1. PROJECT TITLE :
AZOXYSTROBIN + FLUDIOXONIL + DIFENOCONAZOLE: Magnitude of the Residue on SWEET POTATO (POST HARVEST)

2. JUSTIFICATION AND OBJECTIVES:

IR-4 has received a request for the minor use of azoxystrobin + fludioxonil + difenoconazole on sweet potato (post harvest) for control of *Rhizopus* soft rot caused by *Rhizopus stolonifer* and black rot caused by *Ceratocystis fimbriata*.

To establish these tolerances, it is required that the magnitude of the residue in or on the commodity be determined as per EPA Series 860 Guidelines. The purpose of this study is to collect and analyze treated and untreated residue samples from appropriate field sites according to the application parameters requested to provide the sponsor with residue chemistry data to support a pesticide tolerance. Tolerances have been established for azoxystrobin, fludioxonil and difenoconazole on crop subgroup 1C (Vegetable, tuberous and corm) and a post harvest use exists for potato with the same use pattern and thus could potentially cover sweet potato. However, this residue data is required for European exports.

To determine the magnitude of residues of total azoxystrobin + fludioxonil + difenoconazole in or on sweet potato (post harvest), this protocol will be employed using appropriate Standard Operating Procedures (SOP's) and will be conducted under provisions outlined in 40 CFR Part 160 (**IN ACCORDANCE WITH EPA's GOOD LABORATORY PRACTICE STANDARDS**). Canadian field/processing/analytical trials, if any, will be conducted at facilities consistent with the provisions outlined in the Organization for Economic Cooperation and Development (OECD) Series on Principles of Good Laboratory Practice and Compliance Monitoring.

All study participants are **reminded** and **encouraged** to follow all appropriate campus, local, state (or provincial) and national regulations and laws in association with the safe use of pesticides.

3. SPONSOR/TESTING FACILITY NAME, ADDRESS AND PHONE:

IR-4 Project Headquarters, 500 College Road East, Suite 201 W, Princeton, NJ 08540, (732) 932-9575, FAX# (609) 514-2612.

4. STUDY DIRECTOR[1]:

Grace Lennon, IR-4 Project Headquarters, 500 College Road East, Suite 201 W, Princeton, NJ 08540, (732) 932-9575 X4627, FAX# (609) 514-2612, E-mail: galennon@njaes.rutgers.edu

5. PROPOSED DATES:

Experimental Start :	04/18
Experimental Termination:	9/19
Study Completion:	4/20

6. PROPOSED TEST SITES:

Field sites:	Refer to Section 23
Laboratory:	Refer to Section 24

7. STUDY AUTHORIZATION:

Deborah Carpenter April 19, 2018	Grace 4-24-18
Sponsor Representative / Date	Grace Lennon/ Study Director / Date

7.1 STUDY DIRECTOR INITIALS: Gal

[1]In case the Study Director is not available, contact Dr. Deborah Carpenter (x4637) or Dr. Daniel Kunkel (x4616) at IR-4 Headquarters (732) 932-9575 for guidance.

8. GOOD LABORATORY PRACTICE COMPLIANCE:

The appropriate cooperative testing facility (field and laboratory) will be responsible for certifying that its portion of the study will be conducted in accordance with EPA's Good Laboratory Practice (GLP) Standards, 40 CFR 160, amended and effective Oct. 16, 1989. A statement of compliance, together with any GLP deviations will be signed and submitted by the appropriate Research Directors in their report or data package.

9. QUALITY ASSURANCE:

Quality Assurance duties and responsibilities will be in conformance with 40 CFR 160.35. A Quality Assurance Statement will be submitted in the final report and shall include the date inspections were made and date(s) the findings were reported to the Study Director and management.

10. TEST SYSTEM/CROP:

SWEET POTATO (POST HARVEST) - Use a commercial variety. Report: variety, source, lot number, date received, and other descriptive information if available.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of a national residue tolerance; **see Section 23 for these assignments**. Refer to Section 11.4 for requirements to differentiate multiple trials by the same field researcher.

11. TEST SYSTEM DESIGN and STATISTICAL METHOD:

11.1 Each test site will consist of one untreated lot and one treated lot.

The individual lots shall be of adequate size to ensure that no more than 50% of the harvestable crop in the sampled lot will be needed to provide the necessary plant material. See Parts 17 & 18 for requirements for residue sampling.

Field trial 18-NC217 will provide samples for processing. The lots must be large enough to provide enough sample weight to meet processing requirements.

11.2 During treatment, drying, handling, packaging, etc. employ adequate buffer zones and/or separation (minimum 5 ft.) between each of the lots and good handling practices in order to prevent contamination between samples. Document buffer distances/separation mechanism (as appropriate) and handling practices in the field data notebook.

11.3 If this pesticide use is not registered on this crop, federal law requires that the treated crop must be destroyed or handled in such a way that it is not consumed as a human food or animal feed.

11.4 An independently prepared tank-mix must be used in each trial if a Field Research Director is assigned more than one trial in this study. Multiple trials at the same site must be conducted using at least 1) different application dates (at least 30 days) or 2) different varieties (confirm with the study director if this option is chosen.)

11.5 Mark lots with identifiable markers containing at minimum the Field ID number and treatment number or treatment name that will persist for the duration of the field research trial or that can be readily replaced.

11.6 This study is not designed for statistical evaluation of field data.

12. TEST SITE PREPARATION:

Select roots from a test site that has been maintained following good local agricultural practices for the production of sweet potato (post-harvest) including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

Note: Do not select sweet potatoes for this postharvest study that have been previously treated with azoxystrobin, fludioxonil or difenoconazole seed treatment or foliar use.

