

INDONESIAN MINISTRY OF AGRICULTURE
NATIONAL PESTICIDE CLEARANCE PROTOCOL
Azoxystrobin + Difenoconazole / Dragon Fruit

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1. PROJECT TITLE:

Magnitude of Azoxystrobin and Difenoconazole Residues in Dragon fruit

2. JUSTIFICATION AND OBJECTIVES

Directorate of Quality and Standardization, D.G. of Processing and Marketing for Agric. Products, MOA has received a request for the minor use of Azoxystrobin and Difenoconazole on Dragon fruit for control of *Colletotrichum gloesporoides* in order to generate data for the establishment of ASEAN MRLs and to be submitted for JMPR evaluation.

To establish these MRLs, it is required that the magnitude of the residue in or on the commodity be determined in a supervised trials following the FAO Manual (2009). 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, in order to provide data and information for JMPR evaluation for the establishment of Codex MRLs.

To determine the magnitude of residues of total Azoxystrobin and Difenoconazole in or on Dragon fruit, this protocol will be employed using appropriate Standard Operating Procedures (SOP's) and will be conducted in accordance with **EPA's good laboratory practice standards** and consistent with the provisions outlined in the Organization for Economic Cooperation and Development (OECD) Series on Principles of Good Laboratory Practice and Compliance Monitoring.

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

Directorate of Quality and Standardization, D.G. of Processing and Marketing for Agric. Products, MOA, Jl. Harsono RM, No. 3, Ragunan, Jakarta Selatan 12550, Republic of Indonesia

4. STUDY DIRECTOR¹:

Dr. Michael P. Braverman IR-4 Project Headquarters, 500 College Road East, Suite 201 W, Princeton, NJ 08540, (732) 932-9575 X4610, FAX# (609) 514-2612, E-mail: braverman@aesop.rutgers.edu

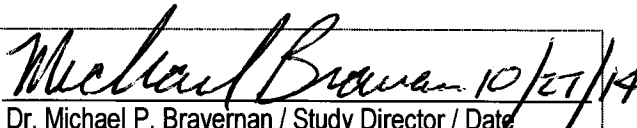
5. PROPOSED DATES:

Experimental Start : 2014
Experimental Termination: 2015
Study Completion: 2015

6. PROPOSED TEST SITES:

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

7. STUDY AUTHORIZATION:

Gardjita Budi/Testing Facility Management / Date Director, Directorate of Quality and Standardization, DG of Processing and Marketing for Agric. Products, MOA gbudi.jkt@gmail.com	 Dr. Michael P. Braverman / Study Director / Date IR-4 Project, Rutgers University braverman@aesop.rutgers.edu
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¹In case the Study Director is not available, contact Dr. Sri Noegrohati (+628122743383) for guidance.

*) FAO Manual on the Submission and Evaluation of Pesticide Residues Data, 2nd Ed, 2009
OECD GUIDELINE FOR THE TESTING OF CHEMICALS, Crop Field Trial, OECD/OCDE 509, © OECD, (2009)

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7.1 STUDY DIRECTOR INITIALS:

M. B.

7.2 Quality Assurance Review

This protocol has been reviewed by Quality Assurance

Mrs. Hasri
bpmptditlin@yahoo.co.id
Quality Assurance

Date

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 Good Laboratory Practice (GLP) Standards, 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 Good Laboratory Practices. 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:

Dragon fruit - Use a commercial variety.

Field trials will be conducted at the appropriate sites to support the establishment/maintenance of national MRLs, **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 consists of one untreated and one treated plots. Each plot consists of a minimum 8 trellis of dragon fruit plants to provide samples for non-decline (standard) trials and 12 trellis of dragon fruit plants to provide samples for a decline trial. Field trials 10993.14-ID02 (DIY) and 10993.14-ID04 (Bogor) provide samples for a decline trial (multiple sampling dates). Alternately there can be 2 treated plots, but each plot must still contain the minimum number of trees and produce the same number of samples as described in Section 18. Even if two treated plots are utilized, the treatment must be the same in both plots. Do not intentionally change concentration, application equipment or any other parameter between plots.

