	0.100	10.0	0.1000	57.93	0.0959	0.0096		98%
6.037	28241C0.01R2	DBA-CA16	Sample	P1-A3	10.0	0.100	10.0	0.1000	61.75	0.1011	0.0101		
6.024	28241C1.0R2	DBA-CA16	Sample	P1-A4	10.0	0.100	400.0	0.0025	149.34	0.2211	0.8845		
6.024	687-2M9 0.10 pg/µL	DBA-CA16	Sample	P1-A4	10.0	0.100	400.0	0.0025	157.80	0.2327	0.9309		91%
6.024	28243		Calibration	Vial 4	10.0				65.86				
6.024	28243	DBC-CA16	Sample	P1-A5	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.024	28243	DBC-CA16	Sample	P1-A5	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.024	28244	DBC-CA16	Sample	P1-A6	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.024	28244	DBC-CA16	Sample	P1-A6	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.010	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				151.84				
6.010	28306	DBC-CA17	Sample	P1-A7	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.010	28306	DBC-CA17	Sample	P1-A7	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.010	28307	DBC-CA17	Sample	P1-A8	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.010	28307	DBC-CA17	Sample	P1-A8	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.024	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				65.31				
6.024	28859	DBC-ID181	Sample	P1-A9	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.024	28859	DBC-ID181	Sample	P1-A9	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.024	28860	DBC-ID181	Sample	P1-B1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.024	28860	DBC-ID181	Sample	P1-B1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
6.024	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				153.74				

Calculated LOD at 0.045 pg/µL = 21

Calculated LOQ at 0.10 pg/µL = 61

"-----" denotes response below area threshold



Calibration STD

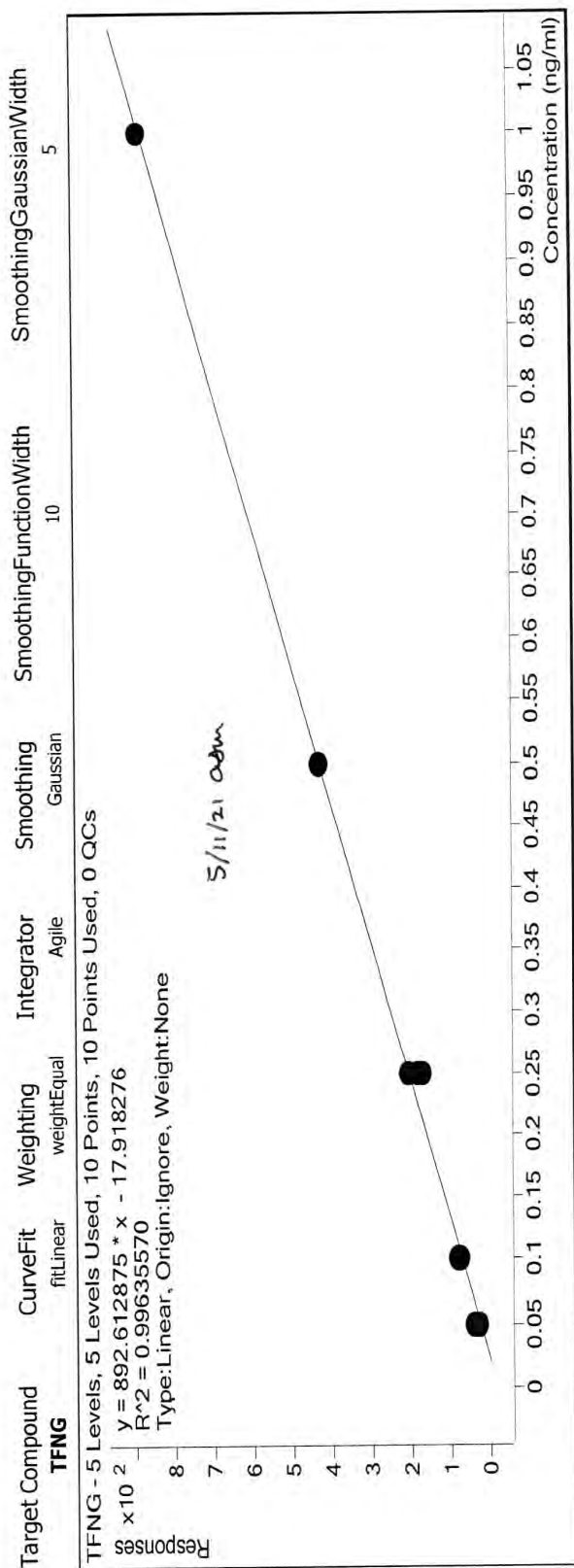
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05102021\05102021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05102021\05102021a_011.d	Calibration	1	☒	730.20	1.0000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05102021\05102021a_010.d	Calibration	2	☒	338.61	0.5000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05102021\05102021a_009.d	Calibration	3	☒	172.88	0.2500
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05102021\05102021a_008.d	Calibration	3	☒	151.84	0.2500
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05102021\05102021a_007.d	Calibration	3	☒	153.74	0.2500
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05102021\05102021a_006.d	Calibration	4	☒	72.96	0.1000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05102021\05102021a_005.d	Calibration	4	☒	65.86	0.1000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05102021\05102021a_004.d	Calibration	4	☒	65.31	0.1000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05102021\05102021a_003.d	Calibration	5	☒	34.87	0.0500
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05102021\05102021a_002.d	Calibration	5	☒	27.26	0.0500

Compound Injection Date
TFNG 5/10/2021

5/11/21 *asm*

08550 - Flonicamid/Onion - Sample Analysis - Field Trials CA16, CA17, ID181 - Extracted 5/10/2021 by ASM

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05102021a_001.d	-----	Condition		Sample	P1-A3	10.0								
05102021a_002.d	7.225	Condition		Sample	P1-A3	10.0								
05102021a_003.d	7.225	Condition		Sample	P1-A3	10.0								
05102021a_004.d	7.239	Condition		Sample	P1-A3	10.0								
05102021a_005.d	7.225	Condition		Sample	P1-A3	10.0								
05102021a_006.d	7.225	Condition		Sample	P1-A3	10.0								
05102021a_007.d	7.212	Condition		Sample	P1-A3	10.0								
05102021a_008.d	7.225	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				39.74				
05102021a_009.d	7.239	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				81.28				
05102021a_010.d	7.225	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				203.42				
05102021a_011.d	7.212	687-2M7 0.50 pg/µL		Calibration	Vial 2	10.0				430.67				
05102021a_012.d	7.212	687-2M6 1.0 pg/µL		Calibration	Vial 1	10.0				884.48				
05102021a_013.d	-----	28241	DBA-CA16	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05102021a_014.d	-----	28241	DBA-CA16	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05102021a_015.d	-----	28304	DBA-CA17	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05102021a_016.d	-----	28304	DBA-CA17	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05102021a_017.d	7.225	687-2M10 0.05 pg/µL		Calibration	Vial 5	10.0				32.99				
05102021a_018.d	7.239	28241CO.01R2	DBA-CA16	Sample	P1-A3	10.0	0.100	10.0	0.1000	70.41	0.0990	0.0099		
05102021a_019.d	7.225	28241CO.01R2	DBA-CA16	Sample	P1-A3	10.0	0.100	10.0	0.1000	74.55	0.1036	0.0104	0.0101	101%
05102021a_020.d	7.239	28241C1.0R2	DBA-CA16	Sample	P1-A4	10.0	0.100	400.0	0.0025	198.03	0.2419	0.9677		
05102021a_021.d	7.212	28241C1.0R2	DBA-CA16	Sample	P1-A4	10.0	0.100	400.0	0.0025	210.31	0.2557	1.0228	0.995	100%
05102021a_022.d	7.225	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				76.45				
05102021a_023.d	7.239	28243	DBC-CA16	Sample	P1-A5	10.0	0.100	10.0	0.1000	84.47	0.1147	0.0115		
05102021a_024.d	7.239	28243	DBC-CA16	Sample	P1-A5	10.0	0.100	10.0	0.1000	76.55	0.1058	0.0106	0.011	
05102021a_025.d	7.239	28244	DBD-CA16	Sample	P1-A6	10.0	0.100	10.0	0.1000	73.60	0.1025	0.0103		
05102021a_026.d	7.225	28244	DBD-CA16	Sample	P1-A6	10.0	0.100	10.0	0.1000	76.94	0.1063	0.0106	0.010	
05102021a_027.d	7.225	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				171.28				
05102021a_028.d	-----	28306	DBC-CA17	Sample	P1-A7	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05102021a_029.d	-----	28306	DBC-CA17	Sample	P1-A7	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05102021a_030.d	-----	28307	DBC-CA17	Sample	P1-A8	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05102021a_031.d	-----	28307	DBC-CA17	Sample	P1-A8	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05102021a_032.d	7.225	687-2M9 0.10 pg/µL		Calibration	Vial 4	10.0				83.30				
05102021a_033.d	-----	28859	DBC-ID181	Sample	P1-A9	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05102021a_034.d	-----	28859	DBC-ID181	Sample	P1-A9	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05102021a_035.d	-----	28860	DBD-ID181	Sample	P1-B1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05102021a_036.d	-----	28860	DBD-ID181	Sample	P1-B1	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05102021a_037.d	7.225	687-2M8 0.25 pg/µL		Calibration	Vial 3	10.0				182.63				
Calculated LOD at 0.045 pg/µL =	22													
Calculated LOQ at 0.10 pg/µL =	71													
"-----" denotes response below area threshold														

**Calibration STD**

D:\MassHunter\Fonicamid\08550_Orion\Data\Results\05102021\05102021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Fonicamid\08550_Orion\Data\Results\05102021\05102021a_011.d	Calibration	1	☒	884.48	1.0000
D:\MassHunter\Fonicamid\08550_Orion\Data\Results\05102021\05102021a_010.d	Calibration	2	☒	430.67	0.5000
D:\MassHunter\Fonicamid\08550_Orion\Data\Results\05102021\05102021a_009.d	Calibration	3	☒	203.42	0.2500
D:\MassHunter\Fonicamid\08550_Orion\Data\Results\05102021\05102021a_008.d	Calibration	3	☒	171.28	0.2500
D:\MassHunter\Fonicamid\08550_Orion\Data\Results\05102021\05102021a_007.d	Calibration	3	☒	182.63	0.2500
D:\MassHunter\Fonicamid\08550_Orion\Data\Results\05102021\05102021a_006.d	Calibration	4	☒	81.28	0.1000
D:\MassHunter\Fonicamid\08550_Orion\Data\Results\05102021\05102021a_005.d	Calibration	4	☒	76.45	0.1000
D:\MassHunter\Fonicamid\08550_Orion\Data\Results\05102021\05102021a_004.d	Calibration	4	☒	83.30	0.1000
D:\MassHunter\Fonicamid\08550_Orion\Data\Results\05102021\05102021a_003.d	Calibration	5	☒	39.74	0.0500
D:\MassHunter\Fonicamid\08550_Orion\Data\Results\05102021\05102021a_002.d	Calibration	5	☒	32.99	0.0500

Compound
Flonicamid

Injection Date
5/19/2021

5/20/21 *asm*

08550 - Flonicamid/Onion - OR405 Field Samples - Extracted 5/19/2021 by ASM

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp (pg/µL)	Conc (pg/µL)	ppm	Ave ppm	% Rec
05192021a_001.d	7.735	Condition		Sample	P1-A3	10.0								
05192021a_002.d	7.721	Condition		Sample	P1-A3	10.0								
05192021a_003.d	7.735	Condition		Sample	P1-A3	10.0								
05192021a_004.d	7.735	Condition		Sample	P1-A3	10.0								
05192021a_005.d	7.735	Condition		Sample	P1-A3	10.0								
05192021a_006.d	7.735	Condition		Sample	P1-A3	10.0								
05192021a_007.d	7.735	Condition		Sample	P1-A3	10.0								
05192021a_008.d	7.735	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				21.83				
05192021a_009.d	7.735	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				33.82				
05192021a_010.d	7.735	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				94.30				
05192021a_011.d	7.735	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				185.81				
05192021a_012.d	7.735	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				371.86				
05192021a_013.d	-----	28265	GA-OR405	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05192021a_014.d	-----	28265	GA-OR405	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
**05192021a_015.d	7.735	28265CO-01R1	GA-OR405	Sample	P1-A2	10.0	0.100	10.0	0.1000	27.70	0.0743	0.0074		
**05192021a_016.d	7.735	28265CO-01R1	GA-OR405	Sample	P1-A2	10.0	0.100	10.0	0.1000	21.62	0.0579	0.0058		
05192021a_017.d	7.735	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				42.64				
05192021a_018.d	7.721	28265CO.01R2	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	28.00	0.0751	0.0075		
05192021a_019.d	7.735	28265CO.01R2	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	31.36	0.0842	0.0084		80%
05192021a_020.d	7.735	28267	GC-OR405	Sample	P1-A4	10.0	0.100	10.0	0.1000	2448.08	6.5989	0.6599		
05192021a_021.d	7.721	28267	GC-OR405	Sample	P1-A4	10.0	0.100	10.0	0.1000	2458.33	6.6266	0.6627		
05192021a_022.d	7.735	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				89.65				
05192021a_023.d	7.735	28268	GB-OR405	Sample	P1-A5	10.0	0.100	10.0	0.1000	2440.35	6.5781	0.6578		
05192021a_024.d	7.735	28268	GB-OR405	Sample	P1-A5	10.0	0.100	10.0	0.1000	2438.41	6.5729	0.6573		
05192021a_025.d	7.748	28267 (Flonicamid)	GC-OR405	Sample	P1-A6	10.0	0.100	400.0	0.0025	63.11	0.1698	0.6792		
05192021a_026.d	7.735	28267 (Flonicamid)	GC-OR405	Sample	P1-A6	10.0	0.100	400.0	0.0025	63.41	0.1706	0.6823	0.68	
05192021a_027.d	7.735	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				35.20				
05192021a_028.d	7.735	28268 (Flonicamid)	GD-OR405	Sample	P1-A7	10.0	0.100	400.0	0.0025	60.32	0.1623	0.6491		
05192021a_029.d	7.735	28268 (Flonicamid)	GD-OR405	Sample	P1-A7	10.0	0.100	400.0	0.0025	71.98	0.1937	0.7748	0.71	
05192021a_030.d	7.735	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				90.55				

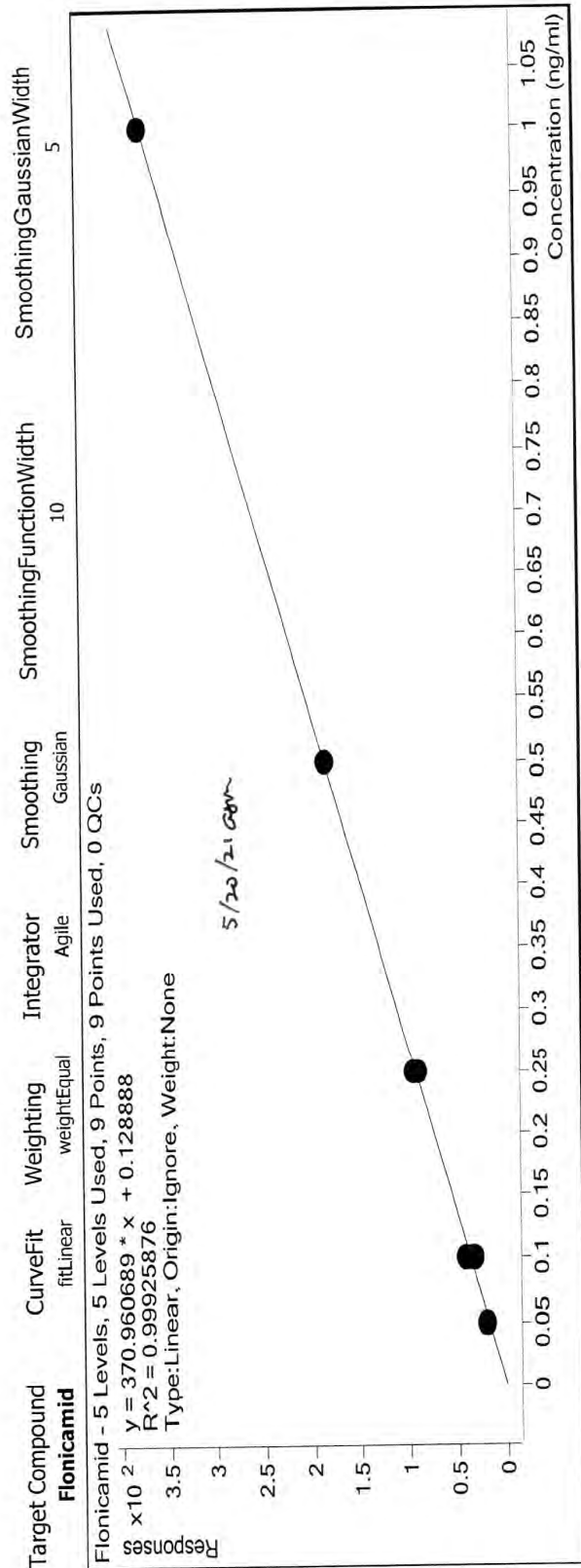
Calculated LOD at 0.045 pg/µL = 17

Calculated LOQ at 0.10 pg/µL = 37

"-----" denotes response below area threshold

Strike through denotes data not used, dilutions used for flonicamid quantitation only

** Denotes data not used, does not meet reproducibility between injections threshold (20%), will reinject



Calibration STD

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05192021\05192021a_012.d
 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05192021\05192021a_011.d
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 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05192021\05192021a_009.d
 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05192021\05192021a_017.d
 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05192021\05192021a_027.d
 D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05192021\05192021a_008.d

Cal Type	Level	Enabled	Response	Exp Conc
Calibration	1	☒	371.86	1.0000
Calibration	2	☒	185.81	0.5000
Calibration	3	☒	94.30	0.2500
Calibration	3	☒	89.65	0.2500
Calibration	3	☒	90.55	0.2500
Calibration	4	☒	33.82	0.1000
Calibration	4	☒	42.64	0.1000
Calibration	4	☒	35.20	0.1000
Calibration	5	☒	21.83	0.0500

Compound
TFNA-AM

Injection Date
5/19/2021

08550 - Flonicamid/Onion - OR405 Field Samples - Extracted 5/19/2021 by ASM

5/20/21 agm

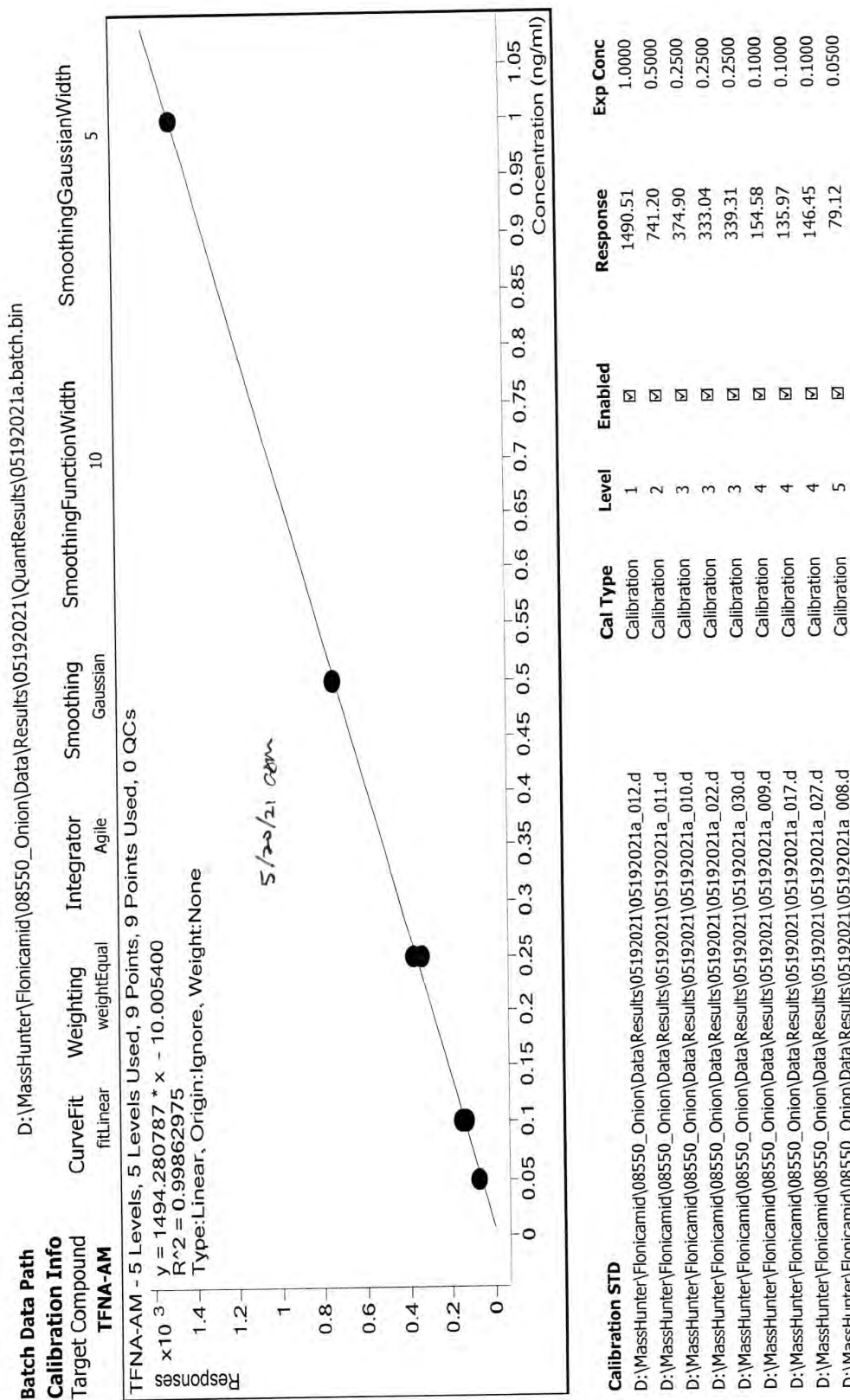
Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05192021a_001.d	5.338	Condition		Sample	P1-A3	10.0								
05192021a_002.d	5.311	Condition		Sample	P1-A3	10.0								
05192021a_003.d	5.324	Condition		Sample	P1-A3	10.0								
05192021a_004.d	5.324	Condition		Sample	P1-A3	10.0								
05192021a_005.d	5.324	Condition		Sample	P1-A3	10.0								
05192021a_006.d	5.324	Condition		Sample	P1-A3	10.0								
05192021a_007.d	5.324	Condition		Sample	P1-A3	10.0								
05192021a_008.d	5.324	Condition		Sample	P1-A3	10.0								
05192021a_009.d	5.324	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				79.12				
05192021a_010.d	5.324	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				154.58				
05192021a_011.d	5.311	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				374.90				
05192021a_012.d	5.311	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				741.20				
05192021a_013.d	5.297	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				1490.51				
05192021a_014.d	-----	28265	GA-OR405	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05192021a_015.d	5.324	28265C0.01R1	GA-OR405	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05192021a_016.d	5.324	28265C0.01R1	GA-OR405	Sample	P1-A2	10.0	0.100	10.0	0.1000	121.99	0.0883	0.0088		
05192021a_017.d	5.324	28265C0.01R1	GA-OR405	Sample	P1-A2	10.0	0.100	10.0	0.1000	132.67	0.0955	0.0095	0.00919	92%
05192021a_018.d	5.324	687-2M15 0.10 pg/µL	GA-CA*20	Calibration	Vial 4	10.0				135.97				
05192021a_019.d	5.324	28265C0.01R2	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	125.63	0.0908	0.0091		
05192021a_020.d	5.324	28267	GC-OR405	Sample	P1-A3	10.0	0.100	10.0	0.1000	117.71	0.0855	0.0085	0.00881	88%
05192021a_021.d	5.311	28267	GC-OR405	Sample	P1-A4	10.0	0.100	10.0	0.1000	204.85	0.1438	0.0144		
05192021a_022.d	5.324	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				209.86	0.1471	0.0147	0.015	
05192021a_023.d	5.324	28268	GD-OR405	Sample	P1-A5	10.0	0.100	10.0	0.1000	333.04	0.1175	0.0118		
05192021a_024.d	5.324	28268	GD-OR405	Sample	P1-A5	10.0	0.100	10.0	0.1000	165.63	0.1160	0.0116	0.012	
05192021a_025.d	-----	28267 (Flonicamid)		Sample	P1-A6	10.0	0.100	400.0	0.0025	-----	<0.10	<0.01		
05192021a_026.d	-----	28267 (Flonicamid)		Sample	P1-A6	10.0	0.100	400.0	0.0025	-----	<0.10	<0.01		
05192021a_027.d	5.338	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				146.45				
05192021a_028.d	-----	28268 (Flonicamid)		Sample	P1-A7	10.0	0.100	400.0	0.0025	-----	<0.10	<0.01		
05192021a_029.d	-----	28268 (Flonicamid)		Sample	P1-A7	10.0	0.100	400.0	0.0025	-----	<0.10	<0.01		
05192021a_030.d	5.324	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				339.31				