13. TEST/CONTROL SUBSTANCE:

Use the Stadium™ Fungicide formulation (1.197 lb azoxystrobin + 1.197 lb fludioxonil + 0.934 lb difenoconazole/gallon) (EPA Reg No. 100-1453, CAS No.: Difenoconazole: 119446-68-3; Azoxystrobin: 131860-33-8; Fludioxonil: 131341-86-1) that has been characterized to meet GLP standards. IR-4 Headquarters personnel will arrange procurement of GLP test substance from the Registrant. Upon receipt, document the lot/batch number, condition, quantity received and if GLP characterized. **Temperature monitoring should begin within 2 days of receipt of the test substance, regardless of where it is held or stored.**

Contact the Study Director if there are any concerns regarding the GLP status, labeled identification, expiration date, etc. of the test substance.

The registrant will provide a copy of the Certificate of Analysis to IR-4 Headquarters.

Store the test substance in a secure, clean, dry area and document storage temperatures.

EPA regulations require that test substance container(s) must be retained until the final study report is completed.

Study completion can be confirmed by contacting the Study Director or the Regional Field Coordinator, or by searching the IR-4 web site; click on "Food Crops" and under the "IR-4 Food Crops Database" click on the "Test Substance Container Disposal Approval" link. URL: http://ir4.rutgers.edu/FoodUse/Food_UseSimple3.cfm

If test substance containers are shipped to another location, the shipment must be conducted in accordance with local, state, and Federal regulations. Registrant representative: Dr. Dirk Drost, (336) 632-7510, FAX# 336-632-6021, e-mail: dirk.drost@syngenta.com.

The registrant will archive a retention sample of the test substance. Control substances are not relevant to this study.

An optional tracking form that may be used to confirm that the correct test substance has been received (with the correct label and Certificate of Analysis) is available at: http//wrir4.ucdavis.edu/Resources/Tricks/default.html.

14. TEST SUBSTANCE APPLICATION:

14.1 Simulate commercial application practices by applying the test substance in a manner that represents a representative application technique that is used by area commercial growers, while following the directions specified in Section 15.

- Use application equipment that will provide uniform application of the test substance (see Protocol Section 15 for the spray volume range).
- The test substance, if applied in a mixture, must be applied to the test system within 30 minutes of mixing, otherwise the mixture must be agitated just prior to making the application to ensure that it is well mixed. (The additional agitation should be documented in Part 6G of the Field Data Book.) The mixture must always be applied to the test system within 2 hours of mixing.
- Each field trial requires a unique spray mixture. Do not use the spray mixture from one field trial on another field trial.

14.2 Applied only to treatment 02. Full Calibrations for output and speed (conveyer systems) must be performed to ensure accurate delivery.
A calibration consists of a minimum of 3 consecutive, documented checks for nozzle or hopper output and speed (equipment). An output calibration is a 3 run discharge of all the nozzles. An output recheck is a single run discharge of all the nozzles. A speed (equipment) calibration is 3 runs. A speed (equipment) recheck is a single run.

Verification of the actual amount of test substance applied will always be made using **the most recent complete calibration data for that equipment.** (Note: When the most recent calibration data is from another trial, a certified true copy of that data must be included in the field data book for this trial.)

Discharge/Output Calibrations:

Is this the first application of test substance in this trial?

YES: A full calibration is required just prior to the first application (allowable the day before the application, but calibration on the day of use is preferred).

NO: A single run recheck may be conducted to confirm consistent delivery (within ±5% of the last complete calibration) just prior to subsequent applications. (Full calibrations are preferred.)

Recheck is required when:

1. Full calibration data from another trial is used.
2. The equipment has been moved from the location where the most recent full calibration or recheck has occurred. (A sprayer that has been calibrated or rechecked at a farm or research station and then used to make an application somewhere else on that same farm or research station is *not* considered to have been "moved".)
3. The equipment has been cleaned.
4. Nozzles are removed and placed back on.

Recheck is not required when the same Field Research Director is making applications on the same day for multiple trials in this study, or multiple treatments in the same trial, unless there have been changes in other application parameters as described above.

Full output calibration is required if:

1. This is the first application in the trial

2. Application parameters or equipment components have changed (other than changing out CO_2 tanks) including:
    a. Nozzle or hopper output
    b. Nozzle size or type
    c. Change in delivery pressure by more than 5% (even if it has been changed back to the pressure used during the initial calibration)
3. A recheck is not within ±5% of the last complete calibration.
4. The discharge of any single nozzle during a run of a full calibration or a recheck is greater than ±5% of the mean of the same run

Target outputs: The use of a target output rather than the mean output may be used in the calculations made prior to the application; however, a full output calibration must be conducted just prior to each use of a target output, and the mean output must be within 5% of the target output. Using a target output rather than a mean output increases the probability that an application rate deviation will occur. Verification of the amount of test substance that has been applied in calculations that use the discharge rate will always be made using the most recent calibration data.

Speed Calibrations:
Speed calibrations, if appropriate, must be performed prior to the first test substance application. For treatment 02, conduct speed calibration on the conveyer line.

Is this the first application of test substance in this trial?

YES: A full speed calibration is required.

NO: A single run recheck may be conducted to confirm consistent speed (±5% of the last complete speed calibration) just prior to subsequent applications.

Full speed calibration is required when:
1. A major equipment change has been made.
2. A complete output calibration is performed.

Speed recheck is required when:
1. Speed calibration data from another trial is used.

Speed recheck is not required when the same Field Research Director is making applications on the same day for multiple trials in this study, or multiple treatments in the same trial, unless there is a major equipment change or the treated lots are located on separate farms.