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

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 (minimum 20 meters) between each of the plots to prevent contamination, but for mist blower application a minimum of 30 meters is strongly preferred. When plants are used as a buffer between the

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untreated and treated plots, a lower distance is needed to prevent contamination, but the minimums indicated above must be observed.

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. **(Delete for granular applications)**
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 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 $\geq$ 30 GPA Example 2, Trial A has a volume of 60 GPA and Trial B has a volume $\geq$ 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	Different granular application type: broadcast or banded (only if label supports both types)
	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 coarse, and extra coarse are appropriate for herbicides only
	G	Different incorporation method for soil-applied test substance: mechanical or irrigation
	H	Different band width for soil applications: band width must vary by at least 50% of the lower width
	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	For test substances that must be applied through drip irrigation: surface drip line or buried drip line
	K	Different planting arrangement for annual crops: single row beds or multi-row beds (two or more rows on each bed)
	L	One trial shall have trellised plants and the other shall not
	M	Different training system for fruit trees (for example, central leader or open center)
	N	Different maturity of trees or bushes in fruit and nut studies—young trees or bushes in one trial and mature trees or bushes in the other; minimum 5 year age difference
	O	Different soil series, type, or texture (only in trials in which applications are made to the soil)
	P	Different formulations of the test substance (within the types generally considered equivalent) (This

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		option may be used only if the alternate formulation is included in Section 13 of this protocol or is added by protocol amendment)
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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.

NO	Field research location*	Characteristic	ID Number	treatment	Responsible
1	Sragen, Central Java Indonesia	Low land	10993.14-ID01	Azoxystrobin +Difenoconazole (non-decline study)	Wita Khairia
2	Kulon Progo (DIY) Indonesia	Low land, Sea shore	10993.13-ID02	Azoxystrobin +Difenoconazole (decline study)	Sri Sulasmi
3	Mojokerto, East Java Indonesia	Low land	10993.14-ID03	Azoxystrobin +Difenoconazole (non-decline study)	Issusila-ningtyas
4	Bogor, West Java Indonesia	High land	10993.14-ID04	Azoxystrobin +Difenoconazole (decline study)	Syanti Asviatuti
5	Lampung, South Sumatera Indonesia	Low land	10993.14-ID05	Azoxystrobin +Difenoconazole (non-decline study)	Erna Ryanti W
6	Malang, East Java Indonesia	High land	10993.14-ID06	Azoxystrobin +Difenoconazole (non-decline study)	Warastin Puji M
7	Vietnam	Coastal	10993.14-VN01	Azoxystrobin +Difenoconazole (non-decline study)	Tran Thanh Tung

*The listing of locations is only approximate and for planning purposes. The exact city within Indonesia may change depending on availability of a field at the proper time. Any changes must still provide

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

12. TEST SITE PREPARATION:

The test site has been prepared and maintained following good local agricultural practices for the production of dragon fruit 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 AMISTAR TOP formulation of **200 g/l azoxystrobin** and **125 g/l difenoconazole** (MOA Reg statement No. 4707/Kpts/SR.140/9/2013, CAS# 131860-33-8 and 119446-68-3) that has been characterized to meet GLP standards provided by Syngenta Indonesia. Copies of the Certificate of Analysis have been submitted to Indonesian Ministry of Agriculture. The test substance is stored in a secure, clean, dry area and document storage temperatures. Temperature monitoring begins within 2 days of receipt of the test substance, regardless of where it is held or stored. Test substance container(s) must be retained until the final study report is completed.

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

Study completion can be confirmed by contacting the Study Director.