Calculated LOD at 0.045 pg/µL = 57

Calculated LOQ at 0.10 pg/µL = 139

"-----" denotes response below area threshold

Strikethrough denotes data not used, dilutions used for flonicamid quantitation only



Compound Injection Date
TFNA 5/19/2021

08550 - Flonicamid/Onion - OR405 Field Samples - Extracted 5/19/2021 by ASM

5/20/21 *asm*

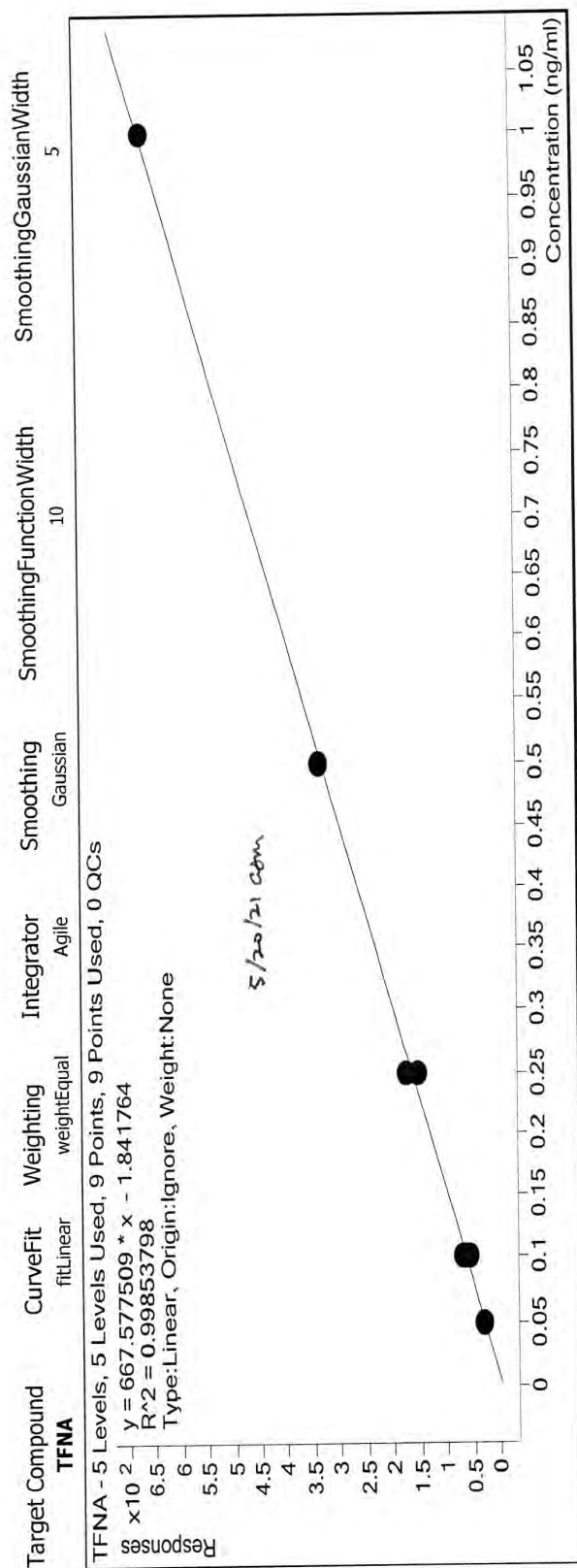
Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05192021a_001.d	6.024	Condition		Sample	P1-A3	10.0								
05192021a_002.d	6.010	Condition		Sample	P1-A3	10.0								
05192021a_003.d	6.024	Condition		Sample	P1-A3	10.0								
05192021a_004.d	6.010	Condition		Sample	P1-A3	10.0								
05192021a_005.d	6.010	Condition		Sample	P1-A3	10.0								
05192021a_006.d	6.010	Condition		Sample	P1-A3	10.0								
05192021a_007.d	6.010	Condition		Sample	P1-A3	10.0								
05192021a_008.d	5.997	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				34.11				
05192021a_009.d	5.997	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				71.11				
05192021a_010.d	5.983	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				177.54				
05192021a_011.d	5.983	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				335.73				
05192021a_012.d	5.983	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				665.56				
05192021a_013.d	-----	28265	GA-OR405	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05192021a_014.d	-----	28265	GA-OR405	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05192021a_015.d	6.024	28265CO.01R1		Sample	P1-A2	10.0	0.100	10.0	0.1000	58.35	0.0902	0.0090		
05192021a_016.d	6.010	28265CO.01R1		Sample	P1-A2	10.0	0.100	10.0	0.1000	54.69	0.0847	0.0085	0.00874	87%
05192021a_017.d	5.997	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				59.04				
05192021a_018.d	6.010	28265CO.01R2	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	58.68	0.0907	0.0091		
05192021a_019.d	6.010	28265CO.01R2	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	59.78	0.0923	0.0092	0.00915	91%
05192021a_020.d	6.010	28267	GC-OR405	Sample	P1-A4	10.0	0.100	10.0	0.1000	82.44	0.1262	0.0126		
05192021a_021.d	5.997	28267	GC-OR405	Sample	P1-A4	10.0	0.100	10.0	0.1000	90.33	0.1381	0.0138	0.013	
05192021a_022.d	5.983	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				153.58				
05192021a_023.d	6.010	28268	GD-OR405	Sample	P1-A5	10.0	0.100	10.0	0.1000	88.19	0.1349	0.0135		
05192021a_024.d	6.010	28268	GD-OR405	Sample	P1-A5	10.0	0.100	10.0	0.1000	96.58	0.1474	0.0147	0.014	
05192021a_025.d	-----	28267 (Flonicamid)		Sample	P1-A6	10.0	0.100	400.0	0.0025		<0.10	<0.01		
05192021a_026.d	-----	28267 (Flonicamid)		Sample	P1-A6	10.0	0.100	400.0	0.0025		<0.10	<0.01		
05192021a_027.d	5.997	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				66.61				
05192021a_028.d	-----	28268 (Flonicamid)		Sample	P1-A7	10.0	0.100	400.0	0.0025		<0.10	<0.01		
05192021a_029.d	-----	28268 (Flonicamid)		Sample	P1-A7	10.0	0.100	400.0	0.0025		<0.10	<0.01		
05192021a_030.d	5.997	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				155.84				

Calculated LOD at 0.045 pg/µL = 28

Calculated LOQ at 0.10 pg/µL = 65

"-----" denotes response below area threshold

Strikethrough denotes data not used, dilutions used for flonicamid quantitation only

**Calibration STD**

D:\MassHunter\FI\onion\08550_Onion\Data\Results\05192021\05192021a_012.d	Calibration	Level	1	Enabled	☒	Response	665.56	Exp Conc	1.0000
D:\MassHunter\FI\onion\08550_Onion\Data\Results\05192021\05192021a_011.d	Calibration	Level	2	Enabled	☒	Response	335.73	Exp Conc	0.5000
D:\MassHunter\FI\onion\08550_Onion\Data\Results\05192021\05192021a_010.d	Calibration	Level	3	Enabled	☒	Response	177.54	Exp Conc	0.2500
D:\MassHunter\FI\onion\08550_Onion\Data\Results\05192021\05192021a_022.d	Calibration	Level	3	Enabled	☒	Response	153.58	Exp Conc	0.2500
D:\MassHunter\FI\onion\08550_Onion\Data\Results\05192021\05192021a_030.d	Calibration	Level	3	Enabled	☒	Response	155.84	Exp Conc	0.2500
D:\MassHunter\FI\onion\08550_Onion\Data\Results\05192021\05192021a_009.d	Calibration	Level	4	Enabled	☒	Response	71.11	Exp Conc	0.1000
D:\MassHunter\FI\onion\08550_Onion\Data\Results\05192021\05192021a_017.d	Calibration	Level	4	Enabled	☒	Response	59.04	Exp Conc	0.1000
D:\MassHunter\FI\onion\08550_Onion\Data\Results\05192021\05192021a_027.d	Calibration	Level	4	Enabled	☒	Response	66.61	Exp Conc	0.1000
D:\MassHunter\FI\onion\08550_Onion\Data\Results\05192021\05192021a_008.d	Calibration	Level	5	Enabled	☒	Response	34.11	Exp Conc	0.0500

Compound Injection Date
TFNG 5/19/2021

08550 - Flonicamid/Onion - OR405 Field Samples - Extracted 5/19/2021 by ASM

5/20/21 *ASM*

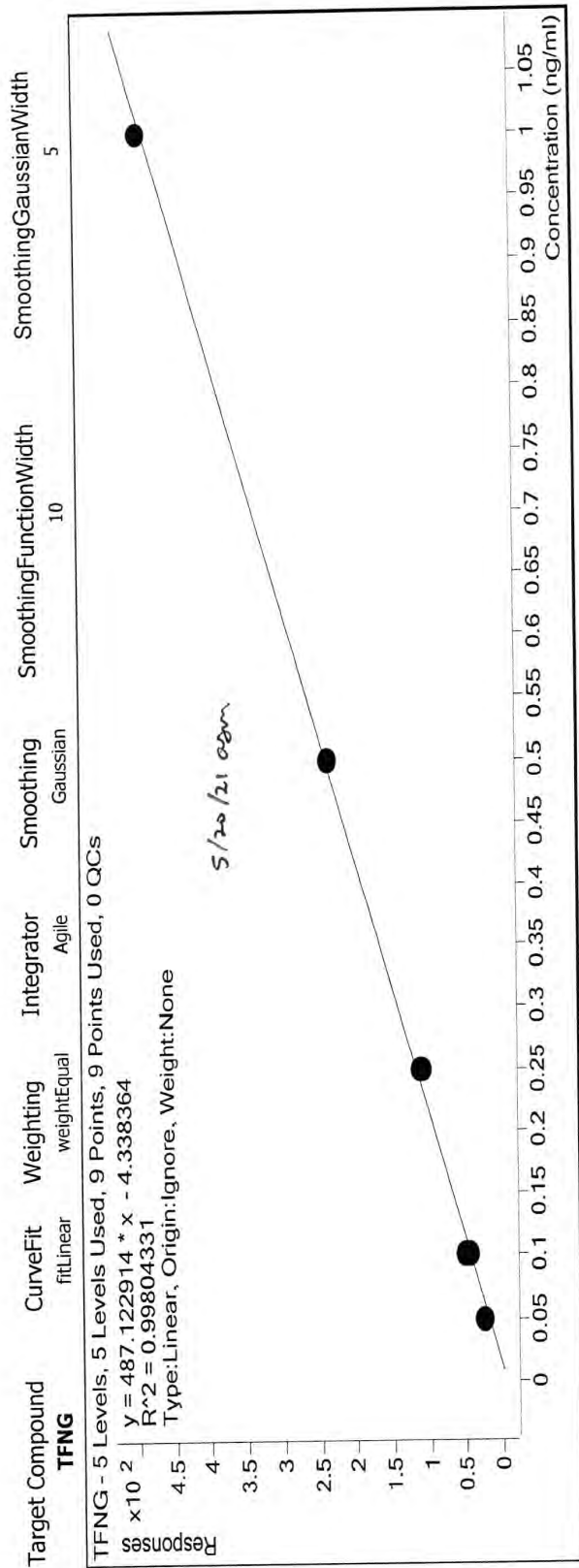
Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp (pg/µL)	Conc (pg/µL)	ppm	Ave ppm	% Rec
05192021a_001.d	7.225	Condition		Sample	P1-A3	10.0								
05192021a_002.d	7.212	Condition		Sample	P1-A3	10.0								
05192021a_003.d	7.225	Condition		Sample	P1-A3	10.0								
05192021a_004.d	7.212	Condition		Sample	P1-A3	10.0								
05192021a_005.d	7.225	Condition		Sample	P1-A3	10.0								
05192021a_006.d	7.212	Condition		Sample	P1-A3	10.0								
05192021a_007.d	7.212	Condition		Sample	P1-A3	10.0								
05192021a_008.d	7.212	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				27.76				
05192021a_009.d	7.225	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				48.85				
05192021a_010.d	7.212	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				108.81				
05192021a_011.d	7.212	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				235.23				
05192021a_012.d	7.198	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				488.50				
05192021a_013.d	-----	28265	GA-OR405	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05192021a_014.d	-----	28265	GA-OR405	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05192021a_015.d	7.225	28265CO.01R1	GA-OR405	Sample	P1-A2	10.0	0.100	10.0	0.1000	44.27	0.0998	0.0100		
05192021a_016.d	7.212	28265CO.01R1	GA-OR405	Sample	P1-A2	10.0	0.100	10.0	0.1000	44.46	0.1002	0.0100	0.0100	100%
05192021a_017.d	7.212	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				44.69				
05192021a_018.d	7.212	28265CO.01R2	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	40.43	0.0919	0.0092		
05192021a_019.d	7.212	28265CO.01R2	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	39.00	0.0890	0.0089	0.00904	90%
05192021a_020.d	-----	28267	GC-OR405	Sample	P1-A4	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05192021a_021.d	-----	28267	GC-OR405	Sample	P1-A4	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05192021a_022.d	7.212	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				109.34				
05192021a_023.d	-----	28268	GD-OR405	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05192021a_024.d	-----	28268	GD-OR405	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05192021a_025.d	-----	28267 (Flonicamid)	GC-OR405	Sample	P1-A6	10.0	0.100	400.0	0.0025	-----	<0.10	<0.01		
05192021a_026.d	-----	28267 (Flonicamid)	GC-OR405	Sample	P1-A6	10.0	0.100	400.0	0.0025	-----	<0.10	<0.01		
05192021a_027.d	7.225	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				51.20				
05192021a_028.d	-----	28268 (Flonicamid)	GD-OR405	Sample	P1-A7	10.0	0.100	400.0	0.0025	-----	<0.10	<0.01		
05192021a_029.d	-----	28268 (Flonicamid)	GD-OR405	Sample	P1-A7	10.0	0.100	400.0	0.0025	-----	<0.10	<0.01		
05192021a_030.d	7.225	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				113.08				

Calculated LOD at 0.045 pg/µL = 18

Calculated LOQ at 0.10 pg/µL = 44

"-----" denotes response below area threshold

Strikethrough denotes data not used, dilutions used for flonicamid quantitation only



Calibration STD

D:\MassHunter\F_\lonicamid\08550_Onion\Data\Results\05192021\05192021a_012.d
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 D:\MassHunter\F_\lonicamid\08550_Onion\Data\Results\05192021\05192021a_008.d

Compound Injection Date
Flonicamid 5/20/2021

08550 - Flonicamid/Onion - OR405 Sample Reinjection - Extracted 5/19/2021 by ASM

5/20/21 *asm*

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05202021a_001.d	-----	Condition		Sample	P1-A2	10.0								
05202021a_002.d	7.721	Condition		Sample	P1-A2	10.0								
05202021a_003.d	7.735	Condition		Sample	P1-A2	10.0								
05202021a_004.d	7.735	Condition		Sample	P1-A2	10.0								
05202021a_005.d	7.735	Condition		Sample	P1-A2	10.0								
05202021a_006.d	7.735	Condition		Sample	P1-A2	10.0								
05202021a_007.d	7.735	Condition		Sample	P1-A2	10.0								
05202021a_008.d	7.721	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				20.79				
05202021a_009.d	7.735	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				39.47				
05202021a_010.d	7.735	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				93.27				
05202021a_011.d	7.735	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				189.99				
05202021a_012.d	7.735	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				377.88				
*05202021a_013.d	-----	28265	GA-OR405	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
*05202021a_014.d	-----	28265	GA-OR405	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05202021a_015.d	7.735	28265C0.01R1	GA-OR405	Sample	P1-A2	10.0	0.100	10.0	0.1000	32.98	0.0902	0.0090		
05202021a_016.d	7.735	28265C0.01R1	GA-OR405	Sample	P1-A2	10.0	0.100	10.0	0.1000	33.79	0.0923	0.0092	0.00913	91%
05202021a_017.d	7.735	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				33.46				
*05202021a_018.d	7.721	28265C0.01R2	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	29.27	0.0804	0.0080		
*05202021a_019.d	7.735	28265C0.01R2	GA-CA*20	Sample	P1-A3	10.0	0.100	10.0	0.1000	34.71	0.0948	0.0095	0.00876	
05202021a_020.d	7.721	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				89.35				

Calculated LOD at 0.045 pg/µL = 16

Calculated LOQ at 0.10 pg/µL = 37

"-----" denotes response below area threshold

*Data previously reported

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05202021\QuantResults\05202021a_batch.bin

Batch Data Path**Calibration Info**

Target Compound

Flonicamid

CurveFit

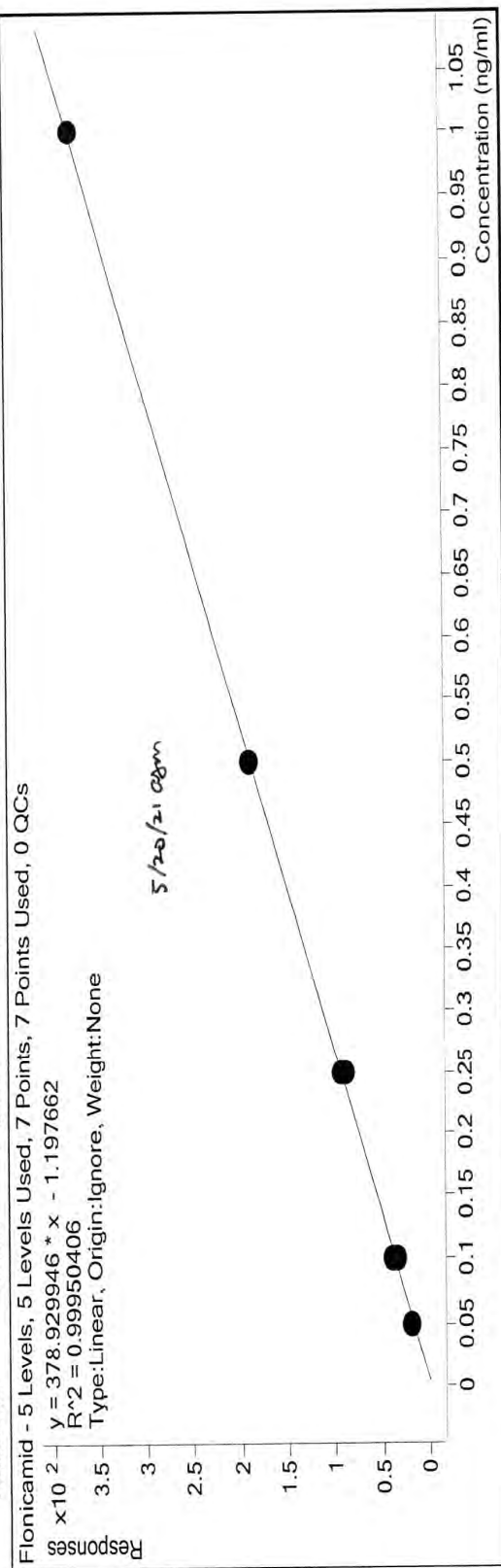
Weighting

Integrator

Smoothing

SmoothingFunctionWidth

SmoothingGaussianWidth

**Calibration STD**

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D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05202021\05202021a_009.d

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05202021\05202021a_017.d

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05202021\05202021a_008.d

Cal Type	Level	Enabled	Response	Exp Conc
Calibration	1	☒	377.88	1.0000
Calibration	2	☒	189.99	0.5000
Calibration	3	☒	93.27	0.2500
Calibration	3	☒	89.35	0.2500
Calibration	4	☒	39.47	0.1000
Calibration	4	☒	33.46	0.1000
Calibration	5	☒	20.79	0.0500

Compound **Flonicamid** Injection Date **5/24/2021**

08550 - Flonicamid/Onion - CA18 & CA*20 Field Samples - Extracted 5/24/2021 by ASM