14.3 Actual Application Rate: Record actual application pass-times in the Field Data Book and verify the accuracy of the application against the protocol rate. **The application is considered acceptable if the accuracy is within -5% and +10% of the target rate specified in Section 15. If the application did not meet this range, the Study Director must be notified of this deviation before proceeding with this trial.**

The submitted Field Data Book shall contain the original calibration data or a true copy of all calibrations referenced, along with the original data from the rechecks performed for this trial.

15. APPLICATION TREATMENTS AND TIMING: All trials:

Prior to treatment, remove soil and plant debris from sweet potatoes using standard commercial practice.

Trt #	Treatment	Target Rate of active ingredient	Target Rate of formulated product*	Application Type	Spray Volume Range
01	Untreated	Not Applicable	Not Applicable	Not Applicable	Not Applicable
02	AZOXYSTROBIN	4.24 g ai/2000 lb (0.00935 lb ai/2000 lb) of roots	29.6 ml (1.0 fl oz)/2000 lb of roots	Conveyor line spray**	Sufficient water for complete coverage ***Suggestion: 0.5 gal/2000lbs***
	FLUDIOXONIL	4.24 g ai/2000 lb (0.00935 lb ai/2000 lb) of roots			
	DIFENOCONAZOLE	3.31 g ai/2000 lb (0.00730 lb ai/2000 lb) of roots			

*The nominal formulation concentration of the test substance will be used in calculating application rates (see Section 13 for the nominal concentration).
**To ensure proper coverage, roots should be tumbling as they are treated.

All trials: Make one postharvest application of the appropriate treatment type (to the respective lot) as described above and below:

Treatment 02 Conveyor Line Spray:
Make one application as an in-line aqueous spray application. Ensure proper coverage of roots by mixing the fungicide solution in an appropriate amount of water (suggest 0.5 gal/2000lbs) for complete coverage of the roots (1.0 fl oz / 2,000 lb of roots). Roots should be tumbling as they are treated. Use T-jet, CDA or similar application system and apply as a postharvest conveyor line spray. Ensure roots are dry before sampling. (for the conveyor line spray).

The conveyor line spray can be mimicked by laying out a known weight of sweet potatoes, in close proximity to each other, on a porous surface (wire mesh). Prepare one spray mix in sufficient water (suggest 0.5 gal/2000lbs) to cover all the roots (based on the weight of the sweet potatoes being treated) at the rate of 1.0 fl oz/2000 lb of roots. Spray approximately one-half of the mixture on the sweet potatoes in a uniform manner. Immediately turn the roots over and spray the remaining half of the spray mixture. (DO NOT ALLOW the spray to dry between treating each side). **Allow roots to dry prior to sampling.**

16. SUPPLEMENTAL CROP TREATMENTS:

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Only EPA-registered maintenance pesticides should be used; apply according to labeled directions. Make identical applications to the untreated and treated lots.

Consult with Study Director if no registered pesticides are available to control the pests. Document all supplemental crop treatments. DO NOT USE pesticides that are similar to the test substance or other chemicals that might interfere with analysis of the test substance. If unsure, **contact the Study Director**.

Note: Do not select sweet potatoes for the postharvest study that have been previously treated with azoxystrobin, fludioxonil or difenoconazole for seed treatment or foliar use.

17.1 RESIDUE SAMPLE COLLECTION:

All trials: Collect two samples from treatments 01 (Samples A & B) and 02 (Samples C & D). Each sample should be representative of the entire lot. On the day of the test substance application, collect treated samples after the fungicide has dried on the roots. Starting with the untreated lot, collect 12 sweet potato roots per sample. Each sample should be collected during a separate run through the entire lot.

The untreated samples may be collected prior to handling the test substance on the day of application.

Root samples should weigh a minimum of 4 lb (but preferably not more than 6 lb).

Wait for the roots to dry before cutting/subsampling. **If the sweet potatoes are large [greater than approximately 4 inches (10 cm) in diameter], then cut them with a clean knife into at least 4 slices and retain all of the slices for the sample.** Only if necessary to reduce the sample size, cut each root longitudinally into quarters with a clean knife on an uncontaminated surface. Reduce gross sample weight by retaining only opposite quarters from each root for the sample.

Follow proper handling practices with clean or gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for the untreated lot(s) before proceeding to the treated lot(s).

Place all samples in plastic-lined cloth bags. Bags may be obtained from the Field Research Coordinator (Section 23). Identify each sample bag** with correct Field ID number, Test Substance (chemical name listed in Section 15), complete sample ID (see Section 18) and harvest/sampling dates. After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

When using **IR-4 plastic lined cloth residue sample bags, complete attached sample tag as follows:
Field ID Number; Crop Fraction; Test Substance (enter the chemical name listed in Section 15); **Sample ID; Trt#; Harvest Date; Sample Date; Field Research Director** (enter name and telephone number).

17.2 RESIDUE SAMPLE COLLECTION 18-NC217 ONLY (samples intended for processing):

Collect one sample from each treatment (TRT01-Sample E) and (TRT02-Sample F) only. Collect approximately 120 lbs. of roots from each treatment. Each sample should be representative of the lot. Immediately after sample collection, ship unfrozen samples overnight in coolers to the processing laboratory. Samples should be processed within 72 hours of sample collection. See shipping instructions in Section 19.1

The untreated samples may be collected prior to handling the test substance on the day of application.