Alternatively, some registrants will archive the test substance containers(s). 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:

The registrant (Syngenta) will archive a retention sample of the test substance. The registrant representative is:

Nanin Noorhajati

Crop Protection Regulatory Manager, Indonesia

PT. Syngenta Indonesia

Perkantoran Hijau Arkadia

Tower C, 9th Floor

Jl. TB Simatupang Kav. 88

Jakarta 12520

INDONESIA

Email : nanin.noorhajati@syngenta.com

Telp: +62 21 3042 1082 / Fax: +62 21 7883 6165 / IP Phone: 88701082

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 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.
- Agitate the test substance during the application, if practical, to ensure that it is well mixed.

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 or mistblower 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 calibrate².

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

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. The recalibration is required even if the equipment has been changed back to the parameters of the initial calibration. It is not necessary to recalibrate when CO₂ tanks are changed or when equipment is transported offsite or cleaned, 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 ($\pm 5\%$ of the last complete calibration) just prior to subsequent applications.

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 is greater than 5% (this statement does not apply to airblast or mistblower 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 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 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.

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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	AMISTAR TOP	151 grams azoxystrobin and 94 grams difenoconazole /hectare	756 ml/hectare	Foliar	300 to 1525 L/Ha

*The nominal formulation concentration of the test substance will be used in calculating application rates (see Section 13 for the nominal concentration).

** L/Ha=liters per hectare, according to the efficacy study, the spray volume was 1511 L/Ha using Knapsack, but when using mistblower at water flow rate 10 and fan speed 4, the required volume to cover 2000 trellises was 327 L/Ha

Make a total of 3 foliar applications of AMISTAR TOP at 10 day intervals. The first and second sequential applications will be made at 20 (± 2) days and 10 (± 1) days prior to harvest. The third (last application) will be made on the same day as harvest.

16. SUPPLEMENTAL CROP TREATMENTS:

Protect the integrity of the field trial by managing pests that may cause significant damage to the test crop. Only registered 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:

Two different pesticide residues studies: non-decline study and decline study will be carried out in these trials. Collect two samples from each plot or if there are 2 treated plots, one sample per treated plot. Collect each sample in a manner to assure an impartial sample that represents the entire plot (except plot ends, edges and borders). Only marketable fruit should be harvested. In all trials **marketable fruit is defined as being between 70-100% ripeness**.

17.A. Residue Sample Collection For Non-Decline Study

Samples from Sragen (10993.14-ID01), Mojokerto (10993.14-ID03), Lampung (10993.14-ID05), Malang (10993.14-ID06) and Vietnam(10993.14-VN01)are collected at -1, 0, 1 and 7 $\pm$ 1 days after last application, Collect at least 12 fruit weighing at least 2 kg marketable fruits, with scissors or knives, at different quadrants from high and low parts, from each plot, starting from the untreated plot, followed by treated plot. Do not cut or scratch the fruit. Each sample should be collected during a separate run through the entire plot.

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 that day prior to making the last application.

Follow proper handling practices with gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for one plot before proceeding to the next.

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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. Place bags of samples in a cool box.

17.B. Residue Sample Collection For Decline Study

Samples from Bogor (10993.14-ID04) and Kulonprogo (10993.14-ID02) are collected at -1, 0, 1, 3±1, 7±1, and 10±1 days after last application. Collect at least 12 fruit weighing at least 2 kg of marketable fruits, with scissors or knives, at different quadrants from high and low parts, from each plot, starting from the untreated plot, followed by treated plot. Do not cut or scratch the fruit. Each sample should be collected during a separate run through the entire plot.

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 that day prior to making the last application.

Follow proper handling practices with gloved hands and clean tools to prevent transfer of pesticide residue from one sample to another. If practical, complete harvest and sample preparation for one plot before proceeding to the next.

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. Place bags of samples in a cool box.