5/25/21 QRM

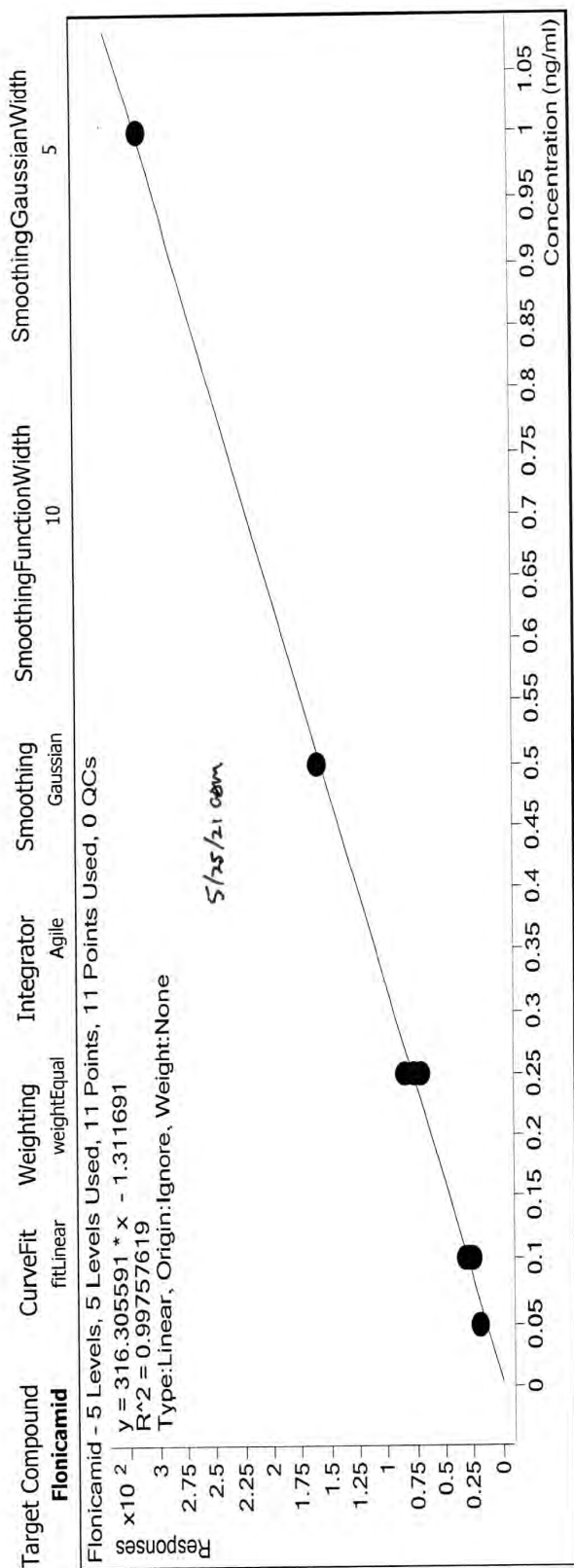
Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05242021a_001.d	7.450	Condition		Sample	P1-A3	10.0								
05242021a_002.d	7.735	Condition		Sample	P1-A3	10.0								
05242021a_003.d	7.721	Condition		Sample	P1-A3	10.0								
05242021a_004.d	7.721	Condition		Sample	P1-A3	10.0								
05242021a_005.d	7.721	Condition		Sample	P1-A3	10.0								
05242021a_006.d	7.735	Condition		Sample	P1-A3	10.0								
05242021a_007.d	7.735	Condition		Sample	P1-A3	10.0								
05242021a_008.d	7.735	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				20.94				
05242021a_009.d	7.735	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				29.50				
05242021a_010.d	7.735	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				84.25				
05242021a_011.d	7.735	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				159.89				
05242021a_012.d	7.721	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				314.53				
05242021a_013.d	-----	28225	GA-CA18	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05242021a_014.d	-----	28225	GA-CA18	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05242021a_015.d	7.721	28225C0.01R3	GA-CA18	Sample	P1-A2	10.0	0.100	10.0	0.1000	26.81	0.0889	0.0089		
05242021a_016.d	7.721	28225C0.01R3	GA-CA18	Sample	P1-A2	10.0	0.100	10.0	0.1000	30.35	0.1001	0.0100	0.00945	94%
05242021a_017.d	7.735	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				26.46				
05242021a_018.d	7.735	28225C0.10R1	GA-CA18	Sample	P1-A3	10.0	0.100	40.0	0.0250	68.46	0.2206	0.0882		
05242021a_019.d	7.721	28225C0.10R1	GA-CA18	Sample	P1-A3	10.0	0.100	40.0	0.0250	74.43	0.2395	0.0958	0.0970	92%
05242021a_020.d	7.735	28227	GC-CA18	Sample	P1-A4	10.0	0.100	10.0	0.1000	3609.75	11.4164	1.4416		
05242021a_021.d	7.735	28227	GC-CA18	Sample	P1-A4	10.0	0.100	10.0	0.1000	3623.72	11.4605	1.4461		
05242021a_022.d	7.735	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				76.33				
05242021a_023.d	7.735	28228	GD-CA18	Sample	P1-A5	10.0	0.100	10.0	0.1000	3925.79	12.4155	1.2416		
05242021a_024.d	7.735	28228	GD-CA18	Sample	P1-A5	10.0	0.100	10.0	0.1000	3947.29	12.4835	1.2483		
05242021a_025.d	7.735	28851	GC-CA*20	Sample	P1-A6	10.0	0.100	10.0	0.1000	2328.99	7.3672	0.7367		
05242021a_026.d	7.735	28851	GC-CA*20	Sample	P1-A6	10.0	0.100	10.0	0.1000	2393.34	7.5707	0.7571		
05242021a_027.d	7.735	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				32.43				
05242021a_028.d	7.735	28852	GD-CA*20	Sample	P1-A7	10.0	0.100	10.0	0.1000	2984.82	9.4407	0.9441		
05242021a_029.d	7.735	28852	GD-CA*20	Sample	P1-A7	10.0	0.100	10.0	0.1000	2965.69	9.3802	0.9380		
05242021a_030.d	7.735	28227 (Flonicamid)	GC-CA18	Sample	P1-A8	10.0	0.100	200.0	0.0050	203.41	0.6472	1.2945		
05242021a_031.d	7.735	28227 (Flonicamid)	GC-CA18	Sample	P1-A8	10.0	0.100	200.0	0.0050	214.64	0.6827	1.3655	1.3	
05242021a_032.d	7.735	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				77.04				
05242021a_033.d	7.735	28228 (Flonicamid)	GD-CA18	Sample	P1-A9	10.0	0.100	200.0	0.0050	219.29	0.6974	1.3949		
05242021a_034.d	7.735	28228 (Flonicamid)	GD-CA18	Sample	P1-A9	10.0	0.100	200.0	0.0050	216.28	0.6879	1.3758	1.4	
05242021a_035.d	7.735	28851 (Flonicamid)	GC-CA*20	Sample	P1-B1	10.0	0.100	200.0	0.0050	129.24	0.4127	0.8255		
05242021a_036.d	7.735	28851 (Flonicamid)	GC-CA*20	Sample	P1-B1	10.0	0.100	200.0	0.0050	140.20	0.4474	0.8948	0.86	
05242021a_037.d	7.735	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				27.05				
05242021a_038.d	7.735	28852 (Flonicamid)	GD-CA*20	Sample	P1-B2	10.0	0.100	200.0	0.0050	157.94	0.5035	1.0069		
05242021a_039.d	7.735	28852 (Flonicamid)	GD-CA*20	Sample	P1-B2	10.0	0.100	200.0	0.0050	172.57	0.5497	1.0995	1.1	
05242021a_040.d	7.735	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				70.26				

Calculated LOD at 0.045 pg/µL = 13

Calculated LOQ at 0.10 pg/µL = 30

"-----" denotes response below area threshold

Strikethrough denotes data not used, dilutions used for flonicamid quantitation only

**Calibration STD**

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_011.d	Calibration	1	☒	314.53	1.0000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_010.d	Calibration	2	☒	159.89	0.5000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_010.d	Calibration	3	☒	84.25	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_022.d	Calibration	3	☒	76.33	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_032.d	Calibration	3	☒	77.04	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_040.d	Calibration	3	☒	70.26	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_009.d	Calibration	4	☒	29.50	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_017.d	Calibration	4	☒	26.46	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_027.d	Calibration	4	☒	32.43	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_037.d	Calibration	4	☒	27.05	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_008.d	Calibration	5	☒	20.94	0.0500

Compound Injection Date
TFNA-AM 5/24/2021

08550 - Flonicamid/Onion - CA18 & CA*20 Field Samples - Extracted 5/24/2021 by ASM

5/25/21 ASM

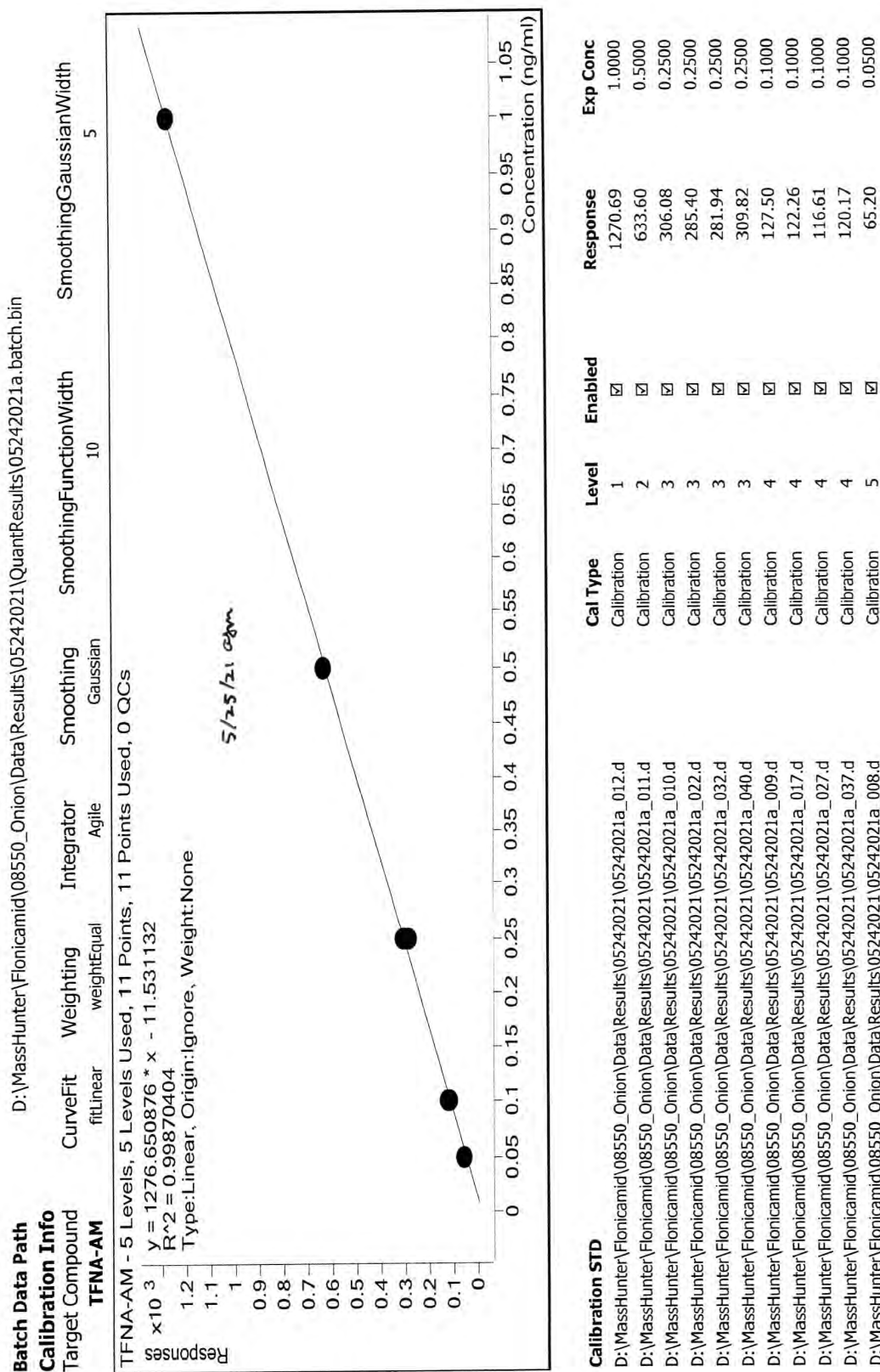
Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	ml Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05242021a_001.d	4.931	Condition		Sample	P1-A3	10.0								
05242021a_002.d	5.324	Condition		Sample	P1-A3	10.0								
05242021a_003.d	5.324	Condition		Sample	P1-A3	10.0								
05242021a_004.d	5.324	Condition		Sample	P1-A3	10.0								
05242021a_005.d	5.324	Condition		Sample	P1-A3	10.0								
05242021a_006.d	5.311	Condition		Sample	P1-A3	10.0								
05242021a_007.d	5.324	Condition		Sample	P1-A3	10.0								
05242021a_008.d	5.324	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				65.20				
05242021a_009.d	5.324	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				127.50				
05242021a_010.d	5.324	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				306.08				
05242021a_011.d	5.324	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				633.60				
05242021a_012.d	5.297	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				1270.69				
05242021a_013.d	-----	28225	GA-CA18	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05242021a_014.d	-----	28225	GA-CA18	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05242021a_015.d	5.324	28225CO.01R3	GA-CA18	Sample	P1-A2	10.0	0.100	10.0	0.1000	115.43	0.0994	0.0099		
05242021a_016.d	5.324	28225CO.01R3	GA-CA18	Sample	P1-A2	10.0	0.100	10.0	0.1000	108.90	0.0943	0.0094	0.00969	97%
05242021a_017.d	5.324	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				122.26				
05242021a_018.d	5.311	28225CO.10R1	GA-CA18	Sample	P1-A3	10.0	0.100	40.0	0.0250	284.99	0.2323	0.0929		
05242021a_019.d	5.324	28225CO.10R1	GA-CA18	Sample	P1-A3	10.0	0.100	40.0	0.0250	286.38	0.2334	0.0933	0.0931	93%
05242021a_020.d	5.324	28227	GC-CA18	Sample	P1-A4	10.0	0.100	10.0	0.1000	535.38	0.4284	0.0428		
05242021a_021.d	5.311	28227	GC-CA18	Sample	P1-A4	10.0	0.100	10.0	0.1000	547.14	0.4376	0.0438	0.043	
05242021a_022.d	5.311	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				285.40				
05242021a_023.d	5.324	28228	GD-CA18	Sample	P1-A5	10.0	0.100	10.0	0.1000	616.44	0.4919	0.0492		
05242021a_024.d	5.324	28228	GD-CA18	Sample	P1-A5	10.0	0.100	10.0	0.1000	626.20	0.4995	0.0500	0.050	
05242021a_025.d	5.324	28851	GC-CA*20	Sample	P1-A6	10.0	0.100	10.0	0.1000	178.16	0.1486	0.0149		
05242021a_026.d	5.324	28851	GC-CA*20	Sample	P1-A6	10.0	0.100	10.0	0.1000	181.03	0.1508	0.0151	0.015	
05242021a_027.d	5.324	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				116.61				
05242021a_028.d	5.324	28852	GD-CA*20	Sample	P1-A7	10.0	0.100	10.0	0.1000	270.78	0.2211	0.0221		
05242021a_029.d	5.324	28852	GD-CA*20	Sample	P1-A7	10.0	0.100	10.0	0.1000	277.44	0.2264	0.0226	0.022	
05242021a_030.d	-----	28227 (Flonicamid)	GC-CA18	Sample	P1-A8	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_031.d	-----	28227 (Flonicamid)	GC-CA18	Sample	P1-A8	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_032.d	5.324	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				281.94				
05242021a_033.d	-----	28228 (Flonicamid)	GD-CA18	Sample	P1-A9	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_034.d	-----	28228 (Flonicamid)	GD-CA18	Sample	P1-A9	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_035.d	-----	28851 (Flonicamid)	GC-CA*20	Sample	P1-B1	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_036.d	-----	28851 (Flonicamid)	GC-CA*20	Sample	P1-B1	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_037.d	5.324	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				120.17				
05242021a_038.d	-----	28852 (Flonicamid)	GD-CA*20	Sample	P1-B2	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_039.d	-----	28852 (Flonicamid)	GD-CA*20	Sample	P1-B2	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_040.d	5.324	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				309.82				

Calculated LOD at 0.045 pg/µL = 46

Calculated LOQ at 0.10 pg/µL = 116

"-----" denotes response below area threshold

Strikethrough denotes data not used, dilutions used for flonicamid quantitation only



Compound Injection Date
TFNA 5/24/2021

08550 - Flonicamid/Onion - CA18 & CA*20 Field Samples - Extracted 5/24/2021 by ASM

5/25/21 *ASM*

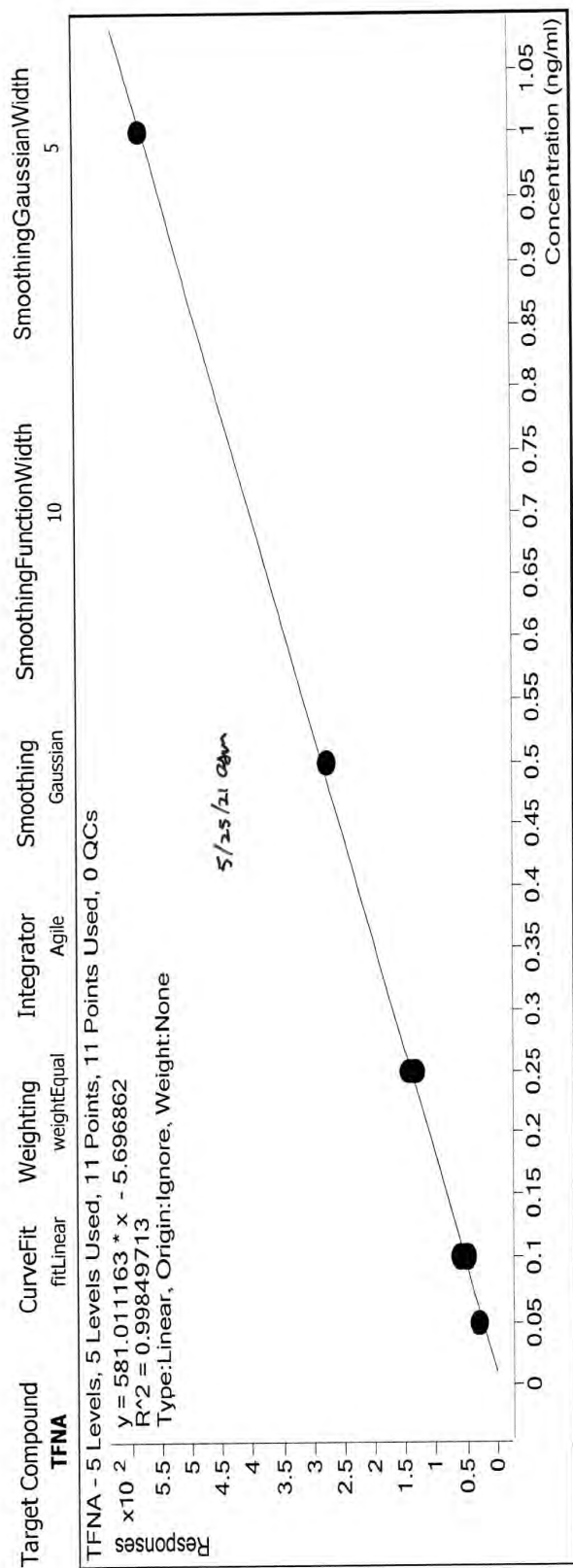
Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05242021a_001.d	-----	Condition		Sample	P1-A3	10.0								
05242021a_002.d	5.983	Condition		Sample	P1-A3	10.0								
05242021a_003.d	5.983	Condition		Sample	P1-A3	10.0								
05242021a_004.d	5.983	Condition		Sample	P1-A3	10.0								
05242021a_005.d	5.983	Condition		Sample	P1-A3	10.0								
05242021a_006.d	5.983	Condition		Sample	P1-A3	10.0								
05242021a_007.d	5.983	Condition		Sample	P1-A3	10.0								
05242021a_008.d	5.969	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				31.15				
05242021a_009.d	5.983	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				61.38				
05242021a_010.d	5.969	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				142.31				
05242021a_011.d	5.969	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				276.05				
05242021a_012.d	5.956	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				581.81				
05242021a_013.d	-----	28225	GA-CA18	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05242021a_014.d	5.997	28225	GA-CA18	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05242021a_015.d	5.997	28225CO.01R3	GA-CA18	Sample	P1-A2	10.0	0.100	10.0	0.1000	52.56	0.1003	0.0100		
05242021a_016.d	5.997	28225CO.01R3	GA-CA18	Sample	P1-A2	10.0	0.100	10.0	0.1000	46.36	0.0896	0.0090	0.00949	95%
05242021a_017.d	5.969	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				52.86				
05242021a_018.d	5.983	28225CO.10R1	GA-CA18	Sample	P1-A3	10.0	0.100	10.0	0.0250	123.91	0.2231	0.0892		
05242021a_019.d	5.997	28225CO.10R1	GA-CA18	Sample	P1-A3	10.0	0.100	10.0	0.0250	126.83	0.2281	0.0912	0.0902	90%
05242021a_020.d	5.997	28227	GC-CA18	Sample	P1-A4	10.0	0.100	10.0	0.1000	167.74	0.2985	0.0299		
05242021a_021.d	5.983	28227	GC-CA18	Sample	P1-A4	10.0	0.100	10.0	0.1000	165.23	0.2942	0.0294	0.030	
05242021a_022.d	5.969	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				130.89				
05242021a_023.d	5.997	28228	GD-CA18	Sample	P1-A5	10.0	0.100	10.0	0.1000	209.86	0.3710	0.0371		
05242021a_024.d	5.997	28228	GD-CA18	Sample	P1-A5	10.0	0.100	10.0	0.1000	225.62	0.3981	0.0398	0.038	
05242021a_025.d	5.997	28851	GC-CA*20	Sample	P1-A6	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05242021a_026.d	5.983	28851	GC-CA*20	Sample	P1-A6	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05242021a_027.d	5.983	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				53.41				
05242021a_028.d	5.983	28852	GD-CA*20	Sample	P1-A7	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05242021a_029.d	5.983	28852	GD-CA*20	Sample	P1-A7	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05242021a_030.d	5.969	28227 (Flonicamid)	GC-CA18	Sample	P1-A8	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_031.d	5.969	28227 (Flonicamid)	GC-CA18	Sample	P1-A8	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_032.d	5.969	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				137.37				
05242021a_033.d	5.983	28228 (Flonicamid)	GD-CA18	Sample	P1-A9	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_034.d	5.983	28228 (Flonicamid)	GD-CA18	Sample	P1-A9	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_035.d	5.983	28851 (Flonicamid)	GC-CA*20	Sample	P1-B1	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_036.d	5.983	28851 (Flonicamid)	GC-CA*20	Sample	P1-B1	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_037.d	5.983	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				49.42				
05242021a_038.d	5.983	28852 (Flonicamid)	GD-CA*20	Sample	P1-B2	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_039.d	5.983	28852 (Flonicamid)	GD-CA*20	Sample	P1-B2	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_040.d	5.983	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				134.67				

Calculated LOD at 0.045 pg/µL = 20

Calculated LOQ at 0.10 pg/µL = 52

"-----" denotes response below area threshold

Strikethrough denotes data not used, dilutions used for flonicamid quantitation only

**Calibration STD**

D:\MassHunter\FI\onion\08550_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\FI\onion\08550_011.d	Calibration	1	☒	581.81	1.0000
D:\MassHunter\FI\onion\08550_010.d	Calibration	2	☒	276.05	0.5000
D:\MassHunter\FI\onion\08550_022.d	Calibration	3	☒	142.31	0.2500
D:\MassHunter\FI\onion\08550_032.d	Calibration	3	☒	130.89	0.2500
D:\MassHunter\FI\onion\08550_040.d	Calibration	3	☒	137.37	0.2500
D:\MassHunter\FI\onion\08550_009.d	Calibration	3	☒	134.67	0.2500
D:\MassHunter\FI\onion\08550_017.d	Calibration	4	☒	61.38	0.1000
D:\MassHunter\FI\onion\08550_027.d	Calibration	4	☒	52.86	0.1000
D:\MassHunter\FI\onion\08550_037.d	Calibration	4	☒	53.41	0.1000
D:\MassHunter\FI\onion\08550_008.d	Calibration	4	☒	49.42	0.1000
D:\MassHunter\FI\onion\08550_008.d	Calibration	5	☒	31.15	0.0500

Compound Injection Date
TFNG 5/24/2021

08550 - Flonicamid/Onion - CA18 & CA*20 Field Samples - Extracted 5/24/2021 by ASM

