1. PROJECT TITLE: FAMOXADONE + CYMOXANIL: Magnitude of the Residue on RADISH

2. JUSTIFICATION AND OBJECTIVES:

IR-4 has received a request for the minor use of famoxadone + cymoxanil on radish for control of foliar diseases including *Alternaria*, downy mildew, white rust and *Cercospora*.

To establish this tolerance, 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.

To determine the magnitude of residues of total famoxadone + cymoxanil in or on radish, 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**).

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]:

Kathryn Homa, IR-4 Project Headquarters, 500 College Road East, Suite 201 W, Princeton, NJ 08540, (732) 932-9575 extension 4604, FAX# (609) 514-2612, E-mail: homa@aesop.rutgers.edu

5. PROPOSED DATES:

Experimental Start :	02/14
Experimental Termination:	11/15
Study Completion:	10/16

6. PROPOSED TEST SITES:

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

7. STUDY AUTHORIZATION:

Deborah Carpenter Feb 20, 2014	Kathy Homa 2/20/14
Sponsor Representative / Date	Kathryn Homa / Study Director / Date

7.1 STUDY DIRECTOR INITIALS: KH

[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:

RADISH - 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 and **one** treated plot.

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

Field trial 08757.14-FL157 will provide samples for a decline trial (multiple sampling dates). The plots must be large enough to provide enough samples on each sampling date to meet sample size requirements.

11.2 Employ adequate buffer zones between each of the plots to prevent contamination. For most application types, a minimum distance of 15 feet is required, but a minimum of 50 feet is strongly preferred. For applications made by airblast, mist blower, or power sprayers, a minimum distance of 50 feet is required, but a minimum of 100 feet is strongly preferred. When plants are used as a buffer between the untreated and treated plots, a lower distance is needed to prevent contamination, but the minimums indicated above must be observed. If another study using a test substance with the same active ingredient is being conducted at the same research site, the untreated plot from one study must be separated from the treated plot(s) of the other by the appropriate buffer zone indicated above.

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 If a Field Research Director is assigned more than one trial in this study, the following requirements must be met:
An independently prepared tank-mix must be used in each trial.
Also, choose at least one option from Set 1 **or** at least two options from Set 2:

Set	Option	Description
1	A	Trial sites must be separated by at least 20 miles (32 km)
	B	First application or planting date (for annual crops) in each trial is separated by at least 30 days
	C	Different crop variety (different size or shape at maturity, rough vs. smooth surface, different amount of foliage shielding the commodity, different rate of growth, or representative of the major varieties grown within the region)—confirm with Study Director if this option will be chosen
2	A	Spray volume must vary by at least 25% of the lower volume (minimum 10 GPA difference) Example 1, Trial A has a volume of 20 GPA and Trial B has a volume ≥ 30 GPA Example 2, Trial A has a volume of 60 GPA and Trial B has a volume ≥ 75 GPA The trial with the lowest spray volume for the first application must remain the lowest for each application; the trial with the highest must remain the highest for each, and so on
	B	Use of an adjuvant (of any suitable type) in the tank mix for one trial vs. no adjuvant in the tank mix for another trial
	C	Different foliar application type: foliar directed or foliar broadcast (Do not use this option if the label instructions for this commodity will specify one type or the other)
	D	Not Applicable
	E	Different types of application equipment be used in each trial (for example, tractor-pulled boom sprayer, tractor-pulled spreader, airblast sprayer, axial fan orchard sprayer, proptec sprayer, cannon mist sprayer, tower sprayer, over-row sprayer, tunnel sprayer, backpack sprayer, waist pack sprayer, hand gun, hand-held spreader, or shaker can)
	F	Different spray droplet size (fine, medium, coarse, very coarse, or extra coarse) This may be accomplished by changing nozzles and/or by changing spray pressure Document in the Field Data Book the droplet size that results from the pressure and nozzles used in the trial (nozzle catalog may be used as a reference) Coarse, very course, and extra course are appropriate for herbicides only
	G	Not Applicable
	H	Not Applicable
	I	Different irrigation type (drip or furrow or sprinkler/over-the-top) (Irrigation must be applied at least once after each application, but over-the-top irrigation must not be applied within one hour of an application, and irrigation is not needed following the last application if samples are to be collected on the same day)
	J	Not Applicable
	K	Different planting arrangement for annual crops: single row beds or multi-row beds (two or more rows on each bed)
	L	Not Applicable
	M	Not Applicable
	N	Not Applicable
	O	Not Applicable
	P	Not Applicable