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 #; 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.A Field Residue Sample Inventory for Non-decline Study: Sragen (10993.14-ID01), Mojokerto (10993.14-ID03), Lampung (10993.14-ID05), Malang (10993.14-ID06) and Vietnam (10993.14-VN01)*

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM NUMBER OF FRUIT	MINIMUM WGT. OF SAMPLE	CROP FRACTION
A	01	Untreated	0	12 fruits	2 kg	fruits
B	01	Untreated	0	12 fruits	2 kg	fruits
C	02	Azoxystrobin + Difenoconazole	-1	12 fruits	2 kg	fruits
D	02	Azoxystrobin + Difenoconazole	-1	12 fruits	2 kg	fruits
E	02	Azoxystrobin + Difenoconazole	0	12 fruits	2 kg	fruits
F	02	Azoxystrobin + Difenoconazole	0	12 fruits	2 kg	fruits
G	02	Azoxystrobin + Difenoconazole	1	12 fruits	2 kg	fruits
H	02	Azoxystrobin + Difenoconazole	1	12 fruits	2 kg	fruits
I	02	Azoxystrobin + Difenoconazole	7±1	12 fruits	2 kg	fruits
J	02	Azoxystrobin + Difenoconazole	7±1	12 fruits	2 kg	fruits

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*Vietnam will take an extra set of duplicate samples(4 rather than 2). Only 2 out of 4 samples need to be shipped to Indonesia for analysis. The other 2 can be retained as a backup in case first set do not arrive frozen. All fruit must be harvested when in a marketable stage. This is defined as at least 70% ripe.

18.B Field Residue Sample Inventory for Decline Study: Bogor (10993.14-ID04) and Kulonprogo (10993.14-ID02)

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM NUMBER OF FRUIT	MINIMUM WGT. OF SAMPLE	CROP FRACTION
A	01	Untreated	0	12 fruits	2 kg	fruits
B	01	Untreated	0	12 fruits	2 kg	fruits
C	02	Azoxystrobin + Difenoconazole	-1	12 fruits	2 kg	fruits
D	02	Azoxystrobin + Difenoconazole	-1	12 fruits	2 kg	fruits
E	02	Azoxystrobin + Difenoconazole	0	12 fruits	2 kg	fruits
F	02	Azoxystrobin + Difenoconazole	0	12 fruits	2 kg	fruits
G	02	Azoxystrobin + Difenoconazole	1	12 fruits	2 kg	fruits
H	02	Azoxystrobin + Difenoconazole	1	12 fruits	2 kg	fruits
I	02	Azoxystrobin + Difenoconazole	3±1	12 fruits	2 kg	fruits
J	02	Azoxystrobin + Difenoconazole	3±1	12 fruits	2 kg	fruits
K	02	Azoxystrobin + Difenoconazole	7±1	12 fruits	2 kg	fruits
L	02	Azoxystrobin + Difenoconazole	7±1	12 fruits	2 kg	fruits
M	02	Azoxystrobin + Difenoconazole	10±1	12 fruits	2 kg	fruits
N	02	Azoxystrobin + Difenoconazole	10±1	12 fruits	2 kg	fruits

18.C Field Residue Sample Inventory for Decline Study: Bogor (10993.14-ID04) and Kulonprogo (10993.14-ID02)

Residue stability samples

The study of stability of residues in dragon fruit harvested at 7 days after last application, stored at ambient, then sampled (Placed in the freezer) at 10 and 14 days after the last application.

O	02	Azoxystrobin + Difenoconazole	10±2	12 fruits	2 kg	fruits
P	02	Azoxystrobin + Difenoconazole	10±2	12 fruits	2 kg	fruits
Q	02	Azoxystrobin + Difenoconazole	14±2	12 fruits	2 kg	fruits
R	02	Azoxystrobin + Difenoconazole	14±2	12 fruits	2 kg	Fruits

All fruit must be harvested when in a marketable stage. This is defined as at least 70% ripe.

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 such as wet ice, gel packs or dry ice and monitoring samples in order to maintain integrity. This does not apply to samples harvested in Section 18C.