5/25/21 *asm*

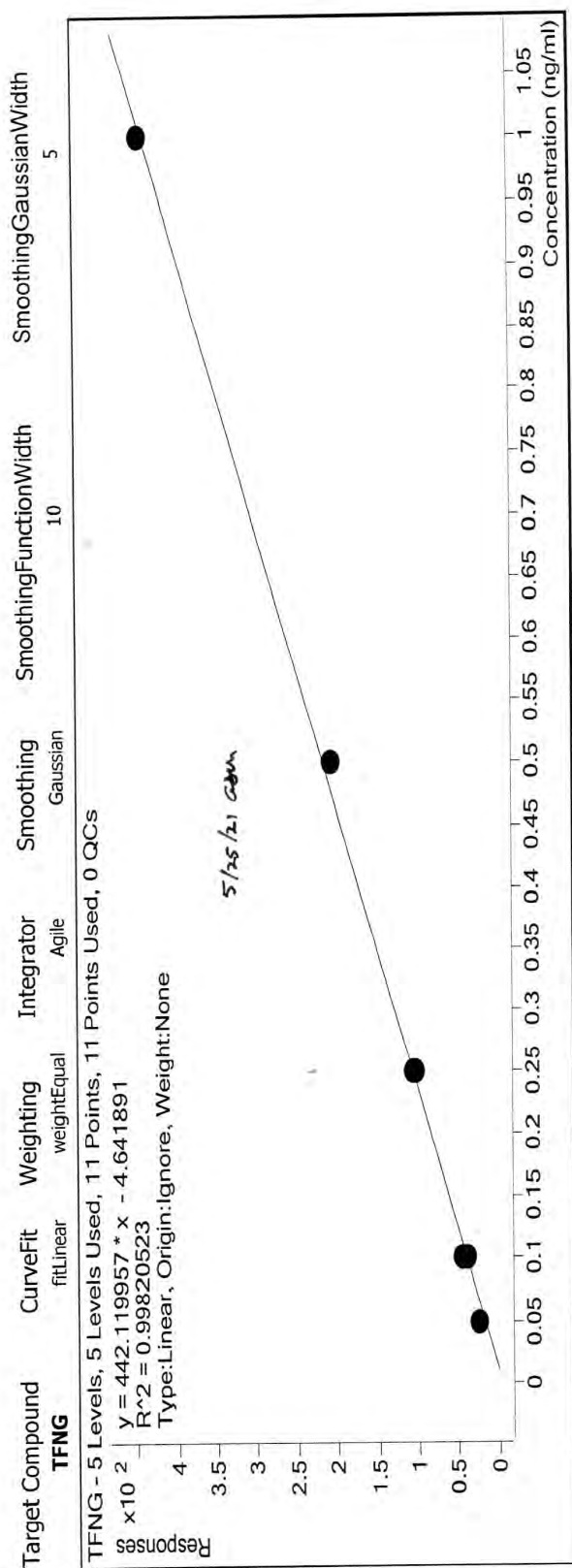
Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05242021a_001.d	6.832	Condition		Sample	P1-A3	10.0								
05242021a_002.d	7.212	Condition		Sample	P1-A3	10.0								
05242021a_003.d	7.212	Condition		Sample	P1-A3	10.0								
05242021a_004.d	7.212	Condition		Sample	P1-A3	10.0								
05242021a_005.d	7.212	Condition		Sample	P1-A3	10.0								
05242021a_006.d	7.212	Condition		Sample	P1-A3	10.0								
05242021a_007.d	7.212	Condition		Sample	P1-A3	10.0								
05242021a_008.d	7.212	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				26.18				
05242021a_009.d	7.212	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				43.50				
05242021a_010.d	7.212	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				106.62				
05242021a_011.d	7.212	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				207.33				
05242021a_012.d	7.198	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				443.86				
05242021a_013.d	-----	28225	GA-CA18	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05242021a_014.d	-----	28225	GA-CA18	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05242021a_015.d	7.212	28225CO.01R3	GA-CA18	Sample	P1-A2	10.0	0.100	10.0	0.1000	42.55	0.1067	0.1017		
05242021a_016.d	7.212	28225CO.01R3	GA-CA18	Sample	P1-A2	10.0	0.100	10.0	0.1000	35.00	0.0897	0.0090	0.00982	98%
05242021a_017.d	7.212	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				40.25				
05242021a_018.d	7.212	28225CO.10R1	GA-CA18	Sample	P1-A3	10.0	0.100	40.0	0.0250	114.71	0.2700	0.1080		
05242021a_019.d	7.212	28225CO.10R1	GA-CA18	Sample	P1-A3	10.0	0.100	40.0	0.0250	102.80	0.2430	0.0972	0.103	103%
05242021a_020.d	7.212	28227	GC-CA18	Sample	P1-A4	10.0	0.100	10.0	0.1000	195.00	0.4516	0.0452		
05242021a_021.d	7.212	28227	GC-CA18	Sample	P1-A4	10.0	0.100	10.0	0.1000	202.19	0.4678	0.0468	0.046	
05242021a_022.d	7.212	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				101.25				
05242021a_023.d	7.212	28228	GD-CA18	Sample	P1-A5	10.0	0.100	10.0	0.1000	229.52	0.5296	0.0530		
05242021a_024.d	7.212	28228	GD-CA18	Sample	P1-A5	10.0	0.100	10.0	0.1000	241.50	0.5567	0.0557	0.054	
05242021a_025.d	7.212	28851	GC-CA*20	Sample	P1-A6	10.0	0.100	10.0	0.1000	51.93	0.1279	0.0128		
05242021a_026.d	7.212	28851	GC-CA*20	Sample	P1-A6	10.0	0.100	10.0	0.1000	51.28	0.1265	0.0126	0.013	
05242021a_027.d	7.212	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				40.28				
05242021a_028.d	7.212	28852	GD-CA*20	Sample	P1-A7	10.0	0.100	10.0	0.1000	80.91	0.1935	0.0194		
05242021a_029.d	7.212	28852	GD-CA*20	Sample	P1-A7	10.0	0.100	10.0	0.1000	87.69	0.2088	0.0209	0.020	
05242021a_030.d	-----	28227 (Flonicamid)	GC-CA18	Sample	P1-A8	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_031.d	-----	28227 (Flonicamid)	GC-CA18	Sample	P1-A8	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_032.d	7.212	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				102.10				
05242021a_033.d	-----	28228 (Flonicamid)	GD-CA18	Sample	P1-A9	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_034.d	-----	28228 (Flonicamid)	GD-CA18	Sample	P1-A9	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_035.d	-----	28851 (Flonicamid)	GC-CA*20	Sample	P1-B1	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_036.d	-----	28851 (Flonicamid)	GC-CA*20	Sample	P1-B1	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_037.d	7.212	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				39.38				
05242021a_038.d	-----	28852 (Flonicamid)	GD-CA*20	Sample	P1-B2	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_039.d	-----	28852 (Flonicamid)	GD-CA*20	Sample	P1-B2	10.0	0.100	200.0	0.0050		<0.10	<0.01		
05242021a_040.d	7.225	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				102.44				

Calculated LOD at 0.045 pg/µL = 15

Calculated LOQ at 0.10 pg/µL = 40

"-----" denotes response below area threshold

Strikethrough denotes data not used, dilutions used for flonicamid quantitation only



Calibration STD

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_012.d	Calibration	Level	1	Enabled	☒	Response	443.86	Exp Conc	1.0000
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D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_032.d	Calibration	Level	3	Enabled	☒	Response	102.10	Exp Conc	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_040.d	Calibration	Level	3	Enabled	☒	Response	102.44	Exp Conc	0.2500
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D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_037.d	Calibration	Level	4	Enabled	☒	Response	39.38	Exp Conc	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05242021\05242021a_008.d	Calibration	Level	5	Enabled	☒	Response	26.18	Exp Conc	0.0500

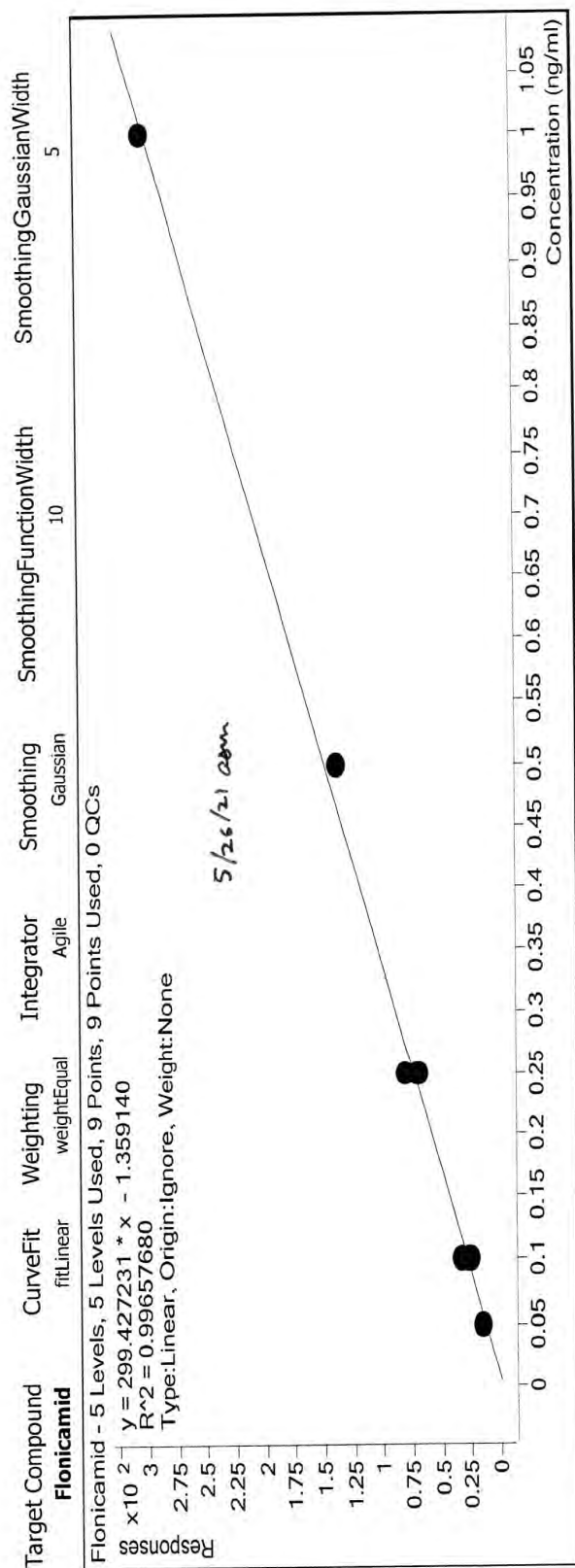
Compound Injection Date
Flonicamid 5/25/2021

08550 - Flonicamid/Onion - Field Samples WA*403, WA404 - Extracted 5/25/2021 by ASM

5/26/21 asm

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp (pg/µL)	Conc (pg/µL)	ppm	Ave ppm	% Rec
05252021a_001.d	7.735	Condition		Sample	P1-A3	10.0								
05252021a_002.d	7.721	Condition		Sample	P1-A3	10.0								
05252021a_003.d	-----	Condition		Sample	P1-A3	10.0								
05252021a_004.d	7.721	Condition		Sample	P1-A3	10.0								
05252021a_005.d	7.721	Condition		Sample	P1-A3	10.0								
05252021a_006.d	7.721	Condition		Sample	P1-A3	10.0								
05252021a_007.d	7.721	Condition		Sample	P1-A3	10.0								
05252021a_008.d	7.721	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				16.82				
05252021a_009.d	7.735	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				33.13				
05252021a_010.d	7.735	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				71.34				
05252021a_011.d	7.721	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				138.24				
05252021a_012.d	7.721	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				302.63				
05252021a_013.d	-----	28861	DBA-WA*403	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05252021a_014.d	-----	28861	DBA-WA*403	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05252021a_015.d	-----	28654	DBA-WA404	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05252021a_016.d	-----	28654	DBA-WA404	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05252021a_017.d	7.721	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				28.40				
05252021a_018.d	7.721	28861C0.01R3	DBA-WA*403	Sample	P1-A3	10.0	0.100	10.0	0.1000	27.45	0.0962	0.0096		
05252021a_019.d	7.721	28861C0.01R3	DBA-WA*403	Sample	P1-A3	10.0	0.100	10.0	0.1000	25.92	0.0911	0.0091	0.00937	94%
05252021a_020.d	7.721	28861C1.0R3	DBA-WA*403	Sample	P1-A4	10.0	0.100	400.0	0.0025	78.83	0.2678	1.0712		
05252021a_021.d	7.735	28861C1.0R3	DBA-WA*403	Sample	P1-A4	10.0	0.100	400.0	0.0025	76.37	0.2596	1.0384	1.05	105%
05252021a_022.d	7.721	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				79.98				
05252021a_023.d	-----	28863	DBC-WA*403	Sample	P1-A5	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05252021a_024.d	-----	28863	DBC-WA*403	Sample	P1-A5	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05252021a_025.d	7.721	28864	DBC-WA*403	Sample	P1-A6	10.0	0.100	10.0	0.1000	27.98	<0.10	<0.01	<0.01	
05252021a_026.d	7.721	28864	DBC-WA*403	Sample	P1-A6	10.0	0.100	10.0	0.1000	26.71	<0.10	<0.01	<0.01	
05252021a_027.d	7.735	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				25.92				
05252021a_028.d	7.735	28656	DBC-WA404	Sample	P1-A7	10.0	0.100	10.0	0.1000	31.34	0.1092	0.0109		
05252021a_029.d	7.721	28656	DBC-WA404	Sample	P1-A7	10.0	0.100	10.0	0.1000	31.19	0.1087	0.0109	0.011	
05252021a_030.d	7.721	28657	DBC-WA404	Sample	P1-A8	10.0	0.100	10.0	0.1000	23.41	<0.10	<0.01	<0.01	
05252021a_031.d	-----	28657	DBC-WA404	Sample	P1-A8	10.0	0.100	10.0	0.1000		<0.10	<0.01	<0.01	
05252021a_032.d	7.735	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				69.82				
Calculated LOD at 0.045 pg/µL = 12														
Calculated LOQ at 0.10 pg/µL = 29														
"-----" denotes response below area threshold														

Printed at: 9:24 AM on: 5/26/2021



Calibration STD

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_011.d	Calibration	1	☒	302.63	1.0000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_010.d	Calibration	2	☒	138.24	0.5000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_009.d	Calibration	3	☒	71.34	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_008.d	Calibration	3	☒	79.98	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_007.d	Calibration	3	☒	69.82	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_006.d	Calibration	4	☒	33.13	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_005.d	Calibration	4	☒	28.40	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_004.d	Calibration	4	☒	25.92	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_003.d	Calibration	5	☒	16.82	0.0500

Compound
TFNA-AM

Injection Date

5/25/2021

08550 - Fonicamid/Onion - Field Samples WA*403, WA404 - Extracted 5/25/2021 by ASM

5/26/21 ASM

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp (pg/µL)	Conc (pg/µL)	ppm	Ave ppm	% Rec
05252021a_001.d	5.352	Condition		Sample	P1-A3	10.0								
05252021a_002.d	5.311	Condition		Sample	P1-A3	10.0								
05252021a_003.d	5.311	Condition		Sample	P1-A3	10.0								
05252021a_004.d	5.311	Condition		Sample	P1-A3	10.0								
05252021a_005.d	5.311	Condition		Sample	P1-A3	10.0								
05252021a_006.d	5.324	Condition		Sample	P1-A3	10.0								
05252021a_007.d	5.311	Condition		Sample	P1-A3	10.0								
05252021a_008.d	5.324	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				65.19				
05252021a_009.d	5.324	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				107.88				
05252021a_010.d	5.311	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				285.65				
05252021a_011.d	5.311	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				600.17				
05252021a_012.d	5.297	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				1203.58				
05252021a_013.d	-----	28861	DBA-WA*403	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05252021a_014.d	-----	28861	DBA-WA*403	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_015.d	-----	28654	DBA-WA404	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_016.d	-----	28654	DBA-WA404	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_017.d	5.324	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				116.19				
05252021a_018.d	5.311	28861C0.01R3	DBA-WA*403	Sample	P1-A3	10.0	0.100	10.0	0.1000	106.47	0.0915	0.0091		
05252021a_019.d	5.311	28861C0.01R3	DBA-WA*403	Sample	P1-A3	10.0	0.100	10.0	0.1000	106.74	0.0917	0.0092	0.00916	92%
05252021a_020.d	5.324	28861C1.0R3	DBA-WA*403	Sample	P1-A4	10.0	0.100	400.0	0.0025	314.76	0.2639	1.0555		
05252021a_021.d	5.324	28861C1.0R3	DBA-WA*403	Sample	P1-A4	10.0	0.100	400.0	0.0025	309.67	0.2597	1.0387	1.05	105%
05252021a_022.d	5.311	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				298.80				
05252021a_023.d	-----	28863	DBC-WA*403	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05252021a_024.d	-----	28863	DBC-WA*403	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_025.d	-----	28864	DBD-WA*403	Sample	P1-A6	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_026.d	-----	28864	DBD-WA*403	Sample	P1-A6	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_027.d	5.324	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				112.62				
05252021a_028.d	-----	28656	DBC-WA404	Sample	P1-A7	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05252021a_029.d	-----	28656	DBC-WA404	Sample	P1-A7	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_030.d	-----	28657	DBD-WA404	Sample	P1-A8	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_031.d	-----	28657	DBD-WA404	Sample	P1-A8	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_032.d	5.311	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				314.58				
Calculated LOD at 0.045 pg/µL =	50													
Calculated LOQ at 0.10 pg/µL =	117													
"-----" denotes response below area threshold														

Batch Data Path

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Calibration Info

Target Compound

TFNA-AM

CurveFit

fit.Linear

Weighting

weightEqual

Integrator

Agile

Smoothing

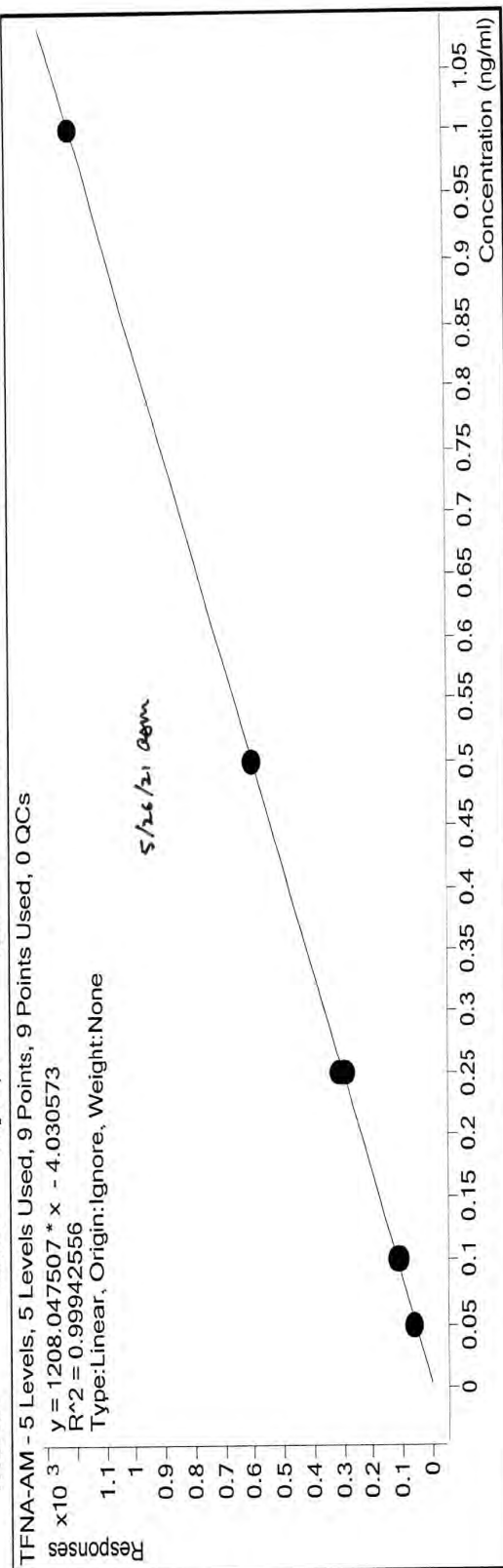
Gaussian

SmoothingFunctionWidth

10

SmoothingGaussianWidth

5



Calibration STD

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D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_010.d

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Cal Type	Level	Enabled	Response	Exp Conc
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Calibration	2	☒	600.17	0.5000
Calibration	3	☒	285.65	0.2500
Calibration	3	☒	298.80	0.2500
Calibration	3	☒	314.58	0.2500
Calibration	4	☒	107.88	0.1000
Calibration	4	☒	116.19	0.1000
Calibration	4	☒	112.62	0.1000
Calibration	5	☒	65.19	0.0500

Compound Injection Date
TFNA 5/25/2021

08550 - Fonicamid/Onion - Field Samples WA*403, WA404 - Extracted 5/25/2021 by ASM

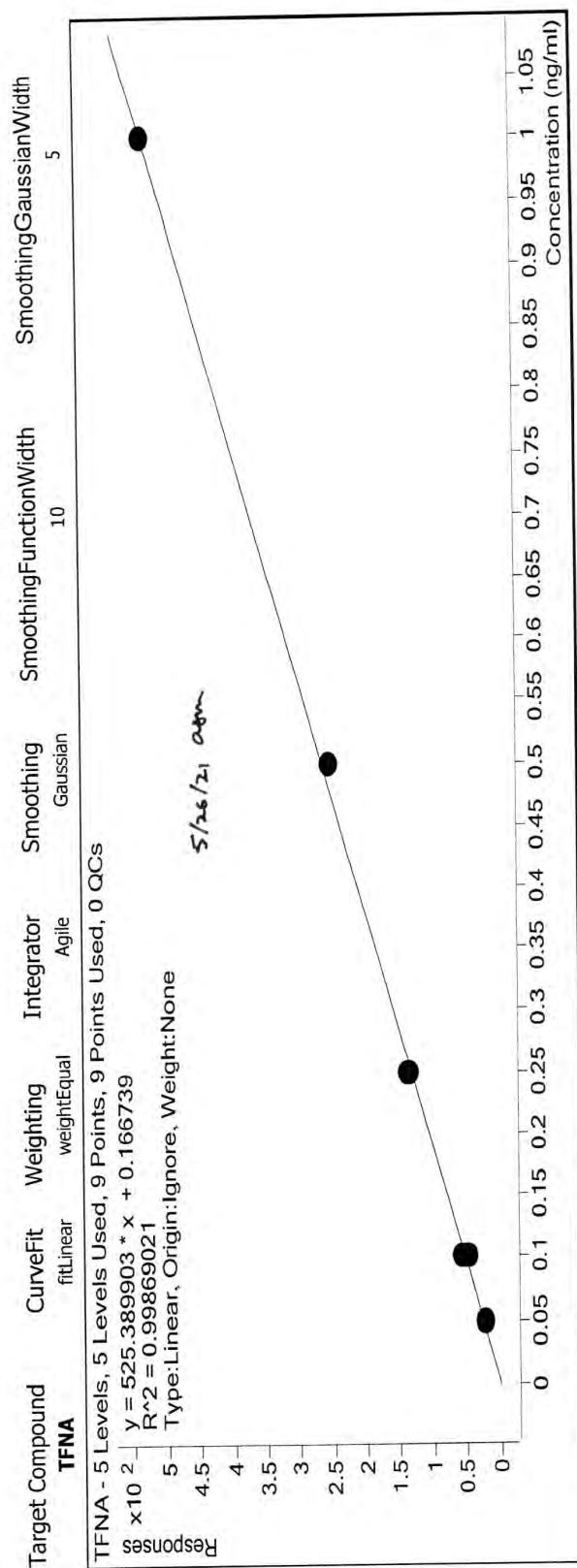
5/26/21 abm

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05252021a_001.d	6.010	Condition		Sample	P1-A3	10.0								
05252021a_002.d	5.983	Condition		Sample	P1-A3	10.0								
05252021a_003.d	5.983	Condition		Sample	P1-A3	10.0								
05252021a_004.d	5.983	Condition		Sample	P1-A3	10.0								
05252021a_005.d	5.983	Condition		Sample	P1-A3	10.0								
05252021a_006.d	5.983	Condition		Sample	P1-A3	10.0								
05252021a_007.d	5.983	Condition		Sample	P1-A3	10.0								
05252021a_008.d	5.969	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				24.89				
05252021a_009.d	5.969	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				46.80				
05252021a_010.d	5.969	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				135.63				
05252021a_011.d	5.956	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				251.30				
05252021a_012.d	5.942	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				528.53				
05252021a_013.d	-----	28861	DBA-WA*403	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05252021a_014.d	-----	28861	DBA-WA*403	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_015.d	-----	28654	DBA-WA404	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_016.d	-----	28654	DBA-WA404	Sample	P1-A2	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_017.d	5.969	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				56.66				
05252021a_018.d	5.983	28861C0.01R3	DBA-WA*403	Sample	P1-A3	10.0	0.100	10.0	0.1000	44.93	0.0852	0.0085		86%
05252021a_019.d	5.997	28861C0.01R3	DBA-WA*403	Sample	P1-A3	10.0	0.100	10.0	0.1000	45.74	0.0867	0.0087	0.00860	
05252021a_020.d	5.983	28861C1.0R3	DBA-WA*403	Sample	P1-A4	10.0	0.100	400.0	0.0025	136.01	0.2586	1.0342		103%
05252021a_021.d	5.969	28861C1.0R3	DBA-WA*403	Sample	P1-A4	10.0	0.100	400.0	0.0025	134.77	0.2562	1.0248	1.03	
05252021a_022.d	5.969	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				133.17				
05252021a_023.d	-----	28863	DBC-WA*403	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
05252021a_024.d	-----	28863	DBC-WA*403	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_025.d	-----	28864	DBD-WA*403	Sample	P1-A6	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_026.d	-----	28864	DBD-WA*403	Sample	P1-A6	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
05252021a_027.d	5.969	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				52.30				
05252021a_028.d	5.997	28656	DBC-WA404	Sample	P1-A7	10.0	0.100	10.0	0.1000	43.01	<0.10	<0.01	<0.01	
05252021a_029.d	5.997	28656	DBC-WA404	Sample	P1-A7	10.0	0.100	10.0	0.1000	41.05	<0.10	<0.01	<0.01	
05252021a_030.d	5.997	28657	DBD-WA404	Sample	P1-A8	10.0	0.100	10.0	0.1000	41.84	<0.10	<0.01	<0.01	
05252021a_031.d	5.983	28657	DBD-WA404	Sample	P1-A8	10.0	0.100	10.0	0.1000	41.91	<0.10	<0.01	<0.01	
05252021a_032.d	5.969	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				138.22				