If these criteria cannot be met to separate multiple trials, the Field Research Director should contact the Study Director to discuss possible alternatives that can be amended to the protocol. Trials conducted in different calendar years are exempt from these requirements.

11.5 Mark plots 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:

Prepare or select a test site that has been maintained following good local agricultural practices for the production of radish including fertilization, irrigation, if necessary and available, and other practices that ensure commercially acceptable crop production.

The test site will have a known pesticide and crop treatment history of a minimum of 1 year and preferably 3 years.

13. TEST/CONTROL SUBSTANCE:

Use the Tanos 50DF formulation of 25% famoxadone + 25% cymoxanil (EPA Reg No. 352-604, CAS# Famoxadone: 131807-57-3; Cymoxanil 57966-95-7) 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

Alternatively, some registrants will archive the test substance containers. If test substance containers are shipped to another location, the shipment must be conducted in accordance with local, state, and Federal regulations. See shipping documents for directions for return of the test substance; if none are given, contact the registrant representative: Sheldon Sumpter, DuPont Agricultural Products, Phone: 302-451-3340, Fax: 302-451-3570; e-mail: sheldon.r.sumpter@dupont.com.

The registrant will archive a retention sample of the test substance.

Control substances are not relevant to this study.

14. TEST SUBSTANCE APPLICATION:

14.1 Simulate commercial application practices by applying the test substance in a manner that represents a major 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 and result in adequate canopy penetration and coverage.
- 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.

For foliar directed applications (generally used for insecticides and fungicides), do not proportionally reduce the application rate (the amount of active ingredient applied per acre). Direct the entire per-acre rate onto the crop. If row widths in the research plots are greater than local commercial practices, then the application rate should be calculated using a local commercial row width. **Contact the Study Director** if guidance is needed.

14.2 Full Calibrations for output and speed must be performed to ensure accurate delivery.
A calibration consists of a minimum of three consecutive, documented checks for nozzle or hopper output and speed (equipment or walking speed). (When the output of an airblast sprayer is calibrated or rechecked, it is not necessary to record the outputs of individual nozzles.) The variation of the total output recorded for any one of the three checks must not be greater than 5% from the mean for the full calibration.

Discharge/Output Calibrations must be performed:
Just prior to the first application of test substance, completely calibrate[2].

Another complete calibration must be performed and documented when application parameters or equipment components have changed between applications. Recalibration is required after any of the following have changed: application type; intended nozzle or hopper output; nozzle size or type, or other equipment that may affect the output etc. A recalibration is required even if the pressure (intended nozzle output) has been changed back to the pressure used at the initial calibration. It is not necessary to fully recalibrate when CO_2 tanks are changed, or when equipment is transported offsite or cleaned, or if nozzles are removed and then placed back on (even if other nozzles have been used in the interim, unless the pressure has also been changed); however a recheck must take place prior to the next application. If the recheck is out of specification (see paragraphs below) a recalibration is required. Use equipment logs to document changes in the equipment parameters.

Rechecking the output, at a minimum, is necessary for multiple applications, as long as parameters have not changed. A single output check may be conducted to confirm consistent delivery (±5% of the last complete calibration) just prior to subsequent applications. **A recheck is also required if the equipment has been moved from the location where the most recent full calibration or recheck has occurred, even if no applications have been made in the interim. (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".)**

The equipment must be completely recalibrated if:
- a recheck results in an output that differs from the mean of the complete calibration by greater than 5%
- the variation of any nozzle's output from the mean output of all of the nozzles during the same run is greater than 5% (this statement does not apply to airblast sprayers)

To minimize the occurrence of application rates that fall outside the protocol range, calculations for the amount of test substance to be applied that are based on the discharge rate should be performed using mean

[2]"Just prior" includes the day prior to the application, but calibration on the day of use is preferred.

output calculated from the most recent complete calibration data (mean of three output checks), not on single-output recheck results. The use of a target output rather than the mean output may be used in the calculations made prior to the application; however a complete 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 complete calibration data.