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.

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

As per the sampling instructions, fruit must not be cut in the field. Subsequently, samples should be shipped as whole fruit. In general samples harvested within 6 hours or less of placement in a freezer can be cooled with wet ice. Samples requiring 6-12 hours of transport can be cooled with gel packs alone or in combination with wet ice or dry ice. Any samples requiring more than 12 hours of transit from harvest to placement in a freezer should be shipped with dry ice. If possible, place samples in a freezer first prior to shipment to the analytical laboratory.

Any samples requiring shipment by airplane (Even within Indonesia) should be placed in hard plastic coolers with the appropriate type of ice. Do not use styrofoam containers for shipment by air and kept cool with an appropriate form of ice.

All samples shipped from Vietnam to Indonesia should be frozen first prior to shipment and then shipped with dry ice at a ratio of 2kg dry ice for each kg of sample. Fruit must be shipped whole or ground with dry ice as described below. All samples must be handled in the same manner. Either ground or shipped whole. Do not ship some whole and some ground. Carefully coordinate the shipment of samples with the lab in Indonesia.

In all cases, control samples must be shipped separate from treated samples. Once they arrive at the destination, control samples and treated samples must be stored in separate freezers.

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. The receiving lab should fully document the integrity of the samples upon arrival.

For analysis, send samples to:

Ms Syanti Asvatiuti MSc
Plant Product Quality Testing Center, Directorate of Food Crops,
Jl. AUP Pasar Minggu, Kotak Pos 7236/Jks.PSM, Jakarta Selatan 12520
Telepon (021) 78835256, 7805652

Special grinding instructions for Vietnam.

Rather than shipping whole fruits to Indonesia, samples may be ground prior to shipping. This will greatly reduce the weight of samples to ship to Indonesia. Weigh fruit samples prior to grinding. Grind control samples along with a sufficient amount of dry ice to keep samples frozen. Grind treated samples after the control samples. Samples should appear as a very fine ground powder after grinding. If grinding a sample in portions. Combine all the portions of an individual sample into a plastic bag and shake to fully mix and distribute all portions to create a uniform ground powdered sample. To allow dry ice to sublime, place loosely closed bags in a deep freezer to allow dry ice to sublime for at least 24 hours. After fully sublimated, remix the sample in the plastic bag and withdraw 5 portions (100 grams) of the homogenised sample, place them in double plastic bag, label them and put them in the same deep-freezer. Ship the 5 portions and retain at least 500 grams of each ground sample in a deep freezer.

Prepare a mixture of azoxystrobin, difenoconazole and chlorpyrifos at a 1 mg/ml concentration. Take 3 whole dragon fruits, weight the mass and carefully disperse the standard solution to apply about 0.5 mg/kg concentration on whole fruit basis on the surface of the fruits avoiding runoff the solution, let it dry, then process the sample the same way as the treated samples. Withdraw 5 portions of the homogenised sample, place them in double plastic bag, label them and put them in the same deep-freezer. Ship with the treated samples together to Indonesia. These test sample portions shall be analysed in duplicate after the field samples were analysed as these data will serve as storage stability test as well.

All samples for analysis must be shipped to the laboratory frozen to Indonesia. Place samples in hard plastic coolers with a ratio of 2 kg of dry ice to each kg of sample.

20. FIELD DOCUMENTATION AND RECORD KEEPING:

All operations, data and observations appropriate to this study should be recorded directly and promptly into the 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 service soil analysis labs). 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 (Weather/irrigation records are 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- Equipment maintenance records with indication of routine vs. non-routine nature of maintenance

21. PROTOCOL/SOP MODIFICATIONS - FIELD RESEARCH:

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Consult with the Study Director 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 Field Data Book and other raw data to the Directorate of Quality and Standardization, D.G. of Processing and Marketing for Agric. Products, MOA, Jakarta, 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.