Calculated LOD at 0.045 pg/µL = 24

Calculated LOQ at 0.10 pg/µL = 53

"-----" denotes response below area threshold



Calibration STD

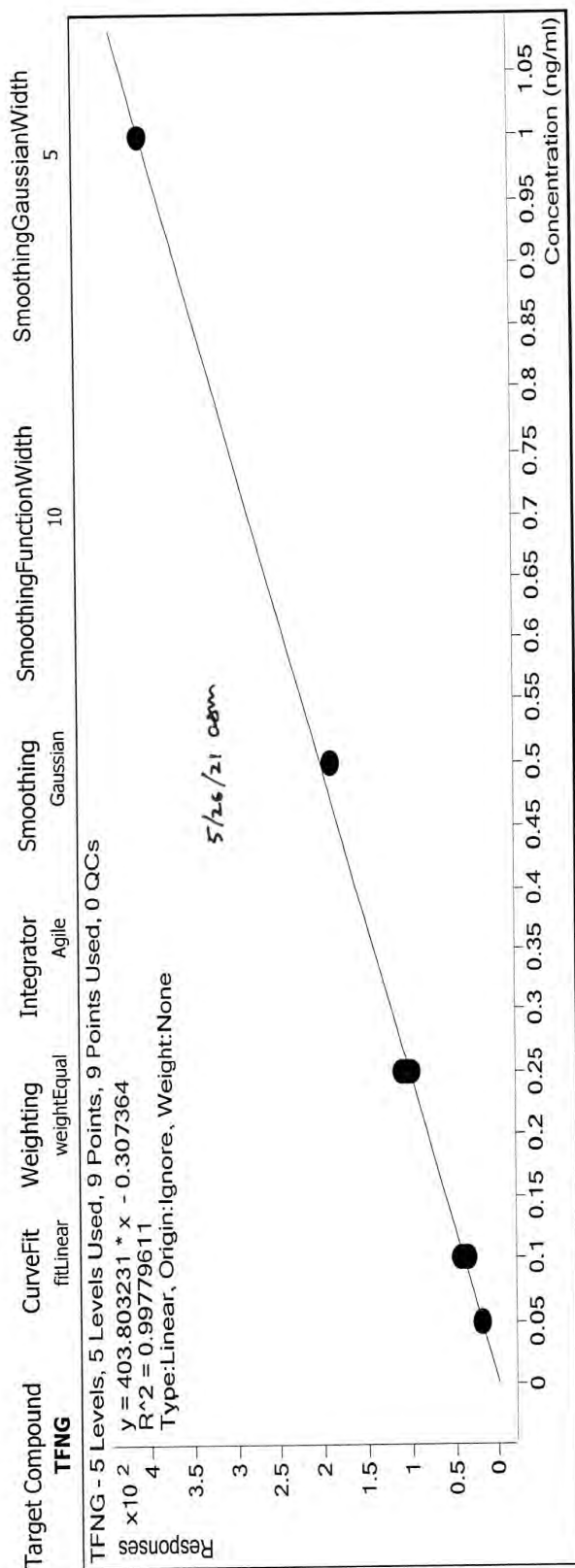
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Compound Injection Date
TFNG 5/25/2021

08550 - Fonicamid/Onion - Field Samples WA*403, WA404 - Extracted 5/25/2021 by ASM

5/26/21 ASM

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
05252021a_001.d	7.225	Condition		Sample	P1-A3	10.0								
05252021a_002.d	7.198	Condition		Sample	P1-A3	10.0								
05252021a_003.d	7.198	Condition		Sample	P1-A3	10.0								
05252021a_004.d	7.212	Condition		Sample	P1-A3	10.0								
05252021a_005.d	7.212	Condition		Sample	P1-A3	10.0								
05252021a_006.d	7.212	Condition		Sample	P1-A3	10.0								
05252021a_007.d	7.212	Condition		Sample	P1-A3	10.0								
05252021a_008.d	7.198	Condition		Sample	P1-A3	10.0								
05252021a_009.d	7.198	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				20.75				
05252021a_010.d	7.212	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				35.57				
05252021a_011.d	7.212	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				105.59				
05252021a_012.d	7.198	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				190.28				
05252021a_013.d	7.198	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				406.58				
05252021a_014.d	-----	28861	DBA-WA*403	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05252021a_015.d	-----	28861	DBA-WA*403	Sample	P1-A1	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05252021a_016.d	-----	28854	DBA-WA404	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05252021a_017.d	-----	28854	DBA-WA404	Sample	P1-A2	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05252021a_018.d	7.198	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				37.74				
05252021a_019.d	7.212	28861CO.01R3	DBA-WA*403	Sample	P1-A3	10.0	0.100	10.0	0.1000	41.71	0.1040	0.0104		
05252021a_020.d	7.212	28861CO.01R3	DBA-WA*403	Sample	P1-A3	10.0	0.100	10.0	0.1000	37.72	0.0942	0.0094		99%
05252021a_021.d	7.212	28861C1.0R3	DBA-WA*403	Sample	P1-A4	10.0	0.100	400.0	0.0025	123.47	0.3065	1.2262		
05252021a_022.d	7.212	28861C1.0R3	DBA-WA*403	Sample	P1-A4	10.0	0.100	400.0	0.0025	114.50	0.2843	1.1373		118%
05252021a_023.d	-----	28863	DBC-WA*403	Calibration	Vial 3	10.0				99.64				
05252021a_024.d	-----	28863	DBC-WA*403	Sample	P1-A5	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05252021a_025.d	-----	28864	DBC-WA*403	Sample	P1-A5	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05252021a_026.d	-----	28864	DBC-WA*403	Sample	P1-A6	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05252021a_027.d	-----	28864	DBC-WA*403	Sample	P1-A6	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05252021a_028.d	7.212	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				42.32				
05252021a_029.d	7.212	28656	DBC-WA404	Sample	P1-A7	10.0	0.100	10.0	0.1000	27.86	<0.10	<0.01		
05252021a_030.d	-----	28656	DBC-WA404	Sample	P1-A7	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05252021a_031.d	-----	28657	DBC-WA404	Sample	P1-A8	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05252021a_032.d	-----	28657	DBC-WA404	Sample	P1-A8	10.0	0.100	10.0	0.1000		<0.10	<0.01		
05252021a_033.d	7.212	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				108.67				
Calculated LOD at 0.045 pg/µL = 18														
Calculated LOQ at 0.10 pg/µL = 40														
"-----" denotes response below area threshold														



Calibration STD

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_012.d
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05252021\05252021a_011.d
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Compound Injection Date
Flonicamid 5/26/2021

08550 - Flonicamid/Onion - Method Validation Extension (2.0 ppm) - Green Onion - Extracted 5/26/2021 by ASM

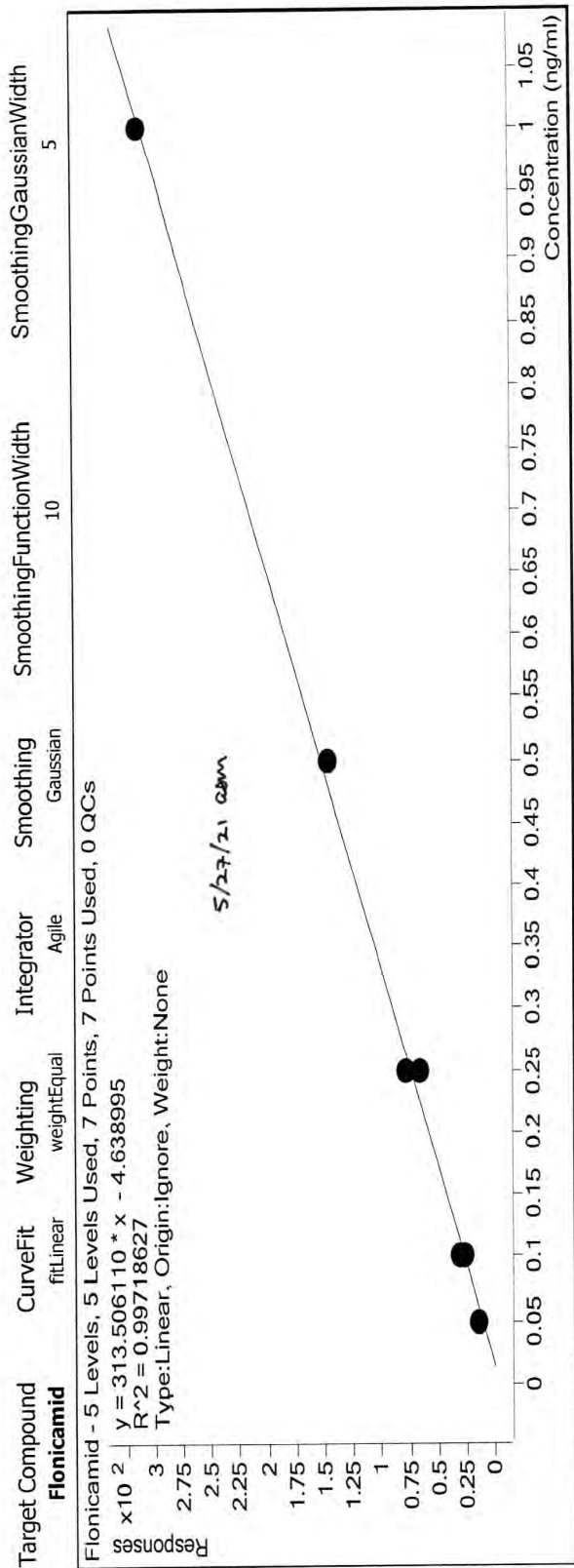
5/27/21 ASM

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec	Ave % Rec	Std Dev
05262021a_001.d	-----	Condition		Sample	P1-A3	10.0										
05262021a_002.d	7.721	Condition		Sample	P1-A3	10.0										
05262021a_003.d	7.721	Condition		Sample	P1-A3	10.0										
05262021a_004.d	7.721	Condition		Sample	P1-A3	10.0										
05262021a_005.d	7.721	Condition		Sample	P1-A3	10.0										
05262021a_006.d	7.721	Condition		Sample	P1-A3	10.0										
05262021a_007.d	7.721	Condition		Sample	P1-A3	10.0										
05262021a_008.d	7.735	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				15.52						
05262021a_009.d	7.721	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				29.95						
05262021a_010.d	7.735	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				78.19						
05262021a_011.d	7.721	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				144.77						
05262021a_012.d	7.721	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				312.98						
05262021a_013.d	-----	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05262021a_014.d	-----	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01			
05262021a_015.d	7.735	28849V2.0R1	GA-CA*20	Sample	P1-A2	10.0	0.100	1000.0	0.0010	69.49	0.2364	2.3644				
05262021a_016.d	7.735	28849V2.0R1	GA-CA*20	Sample	P1-A2	10.0	0.100	1000.0	0.0010	66.74	0.2277	2.2769	2.32	116%		
05262021a_017.d	7.735	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				25.83						
05262021a_018.d	7.721	28849V2.0R2	GA-CA*20	Sample	P1-A3	10.0	0.100	1000.0	0.0010	62.92	0.2155	2.1548				
05262021a_019.d	7.735	28849V2.0R2	GA-CA*20	Sample	P1-A3	10.0	0.100	1000.0	0.0010	67.44	0.2259	2.2991	2.23	111%		
05262021a_020.d	7.721	28849V2.0R3	GA-CA*20	Sample	P1-A4	10.0	0.100	1000.0	0.0010	57.73	0.1989	1.9893				
05262021a_021.d	7.721	28849V2.0R3	GA-CA*20	Sample	P1-A4	10.0	0.100	1000.0	0.0010	61.79	0.2119	2.1189	2.05	103%	110%	7%
05262021a_022.d	7.735	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				65.68						

Calculated LOD at 0.045 pg/µL = 9

Calculated LOQ at 0.10 pg/µL = 27

"-----" denotes response below area threshold



Calibration STD

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05262021\05262021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
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D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05262021\05262021a_010.d	Calibration	2	☒	144.77	0.5000
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D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05262021\05262021a_007.d	Calibration	4	☒	29.95	0.1000
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D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05262021\05262021a_008.d	Calibration	5	☒	15.52	0.0500

Compound
TFNA-AM

Injection Date
5/26/2021

08550 - Flonicamid/Onion - Method Validation Extension (2.0 ppm) - Green Onion - Extracted 5/26/2021 by ASM

5/27/21 2pm

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec	Ave % Rec	Std Dev
05262021a_001.d	-----	Condition		Sample	P1-A3	10.0										
05262021a_002.d	5.311	Condition		Sample	P1-A3	10.0										
05262021a_003.d	5.311	Condition		Sample	P1-A3	10.0										
05262021a_004.d	5.324	Condition		Sample	P1-A3	10.0										
05262021a_005.d	5.311	Condition		Sample	P1-A3	10.0										
05262021a_006.d	5.311	Condition		Sample	P1-A3	10.0										
05262021a_007.d	5.311	Condition		Sample	P1-A3	10.0										
05262021a_008.d	5.324	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				69.28						
05262021a_009.d	5.324	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				126.88						
05262021a_010.d	5.311	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				320.43						
05262021a_011.d	5.311	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				663.01						
05262021a_012.d	5.297	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				1305.08						
05262021a_013.d	-----	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05262021a_014.d	-----	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05262021a_015.d	5.324	28849V2.0R1	GA-CA*20	Sample	P1-A2	10.0	0.100	1000.0	0.0010	279.07	0.2188	2.1881				
05262021a_016.d	5.324	28849V2.0R1	GA-CA*20	Sample	P1-A2	10.0	0.100	1000.0	0.0010	274.17	0.2151	2.1509	2.17	108%		
05262021a_017.d	5.324	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				128.00						
05262021a_018.d	5.324	28849V2.0R2	GA-CA*20	Sample	P1-A3	10.0	0.100	1000.0	0.0010	246.47	0.1940	1.9399				
05262021a_019.d	5.324	28849V2.0R2	GA-CA*20	Sample	P1-A3	10.0	0.100	1000.0	0.0010	257.00	0.2020	2.0201	1.98	99%		
05262021a_020.d	5.324	28849V2.0R3	GA-CA*20	Sample	P1-A4	10.0	0.100	1000.0	0.0010	276.50	0.2169	2.1686				
05262021a_021.d	5.324	28849V2.0R3	GA-CA*20	Sample	P1-A4	10.0	0.100	1000.0	0.0010	274.93	0.2157	2.1567	2.16	108%	105%	5%
05262021a_022.d	5.311	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				284.16						

Calculated LOD at 0.045 pg/µL = 51

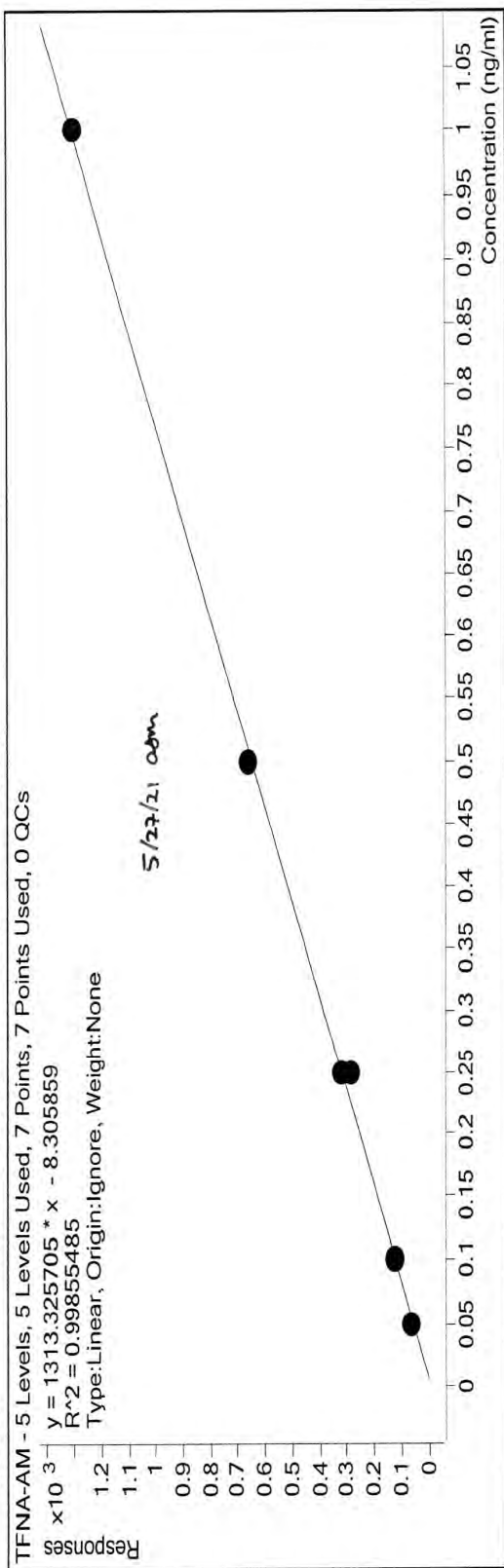
Calculated LOQ at 0.10 pg/µL = 123

"-----" denotes response below area threshold

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\05262021\QuantResults\05262021a.batch.bin

Batch Data Path**Calibration Info**

Target Compound **TFNA-AM** CurveFit **fitLinear** Weighting **weightEqual** Integrator **Agile** Smoothing **Gaussian** SmoothingFunctionWidth **10** SmoothingGaussianWidth **5**

**Calibration STD**

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Cal Type	Level	Enabled	Response	Exp Conc
Calibration	1	☒	1305.08	1.0000
Calibration	2	☒	663.01	0.5000
Calibration	3	☒	320.43	0.2500
Calibration	3	☒	284.16	0.2500
Calibration	4	☒	126.88	0.1000
Calibration	4	☒	128.00	0.1000
Calibration	5	☒	69.28	0.0500

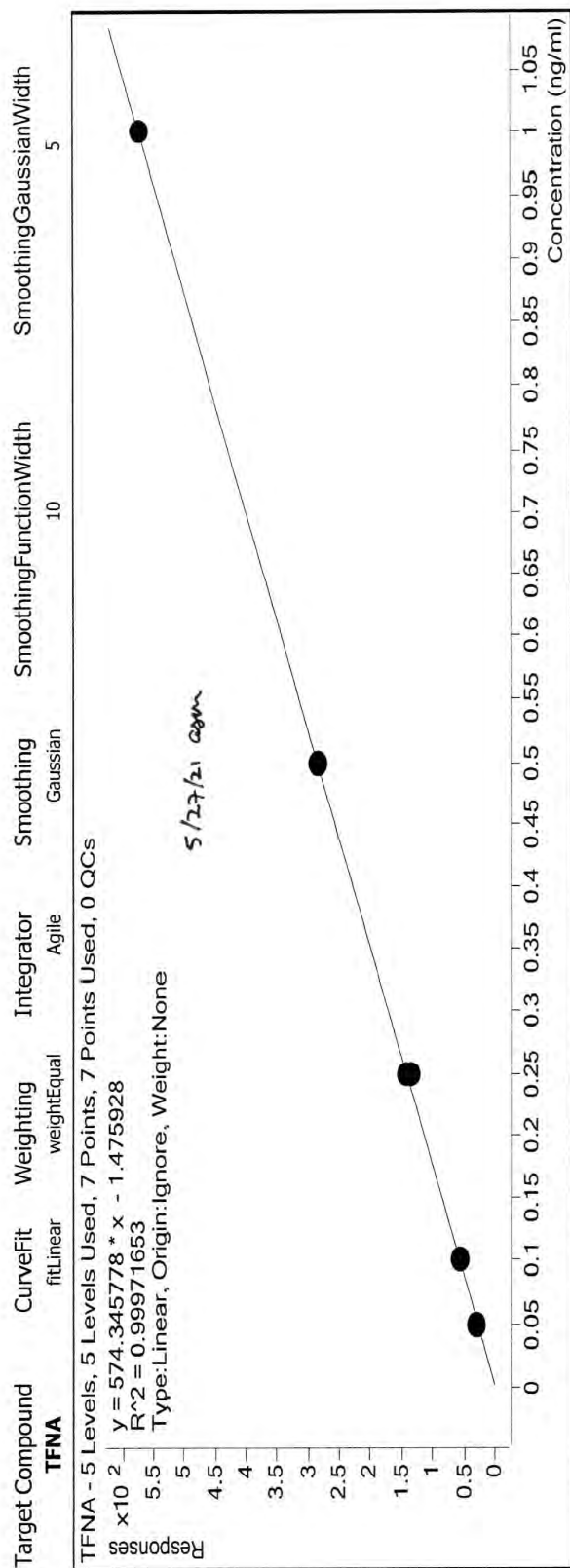
Compound Injection Date
TFNA 5/26/2021

08550 - Flonicamid/Onion - Method Validation Extension (2.0 ppm) - Green Onion - Extracted 5/26/2021 by ASM