A speed calibration must also be performed prior to the first test substance application. Conduct the speed calibration in an area adjacent to the test plot, or on similar terrain. **Speed rechecks are required for multiple applications on different days.** Speed should be recalibrated if a major equipment change has been made, such as from a tractor-pulled sprayer to a backpack sprayer. (When a handgun is used to spray tree fruits or nuts, and each tree is sprayed for a predetermined time from a stationary position, a speed calibration is not required.)

Complete calibration data from another trial (performed on the day of or day prior to the application in this trial) may be used. However, a recheck (single output check **and speed recheck**) must be performed just prior to the application in this trial, but subsequent to any other applications with the application equipment. If more than one field trial in this study has been assigned to the same Field Research Director, it is not necessary to perform separate output rechecks for applications made on the same day in the respective trials, and separate speed rechecks are not required unless the treated plots 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 complete calibrations referenced, along with the original data from the rechecks performed for this trial.

15. APPLICATION TREATMENTS AND TIMING:

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	FAMOXADONE + CYMOXANIL	0.156 lb ai/acre famoxadone + 0.156 lb ai/acre cymoxanil	283.5 grams product/acre +NIS***	Foliar	20-60 GPA

*The nominal formulation concentration of the test substance will be used in calculating application rates (see Section 13 for the nominal concentration).
**GPA=gallons per acre
***All applications shall include a non-ionic surfactant at 0.25% v/v unless the absence of an adjuvant has been chosen to differentiate two trials conducted by the same Field Research Director (see Part 11.4). Include a copy of the label in the Field Data Book.

Make 5 applications at 5 (±1) day intervals with the last application 1 day before harvest. In decline trial 08757.14-FL157, there will be multiple sampling dates, and the last application will be on the day of the first harvest.

If it appears that phytotoxicity has resulted from applications made in this trial, contact the Study Director. If possible, take one or more photographs and send them to the Study Director via email to facilitate the evaluation of crop/ test substance effects.

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 plots.

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**.

17. RESIDUE SAMPLE COLLECTION:

Collect two samples from each plot. Each sample should be representative of the entire plot (except plot ends). Roots and tops may come from the same plants, or from different plants.

All trials except decline trial 08757.14-FL157: At 1 day after the last application, starting with the untreated plot, collect at least 24 marketable plants (tops and roots) from approximately 12 separate areas of each plot. Each sample should be collected during a separate run through the entire plot. Avoid sampling from plot ends.

For decline trial 08757.14-FL157 Only (see sample inventory in Protocol Section 18): Follow the sample collection directions noted above, except that treated samples should be collected at 0, 1, 3 (±1), 7(±1) and 10 (±1) days after the last application.

In trials in which treated samples shall be collected on the day of the last application (0-day PHI), the untreated samples may be collected prior to handling the test substance that day to make the last application.

For all trials: Root samples should weigh a minimum of 4 lbs (but preferably not more than 6 lbs) and each top sample should weigh a minimum of 1 lb (but preferably not more than 2 lbs). Avoid sampling from plot ends.

If excessive soil adheres to the roots, remove it by lightly brushing it off (document what is used to remove the soil or debris, e.g. a clean brush, clean gloved hand, clean dry towel, or similar method). If necessary, lightly rinse off with a minimal amount of clean water (do not scrub), or dip the root briefly in a bucket of water. Pat lightly while drying with clean paper towels. DO NOT RUB WHILE RINSING OR DRYING THE TOPS OR ROOTS.