18. FIELD RESIDUE SAMPLE INVENTORY:

18.1 All trials except processing trial, 18-NC217:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 roots / 4 lb.	Roots
B	01	Untreated	NA	12 roots / 4 lb.	Roots
C	02	Azoxystrobin + Fludioxonil + Difenoconazole (A+F+D)	0	12 roots / 4 lb.	Roots
D	02	(A+F+D)	0	12 roots / 4 lb.	Roots

18.2 PROCESSING RESIDUE SAMPLE INVENTORY: Trial 18-NC217 only

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	12 roots / 4 lb.	Roots
B	01	Untreated	NA	12 roots / 4 lb.	Roots
C	02	(A+F+D)	0	12 roots / 4 lb.	Roots
D	02	(A+F+D)	0	12 roots / 4 lb.	Roots
E	01	Untreated	NA	120 lb.	Roots
F	02	(A+F+D)	0	120 lb.	Roots

19. RESIDUE SAMPLE HANDLING AND SHIPMENT (Samples not for processing):

Sample handling and storage methods can be outlined generally in SOP's, but describe methods fully in the Field Data Book.

For pre-shipment storage, the samples will be held frozen at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours' duration due to freezer cycling, sample movement, etc. If the analytical laboratory is close enough to the field site to permit delivery of the samples by field personnel on the day of sampling, then pre-shipment frozen storage is not required.

Freezer logs will be used to document all sample additions to and removals from storage. All on-site storage temperatures will be monitored and documented.

Contact the designated person (noted below) from the analytical laboratory prior to sample shipment for any specific shipping instructions. **Shipment** of frozen samples will be by freezer truck or express shipment, unless the samples are brought to the analytical laboratory by field trial personnel. Shipments sent via express shipment (overnight carriers such as Federal Express or Airborne) will require the addition of quantities of dry ice sufficient to maintain sample integrity while in transit to the laboratory. If field trial personnel transport the samples to the analytical laboratory directly from the lots and the sampling-to-freezer interval is more than one hour, an appropriate method of cooling and temperature-monitoring shall be used to maintain sample integrity. If the samples are stored frozen at the field trial facility prior to being transferred to the analytical laboratory by field trial personnel, then appropriate methods must be used to keep the samples frozen during transport. These methods should be documented in the FDB.

Document the notification made to the sample destination by use of e-mail, fax, telephone log, Field Data Book communication note, etc.

Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship sample bags. This documentation is needed even when field personnel transport the samples to the analytical laboratory.

For analysis, send samples to: Dr. Royal Fader, IR-4 North Central Research Center, Michigan State Univ., 3900 Collins Road, Lansing, MI 48910-8396, 517-336-4684, FAX# 517-432-2098, email: faderr@msu.edu

19.1 RESIDUE SAMPLE HANDLING AND SHIPMENT (samples for processing):

Contact the processing lab as soon as you know the date you expect to ship the large, fresh samples for processing, so that the lab will be ready to receive them and begin the processing part of the study as needed. If samples for processing are not shipped to the processing facility on the day of harvest, they should be stored in a refrigerator at approximately 4°C until they are shipped. A (document this communication in the field data book). **Insert a true copy of Field Data Book Part 8B and a blank copy of Field Data Book Part 8C (Sample Arrival Check Sheet) into each box or container used to ship samples.** Send samples for processing to: Joshua Bevan, Director, University of Idaho Food Technology Center, 1908 E. Chicago Street, Caldwell, ID 83605; (208) 795-5332; FAX# 208-795-5335; e-mail: jbevan@uidaho.edu

19.2 PROCESSING:

Immediately prior to processing sweet potato roots, remove representative "grab" samples of untreated (1sample) and treated roots (2 samples) from the larger samples (approximately 4-6 lbs. for each sample). Place the "grab" samples in frozen storage at temperatures generally less than -18 °C (0 °F), allowing for normal variations of less than 24 hours duration due to freezer cycling, sample movement, etc.

Using simulated commercial processing (provide detailed description of equipment and procedures), process the roots into baked/washed (peel on) sweet potato, sweet potato puree, sweet potato fries and sweet potato chips Process untreated roots first, followed by treated roots.

Place all samples into suitable clean containers and label them as indicated in the PROCESSED SAMPLE INVENTORY below. Processed samples should be frozen as soon as possible after processing is completed. For these samples, the processing lab should follow Protocol Section 19 procedures required for freezing/shipping samples to the analytical lab.

If processing cannot take place within 3 days of sample collection, then the samples should be stored in typical storage conditions to prevent test substance residue degradation.

Contact the designated person (noted below) from the analytical laboratory prior to shipment of samples for any specific shipping instructions. See shipping instructions for frozen samples in Section 19. Document the notification made to the sample destination by use of e-mail, fax, telephone log, field data book communication note, etc. For analysis of processed fractions, send samples to: Dr. Royal Fader, IR-4 North Central Research Center, Michigan State Univ., 3900 Collins Road, Lansing, MI 48910-8396, 517-336-4684, FAX# 517-432-2098, email: faderr@msu.edu

19.3 PROCESSED SAMPLE INVENTORY:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
G	01	Untreated	NA	12 roots / 4 lbs.	Roots
H	02	(A+F+D) (grab sample)	0	12 roots / 4 lbs.	Roots
I	02	(A+F+D) (grab sample)	0	12 roots / 4 lbs.	Roots
J	01	Untreated	NA	2-4lbs.	Raw washed roots
K	02	(A+F+D)	0	2-4lbs.	Raw washed roots
L	01	Untreated	NA	2-4lbs.	Raw washed, peeled roots
M	02	(A+F+D)	0	2-4lbs.	Raw washed, peeled roots
N	01	Untreated	NA	2-4lbs.	Wet peel from sample L
O	02	(A+F+D)	0	2-4lbs.	Wet peel from sample M
P	01	Untreated	NA	2-4lbs.	Baked washed root with peel
Q	02	(A+F+D)	0	2-4lbs.	Baked washed root with peel
R	01	Untreated	NA	2-4lbs.	Puree
S	02	(A+F+D)	0	2-4lbs.	Puree
T	01	Untreated	NA	2-4lbs.	Fries
U	02	(A+F+D)	0	2-4lbs.	Fries
V	01	Untreated	NA	2-4lbs.	Chips
W	02	(A+F+D)	0	2-4lbs.	Chips
X	01	Untreated	NA	2-4lbs.	Flakes
Y	02	(A+F+D)	0	2-4lbs.	Flakes

20. FIELD DOCUMENTATION AND RECORD KEEPING:

All operations, data and observations appropriate to this study should be **recorded directly and promptly into the IR-4 Field Data Book**.