5/27/21 *asm*

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec	Ave % Rec	Std Dev
05262021a_001.d	-----	Condition		Sample	P1-A3	10.0										
05262021a_002.d	5.969	Condition		Sample	P1-A3	10.0										
05262021a_003.d	5.969	Condition		Sample	P1-A3	10.0										
05262021a_004.d	5.969	Condition		Sample	P1-A3	10.0										
05262021a_005.d	5.969	Condition		Sample	P1-A3	10.0										
05262021a_006.d	5.969	Condition		Sample	P1-A3	10.0										
05262021a_007.d	5.969	Condition		Sample	P1-A3	10.0										
05262021a_008.d	5.969	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				31.28						
05262021a_009.d	5.969	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				55.29						
05262021a_010.d	5.969	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				143.56						
05262021a_011.d	5.969	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				284.44						
05262021a_012.d	5.956	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				574.45						
05262021a_013.d	-----	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05262021a_014.d	-----	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05262021a_015.d	5.969	28849V2.0R1	GA-CA*20	Sample	P1-A2	10.0	0.100	1000.0	0.0010	125.93	0.2218	2.2183				
05262021a_016.d	5.983	28849V2.0R1	GA-CA*20	Sample	P1-A2	10.0	0.100	1000.0	0.0010	116.20	0.2049	2.0488	2.13	107%		
05262021a_017.d	5.969	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				57.05						
05262021a_018.d	5.983	28849V2.0R2	GA-CA*20	Sample	P1-A3	10.0	0.100	1000.0	0.0010	114.78	0.2024	2.0241				
05262021a_019.d	5.983	28849V2.0R2	GA-CA*20	Sample	P1-A3	10.0	0.100	1000.0	0.0010	117.93	0.2079	2.0791	2.05	103%		
05262021a_020.d	5.969	28849V2.0R3	GA-CA*20	Sample	P1-A4	10.0	0.100	1000.0	0.0010	120.16	0.2118	2.1179				
05262021a_021.d	5.969	28849V2.0R3	GA-CA*20	Sample	P1-A4	10.0	0.100	1000.0	0.0010	121.17	0.2135	2.1353	2.13	106%	105%	2%
05262021a_022.d	5.969	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				135.87						
Calculated LOD at 0.045 pg/µL = 24																
Calculated LOQ at 0.10 pg/µL = 56																

"-----" denotes response below area threshold

**Calibration STD**

D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05262021\05262021a_012.d
 D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05262021\05262021a_011.d
 D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05262021\05262021a_010.d
 D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05262021\05262021a_022.d
 D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05262021\05262021a_009.d
 D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05262021\05262021a_017.d
 D:\MassHunter\Flonicamid\08550_Orion\Data\Results\05262021\05262021a_008.d

Cal Type	Level	Enabled	Response	Exp Conc
Calibration	1	☒	574.45	1.0000
Calibration	2	☒	284.44	0.5000
Calibration	3	☒	143.56	0.2500
Calibration	3	☒	135.87	0.2500
Calibration	4	☒	55.29	0.1000
Calibration	4	☒	57.05	0.1000
Calibration	5	☒	31.28	0.0500

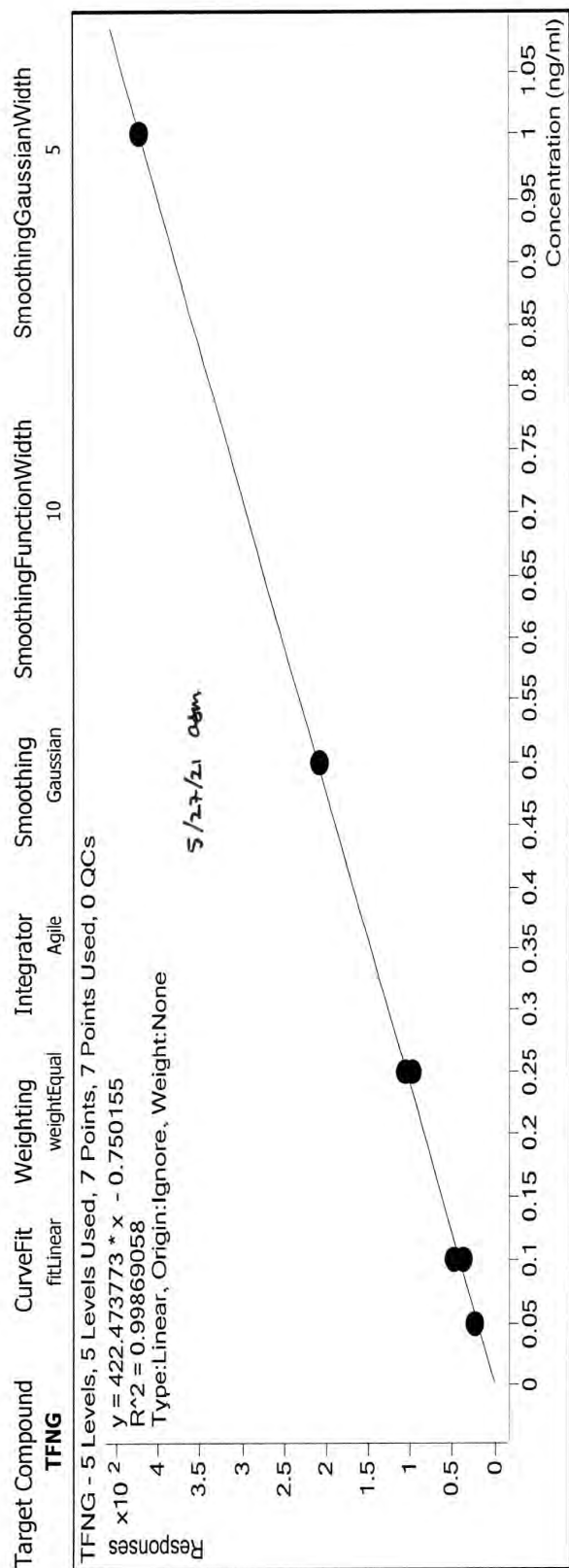
Compound Injection Date
TFNG 5/26/2021

08550 - Flonicamid/Onion - Method Validation Extension (2.0 ppm) - Green Onion - Extracted 5/26/2021 by ASM

5/27/21 *asm*

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp (pg/µL)	Conc (pg/µL)	ppm	Ave ppm	% Rec	Ave %	Std Dev
05262021a_001.d	-----	Condition		Sample	P1-A3	10.0										
05262021a_002.d	7.198	Condition		Sample	P1-A3	10.0										
05262021a_003.d	7.198	Condition		Sample	P1-A3	10.0										
05262021a_004.d	7.212	Condition		Sample	P1-A3	10.0										
05262021a_005.d	7.198	Condition		Sample	P1-A3	10.0										
05262021a_006.d	7.198	Condition		Sample	P1-A3	10.0										
05262021a_007.d	7.198	Condition		Sample	P1-A3	10.0										
05262021a_008.d	7.212	687-2M16 0.05 pg/µL		Calibration	Vial 5	10.0				24.80						
05262021a_009.d	7.212	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				48.00						
05262021a_010.d	7.198	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				106.69						
05262021a_011.d	7.198	687-2M13 0.50 pg/µL		Calibration	Vial 2	10.0				207.99						
05262021a_012.d	7.198	687-2M12 1.0 pg/µL		Calibration	Vial 1	10.0				424.01						
05262021a_013.d	-----	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01				
05262021a_014.d	-----	28849	GA-CA*20	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01			
05262021a_015.d	7.212	28849V2.0R1	GA-CA*20	Sample	P1-A2	10.0	0.100	1000.0	0.0010	98.82	0.2357	2.3568				
05262021a_016.d	7.212	28849V2.0R1	GA-CA*20	Sample	P1-A2	10.0	0.100	1000.0	0.0010	97.94	0.2336	2.3361	2.35	117%		
05262021a_017.d	7.212	687-2M15 0.10 pg/µL		Calibration	Vial 4	10.0				36.37						
05262021a_018.d	7.212	28849V2.0R2	GA-CA*20	Sample	P1-A3	10.0	0.100	1000.0	0.0010	95.58	0.2280	2.2802				
05262021a_019.d	7.212	28849V2.0R2	GA-CA*20	Sample	P1-A3	10.0	0.100	1000.0	0.0010	95.04	0.2267	2.2673	2.27	114%		
05262021a_020.d	7.198	28849V2.0R3	GA-CA*20	Sample	P1-A4	10.0	0.100	1000.0	0.0010	97.49	0.2325	2.3254				
05262021a_021.d	7.198	28849V2.0R3	GA-CA*20	Sample	P1-A4	10.0	0.100	1000.0	0.0010	101.12	0.2411	2.4114	2.37	118%	116%	2%
05262021a_022.d	7.198	687-2M14 0.25 pg/µL		Calibration	Vial 3	10.0				97.46						
Calculated LOD at 0.045 pg/µL = 18																
Calculated LOQ at 0.10 pg/µL = 41																
"-----" denotes response below area threshold																

Printed at: 8:49 AM on: 5/27/2021

**Calibration STD**

D:\MassHunter\Fliconicamid\08550_Onion\Data\Results\05262021\05262021a_012.d
 D:\MassHunter\Fliconicamid\08550_Onion\Data\Results\05262021\05262021a_011.d
 D:\MassHunter\Fliconicamid\08550_Onion\Data\Results\05262021\05262021a_010.d
 D:\MassHunter\Fliconicamid\08550_Onion\Data\Results\05262021\05262021a_022.d
 D:\MassHunter\Fliconicamid\08550_Onion\Data\Results\05262021\05262021a_009.d
 D:\MassHunter\Fliconicamid\08550_Onion\Data\Results\05262021\05262021a_017.d
 D:\MassHunter\Fliconicamid\08550_Onion\Data\Results\05262021\05262021a_008.d

Cal Type	Level	Enabled	Response	Exp Conc
Calibration	1	☒	424.01	1.0000
Calibration	2	☒	207.99	0.5000
Calibration	3	☒	106.69	0.2500
Calibration	3	☒	97.46	0.2500
Calibration	4	☒	48.00	0.1000
Calibration	4	☒	36.37	0.1000
Calibration	5	☒	24.80	0.0500

Compound Injection Date

Flonicamid 7/20/2021

08550 - Flonicamid/Onion - Field Samples TX380 - Extracted 7/20/2021 by ASM

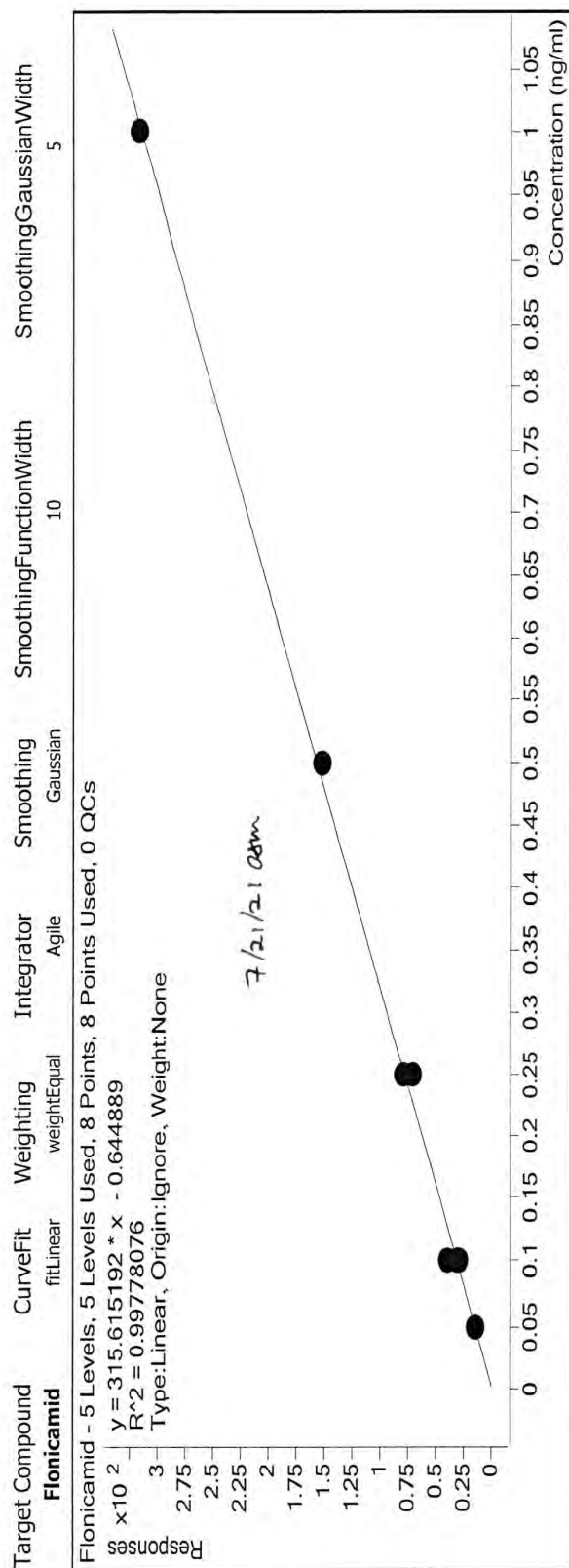
7/21/21 *ASM*

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp (pg/µL)	Conc (pg/µL)	ppm	Ave ppm	% Rec
07202021a_001.d	7.287	Condition		Sample	P1-A3	10.0								
07202021a_002.d	7.694	Condition		Sample	P1-A3	10.0								
07202021a_003.d	7.694	Condition		Sample	P1-A3	10.0								
07202021a_004.d	7.694	Condition		Sample	P1-A3	10.0								
07202021a_005.d	7.694	Condition		Sample	P1-A3	10.0								
07202021a_006.d	7.694	Condition		Sample	P1-A3	10.0								
07202021a_007.d	7.694	Condition		Sample	P1-A3	10.0								
07202021a_008.d	7.694	687-2M21 0.05 pg/µL		Calibration	Vial 5	10.0				15.77				
07202021a_009.d	7.694	687-2M20 0.10 pg/µL		Calibration	Vial 4	10.0				28.79				
07202021a_010.d	7.708	687-2M19 0.25 pg/µL		Calibration	Vial 3	10.0				71.40				
07202021a_011.d	7.694	687-2M18 0.50 pg/µL		Calibration	Vial 2	10.0				152.84				
07202021a_012.d	7.694	687-2M17 1.0 pg/µL		Calibration	Vial 1	10.0				317.95				
07202021a_013.d	-----	29148	DBA-TX380	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
07202021a_014.d	-----	29148	DBA-TX380	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
07202021a_015.d	7.694	29148C0.01R4	DBA-TX380	Sample	P1-A2	10.0	0.100	10.0	0.1000	32.72	0.1057	0.0106		
07202021a_016.d	7.694	29148C0.01R4	DBA-TX380	Sample	P1-A2	10.0	0.100	10.0	0.1000	31.35	0.1014	0.0101	0.0104	104%
07202021a_017.d	7.694	687-2M20 0.10 pg/µL		Calibration	Vial 4	10.0				39.79				
07202021a_018.d	7.694	29148C1.0R4	DBA-TX380	Sample	P1-A3	10.0	0.100	400.0	0.0025	74.91	0.2394	0.9575		
07202021a_019.d	7.694	29148C1.0R4	DBA-TX380	Sample	P1-A3	10.0	0.100	400.0	0.0025	73.87	0.2361	0.9444	0.951	95%
07202021a_020.d	7.694	29150	DBC-TX380	Sample	P1-A4	10.0	0.100	10.0	0.1000	38.19	0.1231	0.0123		
07202021a_021.d	7.694	29150	DBC-TX380	Sample	P1-A4	10.0	0.100	10.0	0.1000	38.87	0.1252	0.0125	0.012	
07202021a_022.d	7.694	687-2M19 0.25 pg/µL		Calibration	Vial 3	10.0				79.00				
07202021a_023.d	7.694	29151	DBD-TX380	Sample	P1-A5	10.0	0.100	10.0	0.1000	56.09	0.1798	0.0180		
07202021a_024.d	7.694	29151	DBD-TX380	Sample	P1-A5	10.0	0.100	10.0	0.1000	62.91	0.2014	0.0201	0.019	
07202021a_025.d	7.694	687-2M20 0.10 pg/µL		Calibration	Vial 4	10.0				31.00				

Calculated LOD at 0.045 pg/µL = 14

Calculated LOQ at 0.10 pg/µL = 31

"-----" denotes response below area threshold



Calibration STD

Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\07202021\07202021a_012.d	1	☒	317.95	1.0000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\07202021\07202021a_011.d	2	☒	152.84	0.5000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\07202021\07202021a_010.d	3	☒	79.00	0.2500
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\07202021\07202021a_022.d	3	☒	79.00	0.2500
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\07202021\07202021a_009.d	4	☒	28.79	0.1000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\07202021\07202021a_017.d	4	☒	39.79	0.1000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\07202021\07202021a_025.d	4	☒	31.00	0.1000
D:\MassHunter\Flonicamid\08550_Orion\Data\Results\07202021\07202021a_008.d	5	☒	15.77	0.0500

Compound Injection Date
TFNA-AM 7/20/2021
 08550 - Flonicamid/Onion - Field Samples TX380 - Extracted 7/20/2021 by ASM

7/21/21 *QSM*

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	Ave ppm	% Rec
07202021a_001.d	-----	Condition		Sample	P1-A3	10.0							
07202021a_002.d	5.270	Condition		Sample	P1-A3	10.0							
07202021a_003.d	5.270	Condition		Sample	P1-A3	10.0							
07202021a_004.d	5.270	Condition		Sample	P1-A3	10.0							
07202021a_005.d	5.270	Condition		Sample	P1-A3	10.0							
07202021a_006.d	5.270	Condition		Sample	P1-A3	10.0							
07202021a_007.d	5.284	Condition		Sample	P1-A3	10.0							
07202021a_008.d	5.270	687-2M21 0.05 pg/µL		Calibration	Vial 5	10.0				55.89			
07202021a_009.d	5.270	687-2M20 0.10 pg/µL		Calibration	Vial 4	10.0				103.23			
07202021a_010.d	5.270	687-2M19 0.25 pg/µL		Calibration	Vial 3	10.0				301.87			
07202021a_011.d	5.256	687-2M18 0.50 pg/µL		Calibration	Vial 2	10.0				585.79			
07202021a_012.d	5.243	687-2M17 1.0 pg/µL		Calibration	Vial 1	10.0				1190.58			
07202021a_013.d	-----	29148	DBA-TX380	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	
07202021a_014.d	-----	29148	DBA-TX380	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	
07202021a_015.d	5.270	29148C0.01R4	DBA-TX380	Sample	P1-A2	10.0	0.100	10.0	0.1000	122.16	0.1033	0.0103	
07202021a_016.d	5.270	29148C0.01R4	DBA-TX380	Sample	P1-A2	10.0	0.100	10.0	0.1000	129.47	0.1095	0.0109	106%
07202021a_017.d	5.270	687-2M20 0.10 pg/µL		Calibration	Vial 4	10.0				123.94			
07202021a_018.d	5.270	29148C1.0R4	DBA-TX380	Sample	P1-A3	10.0	0.100	400.0	0.0025	305.49	0.2575	1.0299	
07202021a_019.d	5.270	29148C1.0R4	DBA-TX380	Sample	P1-A3	10.0	0.100	400.0	0.0025	311.86	0.2628	1.0513	104%
07202021a_020.d	-----	29150	DBC-TX380	Sample	P1-A4	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	
07202021a_021.d	-----	29150	DBC-TX380	Sample	P1-A4	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	
07202021a_022.d	5.270	687-2M19 0.25 pg/µL		Calibration	Vial 3	10.0				300.87			
07202021a_023.d	-----	29151	DBD-TX380	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	
07202021a_024.d	-----	29151	DBD-TX380	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	
07202021a_025.d	5.270	687-2M20 0.10 pg/µL		Calibration	Vial 4	10.0				126.98			

Calculated LOD at 0.045 pg/µL = 53

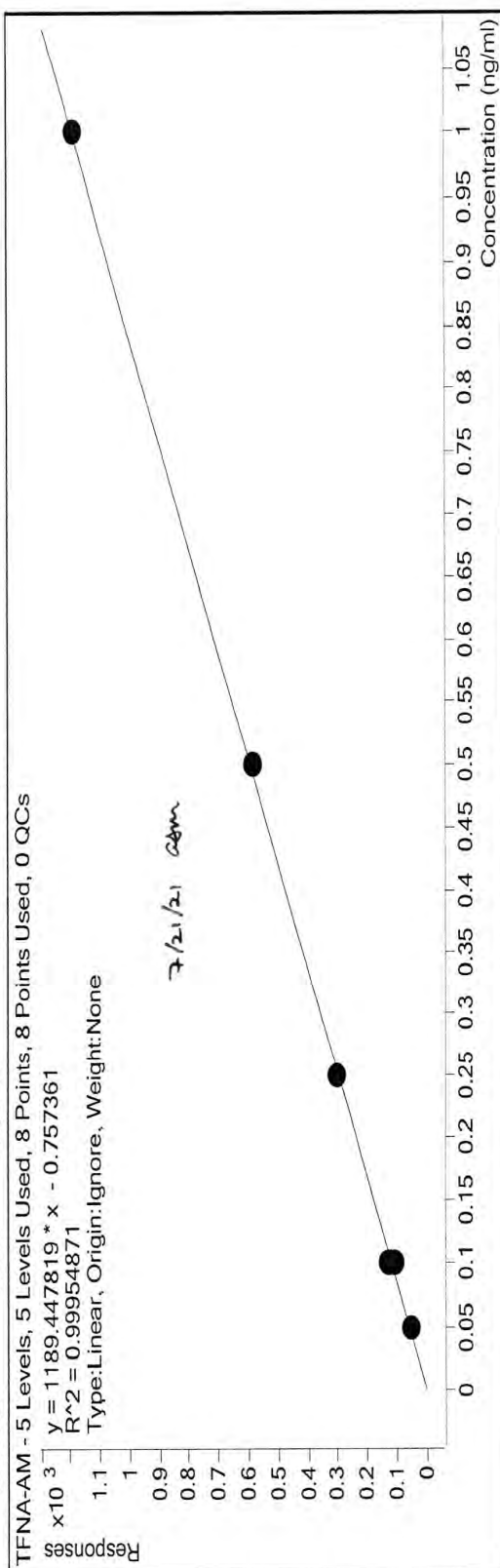
Calculated LOQ at 0.10 pg/µL = 118

"-----" denotes response below area threshold

Batch Data Path D:\MassHunter\Flonicamid\08550_Onion\Data\Results\07202021\QuantResults\07202021a_batch.bin

Calibration Info

Target Compound **TFNA-AM** CurveFit **fitLinear** Weighting **weightEqual** Integrator **Agile** Smoothing **Gaussian** SmoothingFunctionWidth **10** SmoothingGaussianWidth **5**



Calibration STD

D:\MassHunter\Flonicamid\08550_Onion\Data\Results\07202021\07202021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\07202021\07202021a_011.d	Calibration	1	☒	1190.58	1.0000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\07202021\07202021a_010.d	Calibration	2	☒	585.79	0.5000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\07202021\07202021a_009.d	Calibration	3	☒	301.87	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\07202021\07202021a_008.d	Calibration	3	☒	300.87	0.2500
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\07202021\07202021a_017.d	Calibration	4	☒	103.23	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\07202021\07202021a_016.d	Calibration	4	☒	123.94	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\07202021\07202021a_025.d	Calibration	4	☒	126.98	0.1000
D:\MassHunter\Flonicamid\08550_Onion\Data\Results\07202021\07202021a_008.d	Calibration	5	☒	55.89	0.0500