Remove tops and package separately from the roots. Roots should be cut into halves or quarters, unless they are very small. Retain all portions of the cut roots 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 plot(s) before proceeding to the treated plot(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 (common chemical name and formulation), complete sample ID (see Section 18) and harvest/sampling dates. See Section 19 for residue sample handling directions.

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).

18. FIELD RESIDUE SAMPLE INVENTORY:

18.1 FIELD RESIDUE SAMPLE INVENTORY: For all trials except decline trial 08757.14-FL157

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 plants / 1 lb.	Tops
B	01	Untreated	NA	24 plants / 1 lb.	Tops
C	02	FAMOXADONE + CYMOXANIL	1	24 plants / 1 lb.	Tops
D	02	FAMOXADONE + CYMOXANIL	1	24 plants / 1 lb.	Tops
E	01	Untreated	NA	24 roots / 4 lbs.	Roots
F	01	Untreated	NA	24 roots / 4 lbs.	Roots
G	02	FAMOXADONE + CYMOXANIL	1	24 roots / 4 lbs.	Roots
H	02	FAMOXADONE + CYMOXANIL	1	24 roots / 4 lbs.	Roots

18.2 FIELD RESIDUE SAMPLE INVENTORY: For decline trial 08757.14-FL157 ONLY:

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM SAMPLE SIZE	CROP FRACTION
A	01	Untreated	NA	24 plants / 1 lb.	Tops
B	01	Untreated	NA	24 plants / 1 lb.	Tops
I*	02	FAMOXADONE + CYMOXANIL	0	24 plants / 1 lb.	Tops
J*	02	FAMOXADONE + CYMOXANIL	0	24 plants / 1 lb.	Tops
C	02	FAMOXADONE + CYMOXANIL	1	24 plants / 1 lb.	Tops
D	02	FAMOXADONE + CYMOXANIL	1	24 plants / 1 lb.	Tops
K	02	FAMOXADONE + CYMOXANIL	3 (±1)	24 plants / 1 lb.	Tops
L	02	FAMOXADONE + CYMOXANIL	3 (±1)	24 plants / 1 lb.	Tops
M	02	FAMOXADONE + CYMOXANIL	7 (±1)	24 plants / 1 lb.	Tops
N	02	FAMOXADONE + CYMOXANIL	7 (±1)	24 plants / 1 lb.	Tops
O	02	FAMOXADONE + CYMOXANIL	10 (±1)	24 plants / 1 lb.	Tops
P	02	FAMOXADONE + CYMOXANIL	10 (±1)	24 plants / 1 lb.	Tops
E	01	Untreated	NA	24 roots / 4 lbs.	Roots
F	01	Untreated	NA	24 roots / 4 lbs.	Roots
Q*	02	FAMOXADONE + CYMOXANIL	0	24 roots / 4 lbs.	Roots
R*	02	FAMOXADONE + CYMOXANIL	0	24 roots / 4 lbs.	Roots
G	02	FAMOXADONE + CYMOXANIL	1	24 roots / 4 lbs.	Roots
H	02	FAMOXADONE + CYMOXANIL	1	24 roots / 4 lbs.	Roots
S	02	FAMOXADONE + CYMOXANIL	3 (±1)	24 roots / 4 lbs.	Roots
T	02	FAMOXADONE + CYMOXANIL	3 (±1)	24 roots / 4 lbs.	Roots
U	02	FAMOXADONE + CYMOXANIL	7 (±1)	24 roots / 4 lbs.	Roots
V	02	FAMOXADONE + CYMOXANIL	7 (±1)	24 roots / 4 lbs.	Roots
W	02	FAMOXADONE + CYMOXANIL	10 (±1)	24 roots / 4 lbs.	Roots
X	02	FAMOXADONE + CYMOXANIL	10 (±1)	24 roots / 4 lbs.	Roots

***Note: Samples I and J are collected before samples C and D and Q and R are collected before samples G and H. Sample ID's are listed this way to keep samples C, D, G and H the same for all trials. Collect the untreated control samples at the 0 day sample interval.**

19. RESIDUE SAMPLE HANDLING AND SHIPMENT:

After residue sample collection, store samples in a freezer. If the samples cannot be placed into a freezer within approximately one hour, use an appropriate method of cooling and temperature-monitoring samples in order to maintain integrity.