The content of the Field Data Book should be **sufficiently detailed to completely reconstruct the field trial**. At a minimum, collect and maintain the following raw data:

20.01- Names of all personnel conducting specific research functions

20.02- Amendments and deviations from protocol and standard operating procedures (including copies of signed protocol changes received prior to submission of the Field Data Book to the Regional Field Coordinator).

20.03- Test site information

20.04- Test substance receipt, use and container/substance disposition records

20.05- Test substance storage conditions (including temperatures)

20.06- Data regarding calibration and use of application equipment

20.07- Treatment application data

20.08- Crop maintenance pesticides and cultural practices, test lot history, and soil information.

20.09 - If appropriate, a brief description of holding/storage conditions of the roots between harvest and treatment.

20.10- Residue sample identification, collection, storage conditions and handling (Weight measurements are considered estimates for the samples collected from field or processing trials, and the scales/balances used for this purpose do not need to be maintained in strict adherence to GLP.)

20.11- Residue sample shipping information

20.12- Description of crop destruction, or explanation for lack of destruction

20.13- Daily Meteorological/Irrigation records--required from planting of annual crops to harvest. These records do not need to be determined under GLP standards.

20.14- Pass times (if applicable) and other data to confirm amount of material applied to lots

20.15- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance

20.16- Other applicable data requested in the IR-4 Field Data Book necessary for confirmation that the study was conducted in accordance with the protocol.

Compliance with GLP's is not required for the collection of data associated with crop phytotoxicity.

20.1 PROCESSING DOCUMENTATION AND RECORD KEEPING:

At a minimum, collect and maintain the following raw data:

20.1.01- Names of all personnel conducting specific research functions

20.1.02- Deviations from protocol and standard operating procedures

20.1.03- Date sweet potato samples received

20.1.04- Storage temperatures until sweet potato samples are processed into raw washed sweet potato, raw washed peeled sweet potato, wet peel, baked washed (peel on) sweet potato, sweet potato puree, sweet potato fries, sweet potato chips, and sweet potato flakes.

20.1.05- Processing Methodology (SOPs are acceptable)

20.1.06- Data collected and observations made during processing of samples into raw washed sweet potato, raw washed peeled sweet potato, wet peel, baked washed (peel on) sweet potato, sweet potato puree, sweet potato fries, sweet potato chips, and sweet potato flakes.

20.1.07- Storage temperatures of raw washed sweet potato, raw washed peeled sweet potato, wet peel, baked washed (peel on) sweet potato, sweet potato puree, sweet potato fries, sweet potato chips, and sweet potato flakes until shipped.

20.1.08- Date raw washed sweet potato, raw washed peeled sweet potato, wet peel, baked washed (peel on) sweet potato, sweet potato puree, sweet potato fries, sweet potato chips, and sweet potato flakes are shipped to analytical laboratory

A processing summary report should be prepared and submitted to the sponsor representative. When the processing summary report is completed the report and all original raw data will be sent to IR-4 Headquarters in Princeton, NJ (when an original document cannot be provided a "true copy" will be provided). All original raw data

shall be secured in the archives of IR-4 Headquarters, Princeton, NJ. A "true copy" of the raw data and the final processing report shall be secured in the archives of the Processing Research Director/Testing Facility.

21. PROTOCOL/SOP MODIFICATIONS - FIELD RESEARCH:

Consult with the Study Director and with the Regional/ARS Field Research Coordinator to discuss desired changes in the protocol prior to occurrence. If appropriate, an amendment will be issued.

Any deviations from the protocol will require the Field Research Director to complete a written report outlining the changes. **Provide this report to the Study Director promptly** (e.g. within 14 days of occurrence or recognition) for review and signature.

All deviations from the approved SOP's also require documentation and **approval by the Study Director**.

22. FIELD RESEARCH REPORT/ARCHIVING:

The Field Research Director will forward the completed originals of the IR-4 Field Data Book and other raw data to the Regional/ARS Field Research Coordinator as soon as possible after the shipment of residue samples.

The Field Research Director will maintain a complete certified true copy of these field documents.

The original IR-4 Field Data Book and other raw data will be forwarded to IR-4 Headquarters for reporting and archiving.

23. FIELD PERSONNEL / ID NO. / REGIONAL/ARS FIELD RESEARCH LOCATION

If a Field Research Director is assigned more than one trial in this study, refer to Section 11.4 for requirements to differentiate the trials.