Compound Injection Date
TFNA 7/20/2021

08550 - Flonicamid/Onion - Field Samples TX380 - Extracted 7/20/2021 by ASM

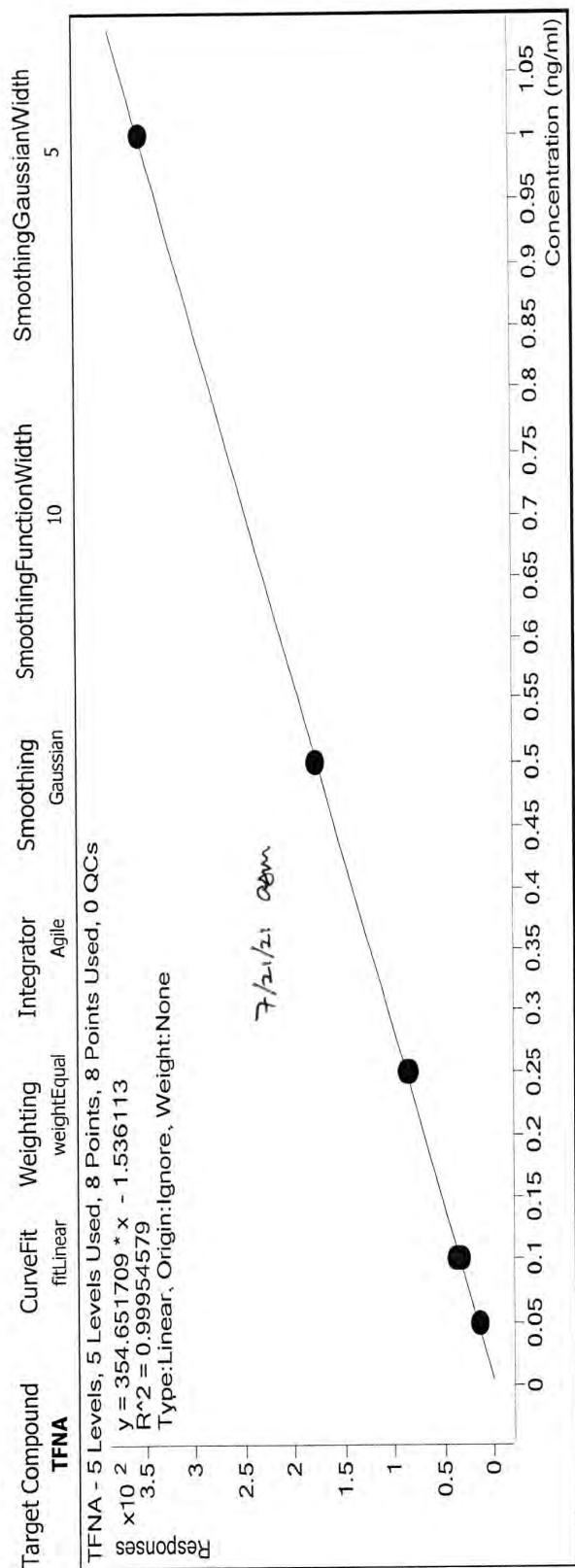
7/21/21 *adm*

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp (pg/µL)	Conc (pg/µL)	ppm	Ave ppm	% Rec
07202021a_001.d	-----	Condition		Sample	P1-A3	10.0								
07202021a_002.d	5.956	Condition		Sample	P1-A3	10.0								
07202021a_003.d	5.956	Condition		Sample	P1-A3	10.0								
07202021a_004.d	5.956	Condition		Sample	P1-A3	10.0								
07202021a_005.d	5.956	Condition		Sample	P1-A3	10.0								
07202021a_006.d	5.956	Condition		Sample	P1-A3	10.0								
07202021a_007.d	5.956	Condition		Sample	P1-A3	10.0								
07202021a_008.d	5.956	687-2M21 0.05 pg/µL		Calibration	Vial 5	10.0				15.06				
07202021a_009.d	5.956	687-2M20 0.10 pg/µL		Calibration	Vial 4	10.0				32.94				
07202021a_010.d	5.942	687-2M19 0.25 pg/µL		Calibration	Vial 3	10.0				82.96				
07202021a_011.d	5.942	687-2M18 0.50 pg/µL		Calibration	Vial 2	10.0				177.30				
07202021a_012.d	5.929	687-2M17 1.0 pg/µL		Calibration	Vial 1	10.0				353.29				
07202021a_013.d	-----	29148	DBA-TX380	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
07202021a_014.d	-----	29148	DBA-TX380	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
07202021a_015.d	5.969	29148C0.01R4	DBA-TX380	Sample	P1-A2	10.0	0.100	10.0	0.1000	34.81	0.1025	0.0102		
07202021a_016.d	5.969	29148C0.01R4	DBA-TX380	Sample	P1-A2	10.0	0.100	10.0	0.1000	35.97	0.1057	0.0106	0.0104	104%
07202021a_017.d	5.942	687-2M20 0.10 pg/µL		Calibration	Vial 4	10.0				37.02				
07202021a_018.d	5.956	29148C1.0R4	DBA-TX380	Sample	P1-A3	10.0	0.100	400.0	0.0025	96.55	0.2766	1.1063		
07202021a_019.d	5.956	29148C1.0R4	DBA-TX380	Sample	P1-A3	10.0	0.100	400.0	0.0025	98.84	0.2830	1.1321	1.12	112%
07202021a_020.d	-----	29150	DBC-TX380	Sample	P1-A4	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
07202021a_021.d	-----	29150	DBC-TX380	Sample	P1-A4	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
07202021a_022.d	5.942	687-2M19 0.25 pg/µL		Calibration	Vial 3	10.0				85.85				
07202021a_023.d	-----	29151	DBD-TX380	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
07202021a_024.d	-----	29151	DBD-TX380	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01	<0.01	
07202021a_025.d	5.942	687-2M20 0.10 pg/µL		Calibration	Vial 4	10.0				36.73				

Calculated LOD at 0.045 pg/µL = 14

Calculated LOQ at 0.10 pg/µL = 34

"-----" denotes response below area threshold



Calibration STD

D:\MassHunter\F\lonicamid\08550_Onion\Data\Results\07202021\07202021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\F\lonicamid\08550_Onion\Data\Results\07202021\07202021a_011.d	Calibration	1	☒	353.29	1.0000
D:\MassHunter\F\lonicamid\08550_Onion\Data\Results\07202021\07202021a_010.d	Calibration	2	☒	177.30	0.5000
D:\MassHunter\F\lonicamid\08550_Onion\Data\Results\07202021\07202021a_009.d	Calibration	3	☒	82.96	0.2500
D:\MassHunter\F\lonicamid\08550_Onion\Data\Results\07202021\07202021a_017.d	Calibration	3	☒	85.85	0.2500
D:\MassHunter\F\lonicamid\08550_Onion\Data\Results\07202021\07202021a_016.d	Calibration	4	☒	32.94	0.1000
D:\MassHunter\F\lonicamid\08550_Onion\Data\Results\07202021\07202021a_015.d	Calibration	4	☒	37.02	0.1000
D:\MassHunter\F\lonicamid\08550_Onion\Data\Results\07202021\07202021a_014.d	Calibration	4	☒	36.73	0.1000
D:\MassHunter\F\lonicamid\08550_Onion\Data\Results\07202021\07202021a_008.d	Calibration	5	☒	15.06	0.0500

Compound Injection Date
TFNG 7/20/20217/21/21 *adm*

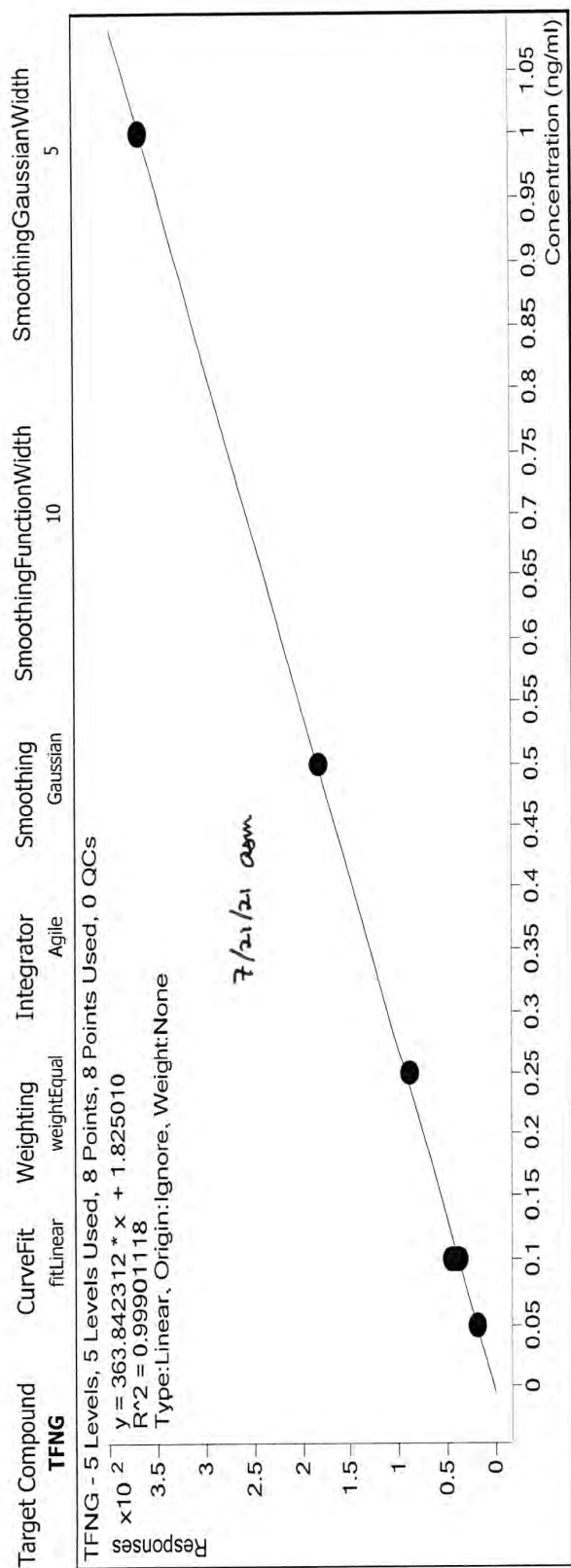
08550 - Flonicamid/Onion - Field Samples TX380 - Extracted 7/20/2021 by ASM

Data File	RT	SampleName	Sample Info	Sample Type	Vial Pos	Inj Vol (µL)	g	mL Vol	mg Inj	Resp	Conc (pg/µL)	ppm	Ave ppm	% Rec
07202021a_001.d	-----	Condition		Sample	P1-A3	10.0								
07202021a_002.d	7.171	Condition		Sample	P1-A3	10.0								
07202021a_003.d	7.158	Condition		Sample	P1-A3	10.0								
07202021a_004.d	7.158	Condition		Sample	P1-A3	10.0								
07202021a_005.d	7.171	Condition		Sample	P1-A3	10.0								
07202021a_006.d	7.171	Condition		Sample	P1-A3	10.0								
07202021a_007.d	7.171	Condition		Sample	P1-A3	10.0								
07202021a_008.d	7.171	687-2M21 0.05 pg/µL		Calibration	Vial 5	10.0				19.97				
07202021a_009.d	7.171	687-2M20 0.10 pg/µL		Calibration	Vial 4	10.0				39.93				
07202021a_010.d	7.171	687-2M19 0.25 pg/µL		Calibration	Vial 3	10.0				89.23				
07202021a_011.d	7.158	687-2M18 0.50 pg/µL		Calibration	Vial 2	10.0				181.08				
07202021a_012.d	7.158	687-2M17 1.0 pg/µL		Calibration	Vial 1	10.0				368.18				
07202021a_013.d	-----	29148	DBA-TX380	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
07202021a_014.d	-----	29148	DBA-TX380	Sample	P1-A1	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
07202021a_015.d	7.171	29148C0.01R4	DBA-TX380	Sample	P1-A2	10.0	0.100	10.0	0.1000	41.14	0.1080	0.0108		
07202021a_016.d	7.171	29148C0.01R4	DBA-TX380	Sample	P1-A2	10.0	0.100	10.0	0.1000	37.58	0.0983	0.0098	0.0103	103%
07202021a_017.d	7.158	687-2M20 0.10 pg/µL		Calibration	Vial 4	10.0				37.93				
07202021a_018.d	7.171	29148C1.0R4	DBA-TX380	Sample	P1-A3	10.0	0.100	400.0	0.0025	105.66	0.2854	1.1415		
07202021a_019.d	7.171	29148C1.0R4	DBA-TX380	Sample	P1-A3	10.0	0.100	400.0	0.0025	104.39	0.2819	1.1276	1.13	113%
07202021a_020.d	-----	29150	DBC-TX380	Sample	P1-A4	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
07202021a_021.d	-----	29150	DBC-TX380	Sample	P1-A4	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
07202021a_022.d	7.171	687-2M19 0.25 pg/µL		Calibration	Vial 3	10.0				88.36				
07202021a_023.d	-----	29151	DBD-TX380	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
07202021a_024.d	-----	29151	DBD-TX380	Sample	P1-A5	10.0	0.100	10.0	0.1000	-----	<0.10	<0.01		
07202021a_025.d	7.171	687-2M20 0.10 pg/µL		Calibration	Vial 4	10.0				44.94				

Calculated LOD at 0.045 pg/µL = 18

Calculated LOQ at 0.10 pg/µL = 38

"-----" denotes response below area threshold

**Calibration STD**

D:\MassHunter\FI\onidamide\08550_Onion\Data\Results\07202021\07202021a_012.d	Cal Type	Level	Enabled	Response	Exp Conc
D:\MassHunter\FI\onidamide\08550_Onion\Data\Results\07202021\07202021a_011.d	Calibration	1	☒	368.18	1.0000
D:\MassHunter\FI\onidamide\08550_Onion\Data\Results\07202021\07202021a_010.d	Calibration	2	☒	181.08	0.5000
D:\MassHunter\FI\onidamide\08550_Onion\Data\Results\07202021\07202021a_009.d	Calibration	3	☒	89.23	0.2500
D:\MassHunter\FI\onidamide\08550_Onion\Data\Results\07202021\07202021a_008.d	Calibration	3	☒	88.36	0.2500
D:\MassHunter\FI\onidamide\08550_Onion\Data\Results\07202021\07202021a_007.d	Calibration	4	☒	39.93	0.1000
D:\MassHunter\FI\onidamide\08550_Onion\Data\Results\07202021\07202021a_017.d	Calibration	4	☒	37.93	0.1000
D:\MassHunter\FI\onidamide\08550_Onion\Data\Results\07202021\07202021a_025.d	Calibration	4	☒	44.94	0.1000
D:\MassHunter\FI\onidamide\08550_Onion\Data\Results\07202021\07202021a_008.d	Calibration	5	☒	19.97	0.0500

ATTACHMENT C: STANDARD USE FORMSIR-4 Western Region Laboratory and
Trace Analytical Laboratory

SOP Number: 601

REFERENCE STANDARD RECEIPT/USEChemical IKI-220 Standard # 687Parent IKI-220 Date Received 05/12/20Source¹ ISK Biosciences Certified By MRIGlobalLot # 9803 Physical Form solidOriginal Expiration Date² 11/06/23 Original Purity² 99.8%Extended Expiration Date/Purity/Recertified By³:

1)	<u> </u>	<u>/</u>	<u> </u>	<u>%/</u>	<u> </u>	Initial	<u> </u>	Date	<u> </u>
2)	<u> </u>	<u>/</u>	<u> </u>	<u>%/</u>	<u> </u>	Initial	<u> </u>	Date	<u> </u>
3)	<u> </u>	<u>/</u>	<u> </u>	<u>%/</u>	<u> </u>	Initial	<u> </u>	Date	<u> </u>
4)	<u> </u>	<u>/</u>	<u> </u>	<u>%/</u>	<u> </u>	Initial	<u> </u>	Date	<u> </u>

Notes Rec'd with dry ice. AKA: Flonicamid. PAI stands for Pesticide Active Ingredient.CoA Rec'd ? Yes CoA and label agree: Yes (M)SDS: Yes Storage: FreezerSignature *Braeson Hung* Date 5/12/20**USE RECORDS**

Location	Date	Amount Used (g)	Balance ID	Initial	Amount Left (g)	Standard Stock #
Freezer 6	<u>5/12/20</u>	Initial		<u>BH</u>	0.3249 g	
<u>Freezer #6</u>	<u>5/20/20</u>	<u>0.02497</u>	<u>Scrappy</u>	<u>BH</u>	<u>0.29993</u>	<u>687-1</u>
<u>Freezer #6</u>	<u>5/03/2021</u>	<u>0.02505</u>	<u>Scrappy</u>	<u>adm</u>	<u>0.27488</u>	<u>687-2</u>

¹Enter the Registrant from the Protocol or the chemical company providing the standard.²Enter the original expiration date and purity and when the standard was received.³When the standard is recertified enter the extended expiration date, purity and company that recertified the standard.

*****Write Standard # on CoA and Original Standard Container*****

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

SOP Number: 601

REFERENCE STANDARD RECEIPT/USE

Chemical TFNA-AM Standard # 688

Parent IKI-220 (Flonicamid) Date Received 05/12/20

Source ¹ISK Biosciences Certified By MRIGlobal

Lot # 0006 Physical Form solid

Original Expiration Date ² 11/19/20 Original Purity ² 99.9%

Extended Expiration Date/Purity/Recertified By ³

1) 11/5/2025 / 99.9 % / MRIGlobal Initial BH Date 11/17/20
 2) / / % / Initial Date
 3) / / % / Initial Date
 4) / / % / Initial Date

Notes Rec'd with dry ice. AKA: 4-Trifluoromethylnicotinamide.

CoA Rec'd ? Yes CoA and label agree: Yes (M)SDS: Yes Storage: Freezer

Signature *Benson Hung* Date 5/12/20

USE RECORDS

Location	Date	Amount Used (g)	Balance ID	Initial	Amount Left (g)	Standard Stock #
Freezer 6	<u>5/12/20</u>	Initial		<u>BH</u>	0.3035 g	
<u>Freezer #6</u>	<u>5/20/20</u>	<u>0.02509</u>	<u>Scrappy</u>	<u>BH</u>	<u>0.27841</u>	<u>688-1</u>
<u>Freezer #6</u>	<u>5/03/21</u>	<u>0.02517</u>	<u>Scrappy</u>	<u>ABM</u>	<u>0.25324</u>	<u>688-2</u>

¹Enter the Registrant from the Protocol or the chemical company providing the standard.

²Enter the original expiration date and purity and when the standard was received.

³When the standard is recertified enter the extended expiration date, purity and company that recertified the standard.

*****Write Standard # on CoA and Original Standard Container*****

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

SOP Number: 601

REFERENCE STANDARD RECEIPT/USE

Chemical TFNA Standard # 689

Parent IKI-220 (Flonicamid) Date Received 05/12/20

Source ¹ISK Biosciences Certified By MRIGlobal

Lot # 0006 Physical Form solid

Original Expiration Date ² 11/17/20 Original Purity ² 100.0%

Extended Expiration Date/Purity/Recertified By ³

1) 11/5/2025 / 100.0 % / MRIGlobal Initial BH Date 11/17/20
 2) / / Initial Date
 3) / / Initial Date
 4) / / Initial Date

Notes Rec'd with dry ice. AKA: 4-Trifluoromethylnicotinic acid.

CoA Rec'd ? Yes CoA and label agree: Yes (M)SDS: Yes Storage: Freezer

Signature  Date 5/12/20

USE RECORDS

Location	Date	Amount Used (g)	Balance ID	Initial	Amount Left (g)	Standard Stock #
Freezer 6	<u>5/12/20</u>	Initial		<u>BH</u>	0.3499 g	
<u>Freezer #6</u>	<u>5/20/20</u>	<u>0.02503</u>	<u>Scrappy</u>	<u>BH</u>	<u>0.32487</u>	<u>689-1</u>
<u>Freezer #6</u>	<u>5/03/21</u>	<u>0.02514</u>	<u>Scrappy</u>	<u>adm</u>	<u>0.29973</u>	<u>689-2</u>

¹Enter the Registrant from the Protocol or the chemical company providing the standard.

²Enter the original expiration date and purity and when the standard was received.

³When the standard is recertified enter the extended expiration date, purity and company that recertified the standard.

*****Write Standard # on CoA and Original Standard Container*****

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

SOP Number: 601

REFERENCE STANDARD RECEIPT/USE

Chemical TFNG Standard # 690

Parent IKI-220 (Flonicamid) Date Received 05/12/20

Source ¹ISK Biosciences Certified By CERI

Lot # 201511 Physical Form solid

Original Expiration Date ² 10/31/21 Original Purity ² 99.7%

Extended Expiration Date/Purity/Recertified By ³:

1)	<u>/</u>	<u>%</u>	Initial	Date
2)	<u>/</u>	<u>%</u>	Initial	Date
3)	<u>/</u>	<u>%</u>	Initial	Date
4)	<u>/</u>	<u>%</u>	Initial	Date

Notes Rec'd with dry ice. AKA: N-(4-trifluoromethylnicotinoyl) glycine. CoA did not indicate the original archival location. Will contact the source.

CoA Rec'd ? Yes CoA and label agree: Yes (M)SDS: Yes Storage: Freezer

Signature *Bronson Harg* Date 5/12/20

USE RECORDS

Location	Date	Amount Used (g)	Balance ID	Initial	Amount Left (g)	Standard Stock #
Freezer 6	5/12/20	Initial		BH	0.3171 g	
Freezer #6	5/20/20	0.02512	Scrappy	BH	0.29198	690-1
Freezer #6	5/03/21	0.02506	Scrappy	ABM	0.26692	690-2

¹Enter the Registrant from the Protocol or the chemical company providing the standard.

²Enter the original expiration date and purity and when the standard was received.

³When the standard is recertified enter the extended expiration date, purity and company that recertified the standard.