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.	Test Crop	Formulation
Ms. Ir. Sri Sulasmi Directorate General of Processing and Marketing of Agric. Product, Ministry of Agriculture, Jl. Harsono RM No. 3 Ragunan, Jakarta, Indonesia 62-8128007500 codex_kementan@yahoo.com ciami_12@yahoo.com sulasmi@deptan.go.id Resp. person: Wita Khairia	10993.14-ID01 Sragen, Central Java	Dragon Fruit	AmistarTop
Sri Sulasmi codex_kementan@yahoo.com ciami_12@yahoo.com sulasmi@deptan.go.id Resp. person: Sri Sulasmi	10993.14-ID02 Kulon Progo (DIY)	Dragon Fruit	AmistarTop
Sri Sulasmi codex_kementan@yahoo.com ciami_12@yahoo.com sulasmi@deptan.go.id Resp. person: Issusila- ningtyas	10993.14-ID03 Mojokerto, East Java	Dragon Fruit	AmistarTop

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Sri Sulasmi codex_kementan@yahoo.com ciami_12@yahoo.com sulasmi@deptan.go.id Resp. person: Syanti Asviastuti	10993.14-ID04 Bogor, West Java	Dragon Fruit	AmistarTop
Sri Sulasmi codex_kementan@yahoo.com ciami_12@yahoo.com sulasmi@deptan.go.id Resp. person: Erna Ryanti W	10993.14-ID05 Lampung, South Sumatera	Dragon Fruit	AmistarTop
Sri Sulasmi codex_kementan@yahoo.com ciami_12@yahoo.com sulasmi@deptan.go.id Resp. person: Warastin Puji M	10993.14-ID06 Malang, East Java	Dragon Fruit	AmistarTop
Tran Thanh Tung tungtran.spcc@gmail.com Southern Pesticide Control and Testing Center - PPD 28 Mac Dinh Chi St, Dist 1, Hochiminh City, Vietnam Phone: 84 8 38241114. Fax.: 84 8 38244187 Mob.: 01644187841	10993.14-VN01 Vietnam	Dragon Fruit	AmistarTop

24. LABORATORY PERSONNEL/ID NO.: LAB ID NO.: not applicable

LABORATORY RESEARCH DIRECTOR/TESTING LABORATORY:
Ms Syanti Asviatuti MSc sasviatuti@yahoo.com
Plant Product Quality Testing Center Directorate of Food Crops
Jl. AUP Pasar Minggu, Kotak Pos 7236/Jks.PSM, Jakarta Selatan 12520
Phone (62 21) 78835256, 7805652

25. LABORATORY SAMPLE INVENTORY:

Treated and untreated samples of Dragon fruits 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.

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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 from the field in a limited access area, at temperatures that will maintain frozen sample integrity (generally less than -20°C) until extraction. The samples must be stored as a whole.

27.A. Laboratory Sample Storage/Preparation For Non-Decline Study

Prior to grinding, defrost the samples just enough to cut the Dragon fruit. Weigh fruit before cutting. Grind the dragon fruit pieces with dry ice for a certain time. Collect representative subsample by quartering. The ground materials are processed immediately or store at -20°C to be used later.

Laboratory Residue Sample Inventory for Non-decline Study: Sragen (10993.14-ID01), Mojokerto (10993.14-ID03), Lampung (10993.14-ID05), Malang (10993.14-ID06) and Vietnam(10993.14-VN01)