Field Research Director	Field ID NO.	RFC	Test Crop
Mr. Roger B. Batts, MAILING: NCSU Campus Box 7654, Raleigh, NC 27695-7654, (919) 515-1668, Fax# (919) 513-7226; e-mail: rbbatts@ncsu.edu. PACKAGES: Room 110, 520 Brickhaven Drive, Raleigh, NC 27606, (919) 515-1668, FAX# 919-513-7226; e-mail: rbbatts@ncsu.edu. TEST SUBSTANCE SHIPPING: Roger B. Batts, NC State Univ, Central Receiving/NCSU, 3240 Ligon Street, Raleigh, NC 27695, (919) 515-1668, FAX# 919-513-7226; e-mail: rbbatts@ncsu.edu	12118.18-NC217 (processing)	SOR	Sweet Potato (Post Harvest)
Cristina M. Marconi, Texas A&M AgriLife Research & Extension Center at Weslaco, 2415 East Highway 83, Weslaco, TX 78596; (956) 969-5655; e-mail: cristina.marconi@agnet.tamu.edu	12118.18-TX342	SOR	Sweet Potato (Post Harvest)
Keri Skiles, Kearney Agricultural Research & Ext. Center (KARE), 9240 S. Riverbend Ave., Parlier, CA 93648, (559) 646-6061, FAX# 559-646-6015; Cell: 559-310-4092; e-mail: kmskiles@ucanr.edu	12118.18-CA54	WSR	Sweet Potato (Post Harvest)
Keri Skiles, Kearney Agricultural Research & Ext. Center (KARE), 9240 S. Riverbend Ave., Parlier, CA 93648, (559) 646-6061, FAX# 559-646-6015; Cell: 559-310-4092; e-mail: kmskiles@ucanr.edu	12118.18-CA55	WSR	Sweet Potato (Post Harvest)

RFC = Regional/ARS Field Coordinator

Location:

ARS: Dr. Alvin Simmons, USDA-ARS, US Vegetable Laboratory, 2700 Savannah Highway, Charleston, SC 29414; Tel: (843) 402-5307, FAX# 843-573-4715; e-mail: Alvin.simmons@ars.usda.gov

NCR: Dr. John C Wise, IR-4 North Central Research Center, Michigan State Univ., 3815 Technology Blvd., Suite 1031B, Lansing, MI 48910-8396, (269) 330-2403, FAX# 517-432-2098; e-mail: wisejohn@msu.edu

NER: Ms. Marylee Ross, University of Maryland, LESREC, 27664 Nanticoke Rd., Salisbury, MD 21801; Tel: (410) 742-8788 x 310, FAX# 410-742-1922; e-mail: mross@umd.edu

SOR: Mr. Roger B. Batts, MAILING: NCSU Campus Box 7654, Raleigh, NC 27695-7654, (919) 515-1668, Fax# (919) 513-7226; e-mail: rbbatts@ncsu.edu
FDBs to be shipped to SOR for QC & processing go to:
ATTN: Southern Region IR-4, University of Florida, PO Box 110720, SW 23rd Drive, Bldg, 685, Gainesville, FL 32611, (352) 294-3983 or (352) 392-1978; Fax: 352-392-1988

WSR: Dr. Michael Horak, Regional Field Coordinator, Western Region IR-4 Project, 4218 Meyer Hall, University of California-Davis, Davis, CA 95616 (530) 752-7634; Cell# 530-219-8466; e-mail: mjhorak@ucdavis.edu

Canada: Ms. Shirley Archambault, Agriculture & Agri-Food Canada, Pest Management Centre, Building 57, 960 Carling Avenue, Ottawa, ON Canada K1A 0C6; Tel: (613) 759-7714; FAX# 613-694-2323; e-mail: archambaultsh@agr.gc.ca

23.1 PROCESSING PERSONNEL/ID NO.: **PROCESSING ID NO.:** **12118.18-IDP02**

PROCESSING RESEARCH DIRECTOR/PROCESSING FACILITY:
Joshua Bevan, Director, University of Idaho Food Technology Center, 1908 E. Chicago Street, Caldwell, ID 83605; (208) 795-5332; FAX# 208-795-5335; e-mail: jbevan@uidaho.edu

24. LABORATORY PERSONNEL/ID NO.: **LAB ID NO.:** **12118.18-MIR04**

LABORATORY RESEARCH DIRECTOR/TESTING LABORATORY:
Dr. Susan Erhardt, IR-4 North Central Research Center, Michigan State Univ., 3815 Technology Blvd., Suite 1031B, Lansing, MI 48910-8396 , (517) 336-4653 FAX# 517-432-2098; e-mail: serhardt@msu.edu

25. LABORATORY SAMPLE INVENTORY:

Treated and untreated samples of sweet potato will be received from each of the field and processing sites in Section 23.

Notify appropriate Field Research Director and Regional/ARS Field Research Coordinator of sample receipt.

26. LABORATORY SAMPLE IDENTIFICATION:

Each sample (raw commodity, crop fractions, storage stability, method validation, etc.) is to be assigned a unique laboratory sample number by the laboratory personnel.

A cross-reference must be maintained between the assigned laboratory sample number and the identification utilized in the Residue Sample Shipping and Identification Sheet.

27. LABORATORY SAMPLE STORAGE/PREPARATION:

Store samples in a limited access area at temperatures (generally less than -20°C) that will maintain frozen sample integrity, until extraction.

The samples may be stored whole or ground, depending on the standard procedure of the analytical laboratory. However, if maceration will cause residue deterioration, then samples must be stored whole until analysis.

Do not composite samples.

The entire sample provided from the field must be ground, if sample is too large to be manageable then contact the Study Director for appropriate subsampling to assure the representative nature of the sample obtained in the field is maintained by the laboratory procedure.

Generally, sample extracts should be stored at ≤ 4°C for no longer than 14 days before analysis.

Storage stability of extracts must be demonstrated if extracts are not analyzed on the same day as they are obtained.

Concurrent fortifications may be used to show extract storage stability, as long as the extracts from the concurrent fortifications have been stored at least as long as the extracts obtained from the weathered samples.

Contact the Study Director if samples extracts are stored greater than 14 days prior to analysis.

All storage temperatures, conditions and location of sample storage are to be monitored and documented.

28. LABORATORY REFERENCE SUBSTANCE:

Obtain the laboratory reference substance(s), difenoconazole, 1,2,4-triazole, triazole alanine (TA), triazole acetic acid (TAA), azoxystrobin, R230310, and fludioxonil from the Registrant. Contact Dr. Dirk Drost, (336) 632-7510, FAX# 336-632-6021, e-mail: dirk.drost@syngenta.com to procure the proper material.