*****Write Standard # on CoA and Original Standard Container*****

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

SOP Number: 602

STANDARD USE FORM - STOCK

Name IKI-220* Purity 99.8 % Source MRIGlobal ISK BiosciencesEE
BH
11/16/21Date Received 05/12/2020 Expiration Date 11/6/2023 Lot # 9803

Stock Solution:

 $(\underline{0.02497} \text{ g} \times 99.8\% \text{ Purity} \times 1000 \text{ mg/g}) / \underline{25} \text{ mL} = \underline{1.0} \text{ mg/mL}$ Solvent: Acetonitrile Storage: FreezerStock Standard # 687-1 Expiration Date 5/20/2020EE
BH
5/20/20Prepared by Broms and Hugg Date 5/20/2020

ALIUOTS

Date	mL Removed	Syringe/Pipette Size	Spike or Dilution	New Std #	Expiration Date	Initial
5/20/20	2.5	5mL Pipetman	D	687-1M1	11/17/20	BH
— DISCARDED 5/03/2021 abm						
2/11/21	2.5	5mL Pipetman	D	687-1M4	5/20/21	abm

Late Entry
5/3/21 abm

* Flonicamid

Revised on: 6/03/08

Page # _____

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

SOP Number: 602

STANDARD USE FORM - STOCK

Name IKI-220 (Flonicamid) Purity 99.8 % Source ISK Biosciences

~~EE-SANITATION~~ EE 11/16/21 ~~abm~~
~~MRE-Global~~ ISK Biosciences

Date Received 5/12/2020 Expiration Date 11/06/2023 Lot # 9803

Stock Solution:

$\frac{0.02505}{(\text{g})} \times 99.8 \% \text{ Purity} \times 1000\text{mg/g}) / 25 \text{ mL} = 1.0 \text{ mg/mL}$

Solvent Acetonitrile Storage: RT/ R ☒ F / Other _____

Stock Standard # 687-2 Expiration Date 5/03/2022

Prepared by Alex McFall Date 5/03/2021

ALIQUOTS

Date	mL Removed	Syringe/Pipette Size	Spike or Dilution	New Std #	Expiration Date	Initial
5/3/21	2.5	5 mL Pipetman	D	687-2M1	11/03/21	abm

Revised on: 10/06/2020

Page # _____

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

SOP Number: 602

STANDARD USE FORM - STOCK

EE BH 11/16/21

Name TFNA-AM* Purity 99.9 % Source MRIGlobal ISK BiosciencesDate Received 05/12/2020 Expiration Date 11/19/2020 Lot # 0006

Stock Solution:

0.02509 g X 99.9 % Purity X 1000mg/g / 25 mL = 1.0 mg/mLSolvent: Acetonitrile Storage: FreezerStock Standard # 688-1 Expiration Date 11/19/2020New CoA
BH
2/10/21Prepared by Bramson Hugg Date 5/20/2020

ALiquots

Date	mL Removed	Syringe/Pipette Size	Spike or Dilution	New Std #	Expiration Date	Initial
5/20/20	2.5	5 mL Pipetman	D	687-1M1	11/17/20	BH
— DISCARDED 5/3/2021 adm —						
2/11/21	2.5	5 mL Pipetman	D	687-1M4	5/20/21	adm

Late Entry
5/3/21 adm

* 4-trifluoromethylnicotinamide

Revised on: 6/03/08

Page # _____

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

SOP Number: 602

STANDARD USE FORM - STOCK

Name TFNA-AM Purity 99.9 % Source ISK BioSciences
EE 5/11/21 dm EE 11/16/21 dm ARE GIDDA

Date Received 5/12/2020 Expiration Date 11/05/2025 Lot # 0006

Stock Solution:

(0.02517 g X 99.9 % Purity X 1000mg/g) / 25 mL) = 1.0 mg/mL

Solvent Acetonitrile Storage: RT/ R ☒ (F) Other _____

Stock Standard # 688-2 Expiration Date 5/03/2022

Prepared by Alex McFall Date 5/03/2021

ALIQUOTS

Date	mL Removed	Syringe/Pipette Size	Spike or Dilution	New Std #	Expiration Date	Initial
5/3/21	2.5	5mL Pipetman	D	687-2M1	11/03/21	adm

Revised on: 10/06/2020

Page # _____

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

SOP Number: 602

STANDARD USE FORM - STOCK

EE BH 11/16/21

Name TFNA* Purity 100.0 % Source MRI Global ~~ISK Biosciences~~Date Received 05/12/2020 Expiration Date 11/17/2020 Lot # 0006

Stock Solution:

0.02503
(g X 100.0 % Purity X 1000mg/g) / 25 mL = 1.0 mg/mLSolvent: Acetonitrile Storage: FreezerStock Standard # 689-1 Expiration Date 11/17/2020

5/20/2021

New CoA

BH

2/10/21

Prepared by Bronson Hung Date 5/20/20

ALIQUOTS

Date	mL Removed	Syringe/Pipette Size	Spike or Dilution	New Std #	Expiration Date	Initial
5/20/20	2.5	5 mL Pipetman	D	687-1M1	11/17/20	BH
	DISCARDED	5/3/2021 agm				
2/11/21	2.5	5 mL Pipetman	D	687-1M4	5/20/21	agm

Late Entry
5/3/21 agm

* 4-trifluoromethylnicotinic acid

Revised on: 6/03/08

Page # _____

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

SOP Number: 602

STANDARD USE FORM - STOCK

Name TFNA Purity 100.0 % Source ISK Biosciences

~~EE 5/11/21 adm~~ EE 11/16/21 adm
~~ARE Global~~ ISK Biosciences

Date Received 5/12/2020 Expiration Date 11/05/2025 Lot # 0006

Stock Solution:

(^{0.02514} 0.02514 g X 100.0 % Purity X 1000mg/g) / 25 mL) = 1.0 mg/mL

Solvent Acetonitrile Storage: RT/ R ☒ (F) Other _____

Stock Standard # 689-2 Expiration Date 5/03/2022

Prepared by Alex Medall Date 5/03/2021

ALIQUOTS

Date	mL Removed	Syringe/Pipette Size	Spike or Dilution	New Std #	Expiration Date	Initial
5/3/21	2.5	5 mL Pipetman	D	687-2M1	11/03/21	adm

Revised on: 10/06/2020

Page # _____

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

SOP Number: 602

STANDARD USE FORM - STOCK

Name TFNG* Purity 99.7% Source CERT Isk Biosciences ^{EE BH 11/16/21}

Date Received 05/12/2020 Expiration Date 10/31/2021 Lot # 201511

Stock Solution:

0.02512
(0.02512 g X 99.7% Purity X 1000mg/g) / 25 mL = 1.0 mg/mL

Solvent: Acetonitrile Storage: Freezer

Sonicated stock solution for 1 mins.

Stock Standard # 690-1 Expiration Date 5/20/2021

Prepared by Brunson Hung Date 5/20/2020

ALIQUOTS

^{EE} Δ 687-1M1 BH 5/20/20

Date	mL Removed	Syringe/Pipette Size	Spike or Dilution	New Std #	Expiration Date	Initial
5/20/20	2.5	5 mL Pipetman	D	687-15 ^Δ	11/17/20	BH
	DISCARDED	5/3/2021 adm				
2/11/21	2.5	5 mL Pipetman	D	687-1M4	5/20/21	adm

Late Entry
5/3/21 adm

* N-(4-trifluoromethylnicotinoyl) glycine

Revised on: 6/03/08

Page # _____

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

SOP Number: 602

STANDARD USE FORM - STOCK

Name TFNG Purity 99.7 % Source ISK Biosciences

EE 5/11/21 am - EE 11/16/21 am
MRI Global - CERT - EE
ISK Biosciences ISK 11/16/21 am
Biosciences

Date Received 5/12/2020 Expiration Date 10/31/2021 Lot # 201511

Stock Solution:

$$(0.02506 \text{ g} \times 99.7 \% \text{ Purity} \times 1000 \text{ mg/g}) / 25 \text{ mL} = 1.0 \text{ mg/mL}$$

Solvent Acetonitrile Storage: RT/ R ☒ F Other _____

Stock Standard # 690-2 Expiration Date 10/31/21 (COA)

Prepared by Alex McFall Date 5/03/2021

EE adm
5/3/21

ALIQUOTS

[illegible]

Revised on: 10/06/2020

Page # _____

SOP Number: 602

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

STANDARD USE FORM - DILUTIONS

Date	Compound 1		Compound 2		Compound 3		Compound 4		Mixture of Compounds		Final Vol (mL)	Final Conc.	New Std#	Solvent	Exp. Date	Storage	Initials
Year 2021	IKI-220 *		TFNA-AM**		TFNA***		TFNG****		Mixed							RT/ R/F/ O	
	Std#	mL	Std#	mL	Std#	mL	Std#	mL	Std#	mL							
2/11	687-1	2.5	688-1	2.5	689-1	2.5	690-1	2.5			25	100 µg/mL	IM4 687-2M1	ACN	5/20/21	F	abm
2/11									IM4 687-2M1	2.5	25	10 µg/mL	IM5 687-2M2	ACN	5/20/21	F	abm
2/11									IM5 687-2M2	2.5	25	1.0 µg/mL	IM6 687-2M3	ACN	5/20/21	F	abm
4/22									IM6 687-2M3	2.5	25	0.10 µg/mL	IM7 687-2M4	ACN	5/20/21	F	abm
4/22									IM7 687-2M4	2.5	25	0.010 µg/mL	IM8 687-2M5	ACN	5/20/21	F	abm
									IM8 687-2M5	2.5	25	1.0 pg/µL	IM9 687-2M6	Δ*	5/6/21	R	abm
									IM9 687-2M6	1.25	25	0.5 pg/µL	IM10 687-2M7	Δ*	5/6/21	R	abm
									IM10 687-2M7	0.625	25	0.25 pg/µL	IM11 687-2M8	Δ*	5/6/21	R	abm
									IM11 687-2M8	0.250	25	0.1 pg/µL	IM12 687-2M9	Δ*	5/6/21	R	abm
									IM12 687-2M9	0.125	25	0.05 pg/µL	IM13 687-2M10	Δ*	5/6/21	R	abm

*Flonicamid, **4-trifluoromethylnicotinamide, ***4-trifluoromethylnicotinic acid, ****N-(4-trifluoromethylnicotinoyl) glycine

ACN=Acetonitrile

□ EE, std numbering should have cont'd at IM4

EE 5/3/21 abm

5:45 Δ 50150 Acetonitrile:water

EE abm

4/22/21 abm

Revised on: 05/12/2020

EE abm 5/3/21

4/22/21

as LE

Page

11/15/21 abm

Att

SOP Number: 602

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

STANDARD USE FORM - DILUTIONS

Date	Compound 1		Compound 2		Compound 3		Compound 4		Mixture		Final Vol. (mL)	Final Conc.	New Std#	Solvent	Exp. Date	Storage	Initials
	Std#	mL	Std#	mL	Std#	mL	Std#	mL	Std#	mL							
Year 2021	IK1-220*		TFNA-AM**		TFNA***		TFNG****		Mixed							RT/ R/F/ O	
4/27								1m8	687-2M5	2.5	25	1.0 pg/μL	1m8 687-2M11	Δ	5/06/21	F	adm
4/27									687-2M5	1.25	25	0.50 pg/μL	1m8 687-2M12	Δ	5/06/21	F	adm
4/27									687-2M5	0.625	25	0.25 pg/μL	1m8 687-2M13	Δ	5/06/21	F	adm
4/27									687-2M5	0.250	25	0.10 pg/μL	1m8 687-2M14	Δ	5/06/21	F	adm
4/27									687-2M5	0.125	25	0.05 pg/μL	1m8 687-2M15	Δ	5/06/21	F	adm
4/28									687-2M5	2.5	25	1.0 pg/μL	1m8 687-2M16	Δ	5/06/21	F	adm

*Flonicamid, **4-trifluoromethylnicotinamide, ***4-trifluoronicotinic acid, ****N-(4-trifluoromethylnicotinoyl) glycine

Δ 5:5 Acetonitrile: Water

* NOTE: Entire sheet completed as late entry 4/29/21 adm

Page _____

Revised on: 6/03/08

SOP Number: 602

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

STANDARD USE FORM - DILUTIONS

Date	Compound 1		Compound 2		Compound 3		Compound 4		Mixture of Compounds		Final Vol (mL)	Final Conc.	New Std#	Solvent	Exp. Date	Storage	Initials
Year 2020	IK1-220 *		TFNA-AM**		TFNA***		TFNG****		Mixed							RT/ R/F/ O	
	Std#	mL	Std#	mL	Std#	mL	Std#	mL	Std#	mL							
5/20	687-1	2.5	688-1	2.5	689-1	2.5	690-1	2.5			25	100µg/mL	687-1M1	ACN	11/17/20	F	BH
5/20									687-1M1	2.5	25	10µg/mL	687-1M2	ACN	11/17/20	F	BH
5/20									687-1M2	2.5	25	1.0µg/mL	687-1M3	ACN	11/17/20	F	BH

*Flonicamid, **4-trifluoromethylnicotinamide, ***4-trifluoromethylnicotinic acid, **** N-(4-trifluoromethylnicotinoyl) glycine
ACN=Acetonitrile

Revised on: 05/12/2020

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SOP Number: 602

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

STANDARD USE FORM - DILUTIONS

Date	Compound 1		Compound 2		Compound 3		Compound 4		Mixture		Final Vol. (mL)	Final Conc.	New Std#	Solvent	Exp. Date	Storage	Initials
	Std#	mL	Std#	mL	Std#	mL	Std#	mL	Std#	mL							
Year 2021	IKI-220*		TFNA-AM**		TFNA***		TFNG****		Mixed							RT/R/F/O	
5/03	687-2	2.5	688-2	2.5	689-2	2.5	690-2	2.5			25	100 µg/mL	687-2M1	ACN	10/31/21	F	CBM
5/03									687-2M1	2.5	25	10 µg/mL	687-2M2	ACN	10/31/21	F	CBM
5/03									687-2M2	2.5	25	1.0 µg/mL	687-2M3	ACN	10/31/21	F	CBM
5/03									687-2M3	2.5	25	0.10 µg/mL	687-2M4	ACN	10/31/21	F	CBM
5/03									687-2M4	2.5	25	0.010 µg/mL	687-2M5	ACN	10/31/21	F	CBM

*Flonicamid, **4-trifluoromethylnicotinamide, ***4-trifluoronicotinic acid, ****N-(4-trifluoromethylnicotinoyl) glycine

Revised on: 6/03/08

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SOP Number: 602

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

STANDARD USE FORM - DILUTIONS

Date	Compound 1		Compound 2		Compound 3		Compound 4		Mixture		Final Vol. (mL)	Final Conc.	New Std#	Solvent	Exp. Date	Storage	Initials
	Std#	mL	Std#	mL	Std#	mL	Std#	mL	Std#	mL							
Year 2021	IKI-220*		TFNA-AM**		TFNA***		TFNG****		Mixed							RT/R/F/O	
5/04									687-2M5	2.5	25	1.0 pg/μL	687-2M6	Δ	5/18/21	F	agm
5/04									687-2M5	1.25	25	0.50 pg/μL	687-2M7	Δ	5/18/21	F	agm
5/04									687-2M5	0.625	25	0.25 pg/μL	687-2M8	Δ	5/18/21	F	agm
5/04									687-2M5	0.250	25	0.10 pg/μL	687-2M9	Δ	5/18/21	F	agm
5/04									687-2M5	0.125	25	0.05 pg/μL	687-2M10	Δ	5/18/21	F	agm

*Flonicamid, **4-trifluoromethylnicotinamide, ***4-trifluoronicotinic acid, ****N-(4-trifluoromethylnicotinoyl) glycine

Δ 5:95 Acetonitrile: Water

Revised on: 6/03/08

Page _____

SOP Number: 602

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

STANDARD USE FORM - DILUTIONS

[illegible]

*Flonicamid, **4-trifluoromethylnicotinamide, ***4-trifluoromethylnicotinic acid, ****N-(4-trifluoromethylnicotinoyl) glycine

Δ 5:95 Acetonitrile: Water

Page _____

Revised on: 6/03/08

SOP Number: 602

IR-4 Western Region Laboratory and
Trace Analytical Laboratory

STANDARD USE FORM - DILUTIONS

Date	Compound 1		Compound 2		Compound 3		Compound 4		Final Volume (mL)	Final Conc.	New Std#	Solvent	Exp. Date	Storage	Initials
	Std#	mL	Std#	mL	Std#	mL	Std#	mL							
							Mixed *							RT/R/ F/O	
5/19/21							687-2M5	2.5	2.5	1.0pg/mL	687-2M12	Δ	6/2/21	F	adm
5/19/21							687-2M5	1.25	2.5	0.50pg/mL	687-2M13	Δ	6/2/21	F	adm
5/19/21							687-2M5	0.625	2.5	0.25pg/mL	687-2M14	Δ	6/2/21	F	adm
5/19/21							687-2M5	0.250	2.5	0.1pg/mL	687-2M15	Δ	6/2/21	F	adm
5/19/21							687-2M5	0.125	2.5	0.05pg/mL	687-2M16	Δ	6/2/21	F	adm
7/20/21							687-2M5	2.5	2.5	1.0pg/mL	687-2M17	Δ	8/03/21	F	adm
7/20/21							687-2M5	1.25	2.5	0.50pg/mL	687-2M18	Δ	8/03/21	F	adm
7/20/21							687-2M5	0.625	2.5	0.25pg/mL	687-2M19	Δ	8/03/21	F	adm
7/20/21							687-2M5	0.250	2.5	0.10pg/mL	687-2M20	Δ	8/03/21	F	adm
7/20/21							687-2M5	0.125	2.5	0.05pg/mL	687-2M21	Δ	8/03/21	F	adm

5/19/21 adm X Mix of Flonicamid, TFNA-AM, TFNA, and TFNG
 43 LE
 11/15/21 adm Δ 5:95 Acetonitrile: Water
 Revised on: 6/03/08

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ATTACHMENT D: CERTIFICATES OF ANALYSIS



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

Certificate of Analysis

IKI-220 PAI, Lot No. 9803

Original data and GLP reserve sample are archived under MRIGlobal Project No. 310260.01.203.09.

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

MRIGlobal
425 Volker Boulevard
Kansas City, Missouri 64110

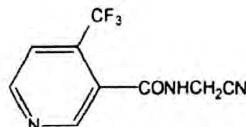
Study Sponsor

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

*On 5/12/20
Rec'd with
Std.
Std # 687
CT 5/12/20*

Compound Identification

Common Names:	IKI-220 PAI; Flonicamid
IUPAC Chemical Name:	N-(cyanomethyl)-4-(trifluoromethyl)nicotinamide
CA Chemical Name:	N-(cyanomethyl)-4-(trifluoromethyl)-3-pyridinecarboxamide
Empirical Formula:	C ₉ H ₆ F ₃ N ₃ O
Molecular Weight:	229.17
CAS Number:	158062-67-0
Structure:	



Experimental Techniques

Purity was determined using HPLC purity profiling methodology.
Water content was determined using Karl Fischer titrimetry.
Identity was previously verified using direct infusion mass spectrometry.

Quality

Purity (%):	99.8 ± 0.0% (w/w)
Water Content (%):	0.04 ± 0.00% (n = 2)
Identity (Direct Infusion):	Confirmed
HPLC Analysis Date:	November 6, 2018
Storage Conditions:	Frozen
Expiration Date:	November 6, 2023

Approved: Candice C. Dillon
Candice C. Dillon, Study Director
MRIGlobal

Date: 11/13/2018



MRIGlobal
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 MRIGlobal Project No. 310260.01.177.04

Data Requirement

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

Performing Laboratory

MRIGlobal
425 Volker Blvd
Kansas City, MO 64110

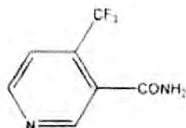
Study Sponsor

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 ₇ H ₅ F ₃ N ₂ O
Molecular Weight:	190.12
CAS Number:	158062-71-6
Lot No.:	0006

Structure:



*On 5/12/20
Rec'd with std.
Std# 688
CT 5/12/20*

Experimental Techniques

Purity was determined using HPLC impurity profiling methodology.
Structure was previously verified by Fourier transform infrared spectroscopy.

Quality

Purity (%):	99.9 ± 0.0 %
Identity:	Confirmed (FT-IR)
Storage Conditions:	Frozen (~ -20°C)
Date of HPLC Analysis:	November 19, 2015
Expiration Date:	November 19, 2020

Approved:

Paul J. Weller
Paul J. Weller, Study Director
Program Manager
MRIGlobal

Date:

January 5, 2016



MRIGlobal
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 samples are archived under MRIGlobal Project No. 310260.01.225.02.

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

MRIGlobal
425 Volker Boulevard
Kansas City, Missouri 64110

Study Sponsor

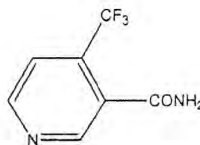
Ishihara Sangyo Kaisha, Ltd.
Biosciences Business Headquarters
3-15 Edobori 1-Chome, Nishi-ku
Osaka 550-0002 JAPAN

Compound Identification**Test Substance:**

TFNA-AM
Lot No. 0006

Common Name:
IUPAC Chemical Name:
Empirical Formula:
Molecular Weight:
CAS Number:
Structure:

TFNA-AM
4-Trifluoromethylnicotinamide
 $C_7H_5F_3N_2O$
190.12
158062-71-6



std # 688

Rec'd by e-mail
on 11/17/20

BLT
11/17/20

Experimental Techniques

Percent purity was determined using high performance liquid chromatography (HPLC) methodology.
Identity was previously confirmed using Fourier transform infrared (FT-IR) spectroscopy.

Quality

Percent Purity:	99.9 ± 0.0% (w/w)
Identity (FT-IR):	Confirmed
HPLC Analysis Date:	November 5, 2020
Storage Conditions:	Frozen (~ -20°C)
Expiration Date:	November 5, 2025

TRUE COPY OF ORIGINAL
THIS STAMP IS IN RED.
INITIALS *CDM*
DATE 2/11/21

Approved: Candice C. Dillon
Candice C. Dillon, Study Director
MRIGlobal

Date: 11/16/2020



MRIGlobal
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 MRIGlobal Project No. 310260.01.177.01

Data Requirement

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

Performing Laboratory

MRIGlobal
425 Volker Blvd
Kansas City, MO 64110

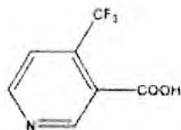
Study Sponsor

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

Compound Identification

Common Name: TFNA
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:

*Recd with std on 5/12/20**Std# 689**CT 5/12/20***Experimental Techniques**

Purity was determined using HPLC purity profiling methodology.
Identity was previously determined using Fourier transform infrared spectroscopy.

Quality

Purity (%): 100.0 ± 0.0 %
Identity: Confirmed (FT-IR)
Storage Conditions: Frozen (~-20°C)
Date of HPLC Analysis: November 17, 2015
Expiration Date: November 17, 2020

Approved: *Paul J. Weller*
Paul J. Weller, Study Director
Program Manager
MRIGlobal

Date: *January 5, 2016*



MRIGlobal
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 samples are archived under MRIGlobal Project No. 310260.01.225.01.

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

MRIGlobal
425 Volker Boulevard
Kansas City, Missouri 64110

Study Sponsor

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

Compound Identification**Test Substance:**

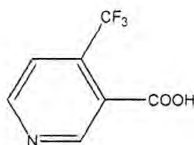
TFNA
Lot No. 0006

Common Name:
IUPAC Chemical Name:
Empirical Formula:
Molecular Weight:
CAS Number:
Structure:

TFNA
4-Trifluoromethylnicotinic acid
 $C_7H_5F_3NO_2$
191.11
158063-66-2

std # 689
Rec'd by e-mail
on 11/17/20

BH 11/17/20

**Experimental Techniques**

Percent purity was determined using high performance liquid chromatography (HPLC) methodology.
Identity was previously confirmed using Fourier transform infrared (FT-IR) spectroscopy.

Quality

Percent Purity: 100.0 ± 0.0% (w/w)
Identity (FT-IR): Confirmed
HPLC Analysis Date: November 5, 2020
Storage Conditions: Frozen (~ -20°C)
Expiration Date: November 5, 2025

Approved: Candice C. Dillon
Candice C. Dillon, Study Director
MRIGlobal

Date: 11/10/2020



Certificate of Analysis

1. Sponsor ISHIHARA SANGYO KAISHA, LTD.

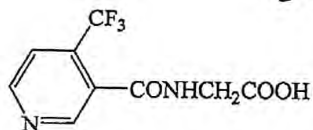
2. Study number 85801

3. Test item

Another name

TFNG

Structural formula

Molecular formula C₉H₇F₃N₂O₃

Molecular weight 248.16

4. Lot number 201511

5. Analytical result

Component	Purity (%)	Analytical method
TFNG	99.7 (%RSD=0.00, n=3)	High-performance liquid chromatography (HPLC)

6. Date of analysis November 1, 2018

7. Expiration date October 31, 2021

8. Storage condition Frozen

9. GLP compliance

This study was conducted in accordance with;

- Notification on the Good Laboratory Practice for Agricultural Chemicals "(Annex) The Standards for the Good Laboratory Practice (GLP) for Agricultural Chemicals" (October 1, 1999, No. 11-Nousan-6283; latest revision; October 31, 2016, No. 28-Syouan-3225)
- OECD Principles of Good Laboratory Practice, November 26, 1997, ENV/MC/CHEM (98)17

Chemicals Evaluation and Research Institute, Japan, Kurume

Date:

November 12, 2018

Study Director Signature:

Yoichi Watanabe

Attachment 4

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 ≤ 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).				
5.5 Are 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 _____