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM NUMBER OF FRUIT	MINIMUM WGT. OF SAMPLE	CROP FRACTION
A	01	Untreated	0	12 fruits	2 kg	fruits
B	01	Untreated	0	12 fruits	2 kg	fruits
C	02	Azoxystrobin + Difenoconazole	-1	12 fruits	2 kg	fruits
D	02	Azoxystrobin + Difenoconazole	-1	12 fruits	2 kg	fruits
E	02	Azoxystrobin + Difenoconazole	0	12 fruits	2 kg	fruits
F	02	Azoxystrobin + Difenoconazole	0	12 fruits	2 kg	fruits
G	02	Azoxystrobin + Difenoconazole	1	12 fruits	2 kg	fruits
H	02	Azoxystrobin + Difenoconazole	1	12 fruits	2 kg	fruits
I	02	Azoxystrobin + Difenoconazole	7±1	12 fruits	2 kg	fruits
J	02	Azoxystrobin + Difenoconazole	7±1	12 fruits	2 kg	fruits

27.B. Laboratory Sample Storage/Preparation For Decline Study

Prior to grinding, defrost the samples just enough to cut the Dragon fruit. Weigh fruit before cutting. For untreated samples, grind the dragon fruit pieces with dry ice for a certain time. For treated samples, cut Dragon fruits in halves. Weigh the first halves as a whole fruit(peel and flesh), cut in smaller pieces and grind with dry ice for a certain time. Weigh the other halves, separate the peel from the flesh, weigh the separated peel and flesh. Cut separated peel and flesh in smaller pieces and grind separately with dry ice for a certain time. Collect representative subsample of whole fruit, peel and flesh by quartering. The ground materials are processed immediately or store at -20°C to be used later

Laboratory Residue Sample Inventory for Decline Study:Bogor (10993.14-ID04) and Kulonprogo (10993.14-ID02)

SAMPLE ID	TRT#	TREATMENT	DAYS AFTER LAST APPLIC.	MINIMUM NUMBER OF FRUIT	MINIMUM WGT. OF SAMPLE	CROP FRACTION
A	01	Untreated	0	12 fruits	2 kg	Whole fruits
B	01	Untreated	0	12 fruits	2 kg	Whole fruits, peel, flesh

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C	02	Azoxystrobin + Difenoconazole	-1	12 fruits	2 kg	Whole fruits
D	02	Azoxystrobin + Difenoconazole	-1	12 fruits	2 kg	Whole fruits, peel, flesh
E	02	Azoxystrobin + Difenoconazole	0	12 fruits	2 kg	Whole fruits
F	02	Azoxystrobin + Difenoconazole	0	12 fruits	2 kg	Whole fruits, peel, flesh
G	02	Azoxystrobin + Difenoconazole	1	12 fruits	2 kg	Whole fruits
H	02	Azoxystrobin + Difenoconazole	1	12 fruits	2 kg	Whole fruits, peel, flesh
I	02	Azoxystrobin + Difenoconazole	3±1	12 fruits	2 kg	Whole fruits
J	02	Azoxystrobin + Difenoconazole	3±1	12 fruits	2 kg	Whole fruits, peel, flesh
K	02	Azoxystrobin + Difenoconazole	7±1	12 fruits	2 kg	Whole fruits
L	02	Azoxystrobin + Difenoconazole	7±1	12 fruits	2 kg	Whole fruits, peel, flesh
M	02	Azoxystrobin + Difenoconazole	10±1	12 fruits	2 kg	Whole fruits
N	02	Azoxystrobin + Difenoconazole	10±1	12 fruits	2 kg	Whole fruits, peel, flesh

Do not composite samples.

The entire sample provide 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 $\leq 4^{\circ}\text{C}$ 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:

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Obtain the laboratory reference substance(s), Azoxystrobin (ICIA5504), Z-isomer of Azoxystrobin([methyl(Z)-2-(2-(6-(2-cyanophenoxy)pyrimidin-4-yloxy)phenyl)-3 methoxyacrylate])and Difenoconazole (CGA 169374) from the Registrant.

Contact person: **Nanin Noorhajati**
Crop Protection Regulatory Manager, Indonesia
PT. Syngenta Indonesia
Perkantoran Hijau Arkadia
Tower C, 9th Floor
Jl. TB Simatupang Kav. 88
Jakarta 12520
INDONESIA
Email : nanin.noorhajati@syngenta.com
Telp: +62 21 3042 1082 / Fax: +62 21 7883 6165 / IP Phone: 88701082

Document the date the analytical standards are received, the source, stated purity, storage conditions, and expiration date (Appendix No. 1, CoA Analytical Standard) .