Document the date the analytical standards are received, the source, stated purity, storage conditions, and expiration date.

Use only reference standards that have been characterized to meet GLP standards.

Archival and characterization of the reference substance (purity, identity, stability and solubility) is the responsibility of the registrant.

29. ANALYTICAL METHODOLOGY:

REFERENCE METHOD:

For Difenoconazole:

1. Residue Method for the Determination of Residues of Difenoconazole (CGA169374) in Various Crops and Processed Crop Fractions. Final Determination by LC-MS/MS, Syngenta, Nov. 11, 2004, REM 147.08.

   AND

2. Determination of 1,2,4-Triazole, Triazole Alanine and Triazole Acetic Acid Residues in Plant and Animal Matrices," Analytical Method No. Meth-160. Rev #2, Morse Laboratories, LLC, April 13, 2005.

For Azoxystrobin:

3. "Residue Analytical Method for the Determination of Residues of Azoxystrobin (ICI5504) and R230310 in Crop Samples. Final Determination by LC-MS/MS"; Syngenta No.: RAM305/03; Dated 25 November, 2004; Authors, Chaggar, S., Crook, S.J., Harron, E.A. and Robinson, N.J.

For Fludioxonil:

4 "Analytical Method for the Determination of Residues of Fludioxonil (CGA173506) in Crop Matrices, Final Determination by LC-MS/MS" (REM 133.06) C.H.Nichols, June 30, 2006.

REFERENCE METHOD MODIFICATIONS/METHOD VALIDATION

The above listed Reference Method(s) may be modified if needed for the test matrix.

The Reference Method, along with any modifications must be validated on the following crop fractions: whole raw sweet potato (will cover many of the processed commodities); chips or fries (cooked commodity) and flakes (dried commodity) prior to residue sample analysis of that crop fraction.

For Difenoconazole (Method 1):
To validate the method, fortify some of the control samples in triplicate with Difenoconazole at a minimum of 3 concentration levels, lowest level of method validation (0.01 ppm or lower), 1.0 ppm, and 10 ppm.
For 1,2,4-Triazole, (T), Triazole Alanine (TA) and Triazole Acetic Acid (TAA) (Method 2)
To validate the methods, fortify some of the control samples in triplicate with T, TA and TAA at a minimum of 3 concentration levels each, lowest level of method validation (0.01 ppm or lower) and 0.1 ppm.
For Azoxystrobin (Method 3):
To validate the method, fortify some of the control samples in triplicate with Azoxystrobin and the z-isomer R230310 at a minimum of 3 concentration levels lowest level of method validation (0.01 ppm or lower), 1.0 ppm and 10 ppm.
For Fludioxonil (Method 4):
To validate the method, fortify some of the control samples in triplicate with fluidoxonil at a minimum of 3 concentration levels lowest level of method validation (0.01 ppm or lower), 1.0 ppm and 10 ppm.

A minimum of 6 fortification samples (recovery spikes) at the lowest level of method validation (LLMV) is required for each analyte on each fraction prior to completion of the analytical phase of the study. **The acceptable recovery range is 70-120%.**

Documented approval from the Study Director is needed for recoveries outside of this range.

Document the exact procedures for sample analysis.

This validated step-by-step Working Method should incorporate all changes from the Reference Method.

Provide the Study Director with a copy of this Working Method and results of method validation prior to treated sample analysis.

If the Working Method has been used successfully on the test matrix or a similar matrix, the Study Director may waive the requirement for method validation. **Contact the Study Director for details**.

SAMPLE ANALYSIS:

Samples will be analyzed separately for the residues of Difenoconazole, the triazole metabolites T, TA & TAA and residues of Azoxystrobin and the z-isomer R230310 and residues of fludioxonil following the Working Methods.

For each field trial associated with this study, analyze at least one untreated and all treated residue samples for each matrix.

Contact the Study Director if residues above the lowest level of method validation for each matrix are detected in the untreated samples.

Any changes or modifications to the Working Method **require Study Director approval**. Whenever possible, **notify the Study Director** prior to occurrence.

Any change or modification to the Working Method must be documented in the raw data and discussed in the final report.

A typical analytical set (or run) should consist of calibration standards, untreated sample(s), concurrent recovery sample(s), and treated sample(s). Each analytical set must begin and end with a calibration standard. Additional calibration standards should be injected with sample analysis to ensure goodness of fit to the standard curve.

Over the course of method validation, residue sample and storage stability (if appropriate) analysis, adequate fortification samples that bracket the actual residues should be analyzed. At least one concurrent fortification sample should be analyzed per analytical set.

The Study Director should be immediately notified if concurrent recoveries deviate from the acceptable recovery range of 70% to 120%.

All efforts will be made to resolve existing recovery problems before continuing forward with additional analytical sets.

If residues in samples are above the highest Working Method validation concentration, additional recovery samples at levels above actual residues must be run in triplicate (3 uniquely extracted samples) as soon as practical. A minimum of 6 fortification samples (recovery spikes) at the lowest level of method validation (LLMV) is required for each analyte on each fraction prior to completion of the analytical phase of the study.

Treated samples may be analyzed using a screening run prior to analysis of treated samples using the working method, if the procedure is covered in the laboratory SOPs and the working method for the study. The peak areas of the treated samples and highest standard from any screening run will not be quantified or reported. (Any data, such as chromatograms, generated during screening run(s) will be kept.)

STORAGE STABILITY ANALYSIS:

As soon as possible after receipt of samples, a minimum of six subsamples of all available crop fractions of the control shall be fortified with azoxystrobin, fludioxonil and difenoconazole at 0.2 ppm.