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.

Shipment of frozen samples will be by freezer truck or express shipment. 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. 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.

For analysis, send samples to: Mr. Thomas Hendricks, 2747 Davis Road, Tifton, GA 31793, (229) 387-2392, FAX# 229-387-2352; e-mail: Tom.Hendricks@ars.usda.gov

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- Plot maps

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

20.06- Test substance storage conditions (including temperatures)

20.07- Data regarding calibration and use of application equipment

20.08- Treatment application data

20.09- Crop maintenance pesticides and cultural practices, test plot history, and soil information. (Reporting soil information from typical farm service soil analysis labs, or past history for the farm, or from official documents, such as the SCS Soil Survey for the test plot area is adequate for this study. The nature of this study is such that soil characteristics do not need to be determined under GLP standards.)

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- Meteorological/Irrigation records (temperature/humidity records for greenhouse trials)--required from planting of annual crops or for a minimum of one month prior to the first application onto perennial crops, until last residue sample collection. 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 plots

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.

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
Ms. Sharon D. Benzen, USDA, ARS, Crop Improvement & Protection Research, 1636 East Alisal Street, Salinas, CA 93905, (831) 755-2828, FAX# 831-755-2814; e-mail: Sharon.Benzen@ars.usda.gov	08757.14-CA*32	ARS
Brent Boutwell, U.C. Coop Extension, Imperial County, 1050 East Holton Street, Holtville, CA 92250-9615, (760) 352-9474 ext 32, FAX# (760) 352-0846, CELL# (760) 604-0801 e-mail: beboutwell@ucanr.edu	08757.14-CA35	WSR
Mr. David Studstill, University of Florida, Plant Sci Res & Edu Unit, 2556 W. Hwy 318, Citra FL 32113-2132; (352) 591-2678 x 251; Cell: 352-494-6514, FAX # 352-591-9860; e-mail: dwstud@ufl.edu	08757.14-FL157 (Decline)	SOR
Mr. Benjamin A. Fraelich, USDA, ARS, Crop Protection & Management, 2747 Davis Road,Tifton, GA 31793, (229) 387-2345, FAX# 229-387-2321; e-mail: Benjamin.Fraelich@ars.usda.gov. (Send US Mail to P.O. Box 748, Tifton, GA 31793)	08757.14-GA*189	ARS
Dr. Robin Bellinder, Horticulture Dept., Rm 164 Plant Science Bldg., Cornell University, Ithaca, NY 14853, (607) 255-7890, Farm: 607-844-8270, FAX# 607-255-0599; e-mail: rrb3@cornell.edu	08757.14-NY336	NER
Leona Horst, USDA, ARS, Application Technology Research, Room 132, Selby Hall, OARDC, 1680 Madison Ave., Wooster, OH 44691-4996, (330) 263-3691, FAX# 330-263-3841; e-mail: Leona.Horst@ars.usda.gov	08757.14-OH*351	ARS
Dr. Daniel J. Heider, University of Wisconsin - IPM Program 1575 Linden Drive Madison, WI 53706 608-262-6491 608-262-4743(FAX) djheider@wisc.edu	08757.14-WI482	NCR

RFC = Regional/ARS Field Coordinator

Location:

ARS: Dr. Paul H. Schwartz, 10300 Baltimore Ave., Rm 119, Bldg. 308, BARC-EAST, Beltsville, MD 20705; Tel: (301) 504-8256, FAX# 301-504-5048; e-mail: schwartp@ba.ars.usda.gov.

NCR: Dr. Satoru Miyazaki, IR-4 North Central Research Center, Michigan State Univ., 3900 Collins Road, Suite 1031B, Lansing, MI 48910-8396; Tel: (517) 336-4611, FAX# 517-432-2098; e-mail: ncrir4@msu.edu.