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:

29.1 REFERENCE METHOD:

1. AOAC Official Method 2007.01, Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate. Liquid Chromatography/Tandem Mass Spectrometry, First Action 2007
2. European Union Reference Laboratory for Pesticide Residues, EURL-FV Multiresidue Method using QuEChERS followed by GC-QqQ/MS/MS and LC-QqQ/MS/MS for Fruits and Vegetables

29.2 REFERENCE METHOD MODIFICATIONS/METHOD VALIDATION

The above listed Reference Method(s) may be modified if needed for the test matrix. Validation of the modified analytical method must be carried out on each crop fractions prior to residue sample analysis of that crop fraction.

To validate the method, fortify some of the test matrices (whole fruit, peel and flesh) obtained from the control samples in triplicate with Azoxystrobin (ICIA5504), Z-isomer of azoxystrobin [methyl(Z)-2-(2-(6-(2-cyanophenoxy)pyrimidin-4-yloxy)phenyl)-3 methoxyacrylate] and Difenoconazole (CGA 169374) at a minimum of 3 concentration levels, lowest level of method validation (0.01 ppm or lower), 0.1 ppm, and 1 ppm.

A minimum of 6 fortification samples (recovery spikes) at the lowest level of method validation (LLMV) is required 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.**

29.3 SAMPLE ANALYSIS:

Samples will be analyzed for the residues of Azoxystrobin, Z-isomer and Difenoconazole 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.

29.3.A For non-decline study: Sragen (10993.14-ID01), Mojokerto (10993.14-ID03), Lampung (10993.14-ID05), Malang (10993.14-ID06) and Vietnam(10993.14-VN01) analyze whole fruit

29.3.B For decline study, Bogor (10993.14-ID04) and Kulonprogo (10993.14-ID02), analyze whole fruit, peel and flesh separately

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 residue sample analysis, adequate concurrent recovery samples that bracket the actual residues should be analyzed. [Concurrent recovery samples must be run at a minimum of two levels over the course of the sample analyses, with at least one concurrent recovery sample below the lowest sample residue (or at the lowest level of method validation) and at least one concurrent recovery sample higher than the highest sample residue found in the treated samples.] 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 prior to completion of the analytical phase of the study.

29.4 STORAGE STABILITY ANALYSIS:

As soon as possible after receipt of samples, a minimum of six subsamples of whole fruit, peel and flesh of the control shall be fortified with Azoxystrobin, Z-isomer and Difenoconazole at 0.1 ppm.

Three samples of each analyte and crop fraction will be analyzed after the appropriate storage period (generally, greater than the longest interval that an individual sample was stored between collecting the sample in the field/processing facility and analysis, unless otherwise specified by the Study Director). The remaining samples will be retained for long-term storage.

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

29.5 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:

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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 Directorate of Quality and Standardization, D.G. of Processing and Marketing for Agric. Products, MOA, Jakarta, Indonesia, 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 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

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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 and sent to the Directorate of Quality and Standardization, D.G. of Processing and Marketing for Agric. Products, MOA, Jakarta, Indonesia, 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.

**IR-4
FIELD DATA BOOK**

**TITLE:
MAGNITUDE OF AZOXYSTROBIN AND DIFENOCONAZOLE RESIDUES IN
DRAGON FRUIT**

PR# 10993

SPONSOR

Directorate of Quality and Standardization,
D.G. of Processing and Marketing for Agric. Products,
Ministry Of Agriculture,
Jl. Harsono RM, No. 3, Ragunan,
Jakarta Selatan 12550,
Republic of Indonesia

STUDY DIRECTOR

Michael Braverman, Ph.D.
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