Sufficient storage stability data covering the storage intervals of samples for azoxystrobin, fludioxonil and difenoconazole have been reported by the registrant (**24 months** for all **three** active ingredients). Storage stability of

azoxystrobin, fludioxonil and difenoconazole will be run only if sample storage exceeds the aforementioned storage years reported by the registrant.

Contact the Study Director to determine if Storage Stability Samples need to be analyzed.

Only if directed by the Study Director, three samples of each analyte and crop fraction will be analyzed after the appropriate storage period. The analysis of storage stability samples may be conducted following a storage period equal to or greater than 90% of the longest storage period of the field –treated samples from collection in the field/processing facility until their analysis. The remaining samples will be retained for long-term storage.

If analysis of treated/control samples is completed within 30 days of harvest analysis of storage fortification samples may not be required. **If appropriate, contact Study Director.**

STATISTICAL METHOD(S):

Utilize regression analysis to determine the linearity of the standard curve (r^2) or the goodness of fit if the standard curve is non-linear.

Criteria for acceptance of the standard curve(s) or other statistical methods shall be determined by Laboratory Research Director and documented in the raw data.

30. DISPOSITION OF SAMPLES:

A minimum of 100 g or all (if less than 100 g) of each of the remaining frozen treated and untreated crop samples is to be retained for at least 12 months after submission of the laboratory report.

Long term fortified storage study samples shall be retained for a period of 1 to 5 years, as appropriate, after submission of the final report.

Sample extracts can be disposed of after data analysis.

The Study Director is to be contacted prior to discarding samples.

31. LABORATORY PROTOCOL/SOP MODIFICATIONS - LABORATORY RESEARCH:

Consult with the Study Director regarding desired changes in the protocol prior to occurrence. If appropriate, an amendment will be issued. Any unauthorized changes to the protocol will require the Laboratory Research Director to complete a written report outlining the changes.

This report should be **provided to the Study Director promptly** (e.g. within 14 days of occurrence) for review and signature.

All deviations from the approved SOP's also require documentation and approval by the Study Director.

32. LABORATORY DOCUMENTATION AND RECORD KEEPING:

All operations, data and observations shall be recorded in the analyst's notebook and log books, which must be signed and dated on date of entry.

At a minimum, collect and maintain the following raw data:

32.01 - Analytical standard(s) receipt, use and disposition records

32.02 - Analytical standard(s) storage conditions

32.03 - Analytical standard(s) dilution calculations and preparation records

32.04 - Sample storage conditions and locations

32.05 - Calculation work sheets

32.06 - All chromatograms, including those that are not reported

32.07 - Chain of custody records

32.08 - Deviations from protocol, Working Method and/or standard operating procedures

32.09 - Name of personnel conducting specific research functions

32.10 - Sample analysis worksheets

32.11- Storage stability fortification records

32.12 - Concurrent recovery fortification records

A study file shall be developed and maintained by the Laboratory Research Director in conjunction with the analysis. It will contain a copy of the protocol, all pertinent raw data, documentation, records, correspondence, and the final analytical summary report. In addition, records of equipment maintenance and calibrations will be kept and periodically archived.

33. LABORATORY RESEARCH REPORT:

The analytical summary report sent to IR-4 HQ shall contain, but not be limited to:

33.01 - Applicable method validation data

33.02 - Applicable storage stability data

33.03 - Residue levels for control and treated samples with concurrent fortified recoveries

33.04 - Complete copy of the analytical Working Method

33.05 - Any modifications or deviations from the protocol and/or Working Method

33.06 - Completed IR-4 residue data reporting form or appropriate reporting form which includes information listed on the IR-4 generic residue data reporting form

33.07 - A minimum of 10 representative chromatograms of treated samples (if fewer than 10 submit all), a minimum of three chromatograms each of control and fortified control samples, chromatograms (one of each concentration) for at least one set of calibration standards for each compound analyzed, and any chromatograms of samples with unusual or inconsistent results

33.08 - Summary of quantitative data associated with samples and spike recovery samples should be provided (e.g. peak heights, injection volumes, sample sizes, final volumes, etc.)

33.09 - Clearly presented example calculations or statistical evaluations

33.10 - Discussion of results (including purpose of method modifications, sample storage conditions, etc.)

33.11 - Summary data associated with calibration standards (dilution and use records, calibration curves, etc.)

34. LABORATORY ARCHIVES:

For studies assigned to the IR-4 Leader Laboratory at the University of California (CAR), University of Florida (FLR), or Michigan State University (MIR): When the final analytical summary report is completed and sent to the sponsor representative, all original raw data including a "true copy" of the final analytical summary report shall be secured in the archives of the Laboratory Research Director/Testing Facility.

For studies assigned to any other analytical laboratory: When the final analytical summary report is completed the analytical report and all original raw data will be sent to IR-4 Project Headquarters, 500 College Road East, Suite 201 W, Princeton, NJ 08540, (732) 932-9575, FAX# (609) 514-2612 (when an original document cannot be provided a "true copy" will be provided). All original raw data shall be secured in the archives of IR-4 Headquarters, Princeton, NJ. A "true copy" of the raw data and the final analytical report shall be secured in the archives of the Laboratory Research Director/Testing Facility.

IR-4
FIELD DATA BOOK

TITLE: AZOXYSTROBIN + FLUDIOXONIL + DIFENOCONAZOLE MAGNITUDE OF THE RESIDUE ON SWEET POTATO (POST HARVEST)

PR# 12118

SPONSOR

IR-4 Project Headquarters
500 College Road East, Suite 201 W
Princeton, NJ 08540
(732) 932-9575, FAX# (609) 514-2612

STUDY DIRECTOR
Grace Lennon
Phone extension - 4627
Email galennon@njaes.rutgers.edu