NER: Ms. Edith L. Lurvey, Dept. of Entomology, 630 W. North Street, Geneva, NY 14456; Tel: (315) 787-2308, FAX# 315-787-2326; e-mail: ell10@cornell.edu.

SOR: Dr. Michelle Samuel-Foo, Food & Env. Tox. Lab., Dept. of Food Science & Human Nutrition, Bldg 685 SW 23rd Drive, IFAS, Univ. of Florida, P.O. Box 110720, Gainesville, FL 32611-0720; Tel: (352) 392-1978 ext. 406, FAX# 352-392-1988; e-mail: mfoo@ufl.edu.

WSR: Ms. Rebecca Sisco, Regional Field Coordinator, Western Region IR-4 Project, Univ. of CA, Dept. of Environmental Toxicology, One Shields Ave., 4218 Meyer Hall, Davis, CA 95616; Tel: (530) 752-7634; FAX# 530-752-2866; e-mail: rsisco@ucdavis.edu.

24. LABORATORY PERSONNEL/ID NO.: LAB ID NO.: 08757.14-TIR01

LABORATORY RESEARCH DIRECTOR/TESTING LABORATORY:
Mr. Thomas A. Hendricks, USDA-ARS, CPMRU, 2747 Davis Road, P.O. Box 748, Tifton, GA 31793 (229) 387-2392, FAX# 229-387-2352; e-mail: Tom.Hendricks@ars.usda.gov

25. LABORATORY SAMPLE INVENTORY:

Treated and untreated samples of radish will be received from each of the field 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 that will maintain frozen sample integrity (generally less than -20°C), 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 $\leq$ 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), famoxadone + cymoxanil, from the Registrant. Contact Sheldon Sumpter, DuPont Agricultural Products, Phone: 302-451-3340, Fax: 302-451-3570; e-mail: sheldon.r.sumpter@dupont.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: (Either 3 & 4 or, 1 or 2)

1. Analytical Method for the Determination of DPX-JE874 and Cymoxanil Residues in Various Matrices. Deirdre DeMario, Gary L. Westberg, Sidney J. Hill, and Edward C. Nathan, Morse Laboratories and E.I. du Pont de Nemours and Company, Laboratory Project ID AMR 3705-95.

OR

2. Famoxadone + Cymoxanil: Magnitude of Residue on Hops, Joseph McClory, Jeromie Holt, E. I. du Pont de Nemours and Company, IR-4 PROJECT No.: 07796 Dated: 09 July 2002.

OR

3. Cymoxanil: "Analytical Method for the Determination of Cymoxanil and IN-KQ960 in Spinach (Leafy Vegetables) Using LC/MS", DuPont-13753, January 22, 2004, E.I. du Pont de Nemours and Company, DuPont Crop Protection, Global Technology Division, Stine-Haskell Research Center Newark, Delaware 19714-0030.

AND

4. Famoxadone: "Magnitude of Residues of Cymoxanil and Famoxadone in Tomatoes Following Application of DPX-KP481 50WG Fungicide at Maximum Label Rates – USA, 2002, DuPont-9822, May 2, 2003, E.I. du Pont de Nemours and Company, DuPont Crop Protection, Global Technology Division, Stine-Haskell Research Center Newark, Delaware 19714-0030.

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 each crop fraction prior to residue sample analysis of that crop fraction.

To validate the method, fortify some of the control samples in triplicate with famoxadone and cymoxanil at a minimum of 3 concentration levels, lowest level of method validation (0.02 ppm or lower for famoxadone and 0.05 ppm or lower for cymoxanil), 0.5 ppm and 5 ppm each.

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 for the total and/or combined residues of famoxadone + cymoxanil following the Working Method.

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 at 0.5 ppm famoxadone and 0.5 ppm cymoxanil.

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 prior to 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:

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: FAMOXADONE + CYMOXANIL MAGNITUDE OF THE RESIDUE ON RADISH

PR# 08757

SPONSOR

IR-4 Project Headquarters
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STUDY DIRECTOR

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