

**AAFC PESTICIDE RESIDUE STUDY PLAN  
INDAZIFLAM: MAGNITUDE OF THE RESIDUE ON ASPARAGUS  
STUDY #: AAFC15-066R**

**AGRICULTURE AND AGRI-FOOD CANADA (AAFC)  
PESTICIDE RESIDUE STUDY PLAN**

**INDAZIFLAM: MAGNITUDE OF THE RESIDUE ON  
ASPARAGUS**

**STUDY #: AAFC15-066R  
(PR#: 11429 – for reference only)**

**STUDY DIRECTOR:  
Greg O'Neill, M.Sc.  
AAFC Minor Use Pesticide Program  
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Ottawa, Ontario K1A 0C6  
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Email: greg.oneill@agr.gc.ca**

**FIELD TRIAL LOCATION:**

Refer to Section 10, page 3

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**1. STUDY TITLE:**

Indaziflam: Magnitude of the Residue on Asparagus

**2. JUSTIFICATION AND OBJECTIVES:**

Agriculture and Agri-Food Canada (AAFC) has received a request for the minor use label expansion of indaziflam on asparagus. To establish a Maximum Residue Limit (MRL)/tolerance, it is required that the magnitude of the residue on the commodity be determined as per PMRA Regulatory Directive 98-02 (June 1, 1998), Residue Chemistry Guidelines and Directive 2010-05 revisions to the residue Chemistry Crop Field Trial requirements (December 21, 2010). 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 registration submission. To determine the magnitude of the residue on asparagus, this study plan will be implemented using applicable Standard Operating Procedures (SOP) and conducted under provisions outlined in Organisation for Economic Cooperation and Development (OECD) Principles of Good Laboratory Practices (GLP) (1997 Revision). Any work conducted in the USA will be conducted according to Environmental Protection Agency (EPA) Good Laboratory Practice standards, 40 CFR Part 160, which are acceptable to OECD standards.

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

AAFC Minor Use Pesticide Program, Building 57, Central Experimental Farm, 960 Carling Avenue, Ottawa, ON K1A 0C6, Tel: (613) 715-5390, Fax: (613) 759-1400.

**4. STUDY DIRECTOR:**

Greg O'Neill, Building 57, Central Experimental Farm, 960 Carling Avenue, Ottawa, ON, K1A 0C6, Phone: (613) 694-2454, Fax: (613) 759-1400, Email: greg.oneill@agr.gc.ca.

**5. COMPLIANCE STATEMENT:**

The test facility and appropriate test sites (field and laboratory) will be responsible for certifying that its portion of the study will be conducted in accordance with the OECD Principles of GLP (1997 Revision). Any work conducted in the USA will be conducted according to EPA Good Laboratory Practice standards, 40 CFR Part 160, amended as effective Oct. 16, 1989, which are acceptable to OECD standards. A statement of compliance, together with any deviations will be signed and submitted by the responsible Study Director in the Final Report and by the Principal Investigator in their Raw Data Field Notebook (RDFN) or Final Analytical Report.

**6. QUALITY ASSURANCE:**

Quality Assurance (QA) duties and responsibilities will be in conformance with the OECD Principles of GLP (1997 Revision). Any work conducted in the USA will be conducted according to EPA Good Laboratory Practice standards, 40 CFR Part 160, which are acceptable to OECD standards. A Quality Assurance Statement will be provided by the QA for each site, for each Raw Data Field Notebook, Final Analytical Report and Final Report. It shall include the type of inspections, the date inspections were made and date(s) any findings were reported to the Study Director, Principal Investigator (if applicable), and management(s).

**7. TEST FACILITY RECORD KEEPING:**

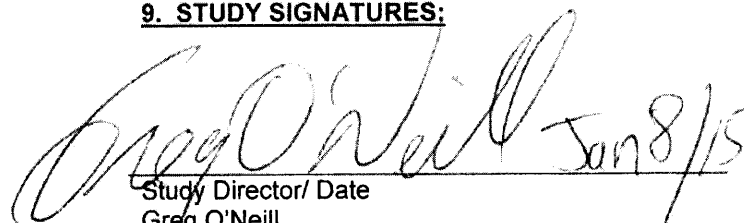
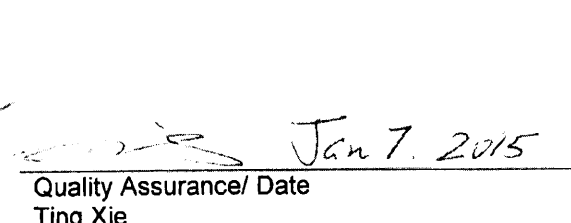
A study file will be initiated and maintained by the Test Facility. Original study plan, amendment(s), and deviation(s) if any, as well as the original raw data (e.g. RDFNs, laboratory data [each of which may contain copies of facility records]), Final Analytical Report and Final Report will be archived by the Test Facility.


**8. PROPOSED DATES:**

Experimental Start:	January 2015
Experimental Termination:	December 2018

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**9. STUDY SIGNATURES:**

 <u>Greg O'Neill Jan 8/15</u>	 <u>Jan 7, 2015</u>
<b>Study Director/ Date</b> Greg O'Neill AAFC Minor Use Pesticide Program Building 57, Central Experimental Farm 960 Carling Avenue Ottawa, ON K1A 0C6 Tel: (613) 694-2454 Fax: (613) 759-1400 Email: greg.oneill@agr.gc.ca	<b>Quality Assurance/ Date</b> Ting Xie AAFC Minor Use Pesticide Program Building 57, Central Experimental Farm 960 Carling Avenue Ottawa, ON K1A 0C6 Tel: (613) 759-6637 Fax: (613) 759-1400 Email: ting.xie@agr.gc.ca

  
Jan 8/15  
**Test Facility Management/ Sponsor Representative/ Date**  
 Ian Gardiner/Submission Manager  
 AAFC Minor Use Pesticide Program  
 Building 57, Central Experimental Farm  
 960 Carling Avenue  
 Ottawa, ON K1A 0C6  
 Phone: (613) 759-1581  
 Fax: (613) 759-1400  
 Email: ian.gardiner@agr.gc.ca

**10. FIELD PERSONNEL/TRIAL ID NO:**

(Responsible for Sections 11-23)

The Principal Investigator and Site Management must sign and return the attached GLP acceptance form (see Appendix A) for each Trial ID #.

<b>PRINCIPAL INVESTIGATOR:</b>	<b>TRIAL ID No. (Zone 2)</b>
Marylee Ross Univ. of MD/LESREC 27664 Nanticoke Rd. Salisbury, MD 21801 Phone: (410) 742-8788 ext. 310 Fax: (410) 742-1922 Email: mross@umd.edu	AAFC15-066R-214
<b>TEST SITE MANAGEMENT:</b>	
Edith Lurvey Cornell University – NYSAES, Entomology Department 630 W. North Street Geneva, NY 14456-1371 Phone: (315) 787-2308 Fax: (315) 787-2326 Email: ell10@cornell.edu	

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<b>PRINCIPAL INVESTIGATOR:</b>	<b>TRIAL ID No. (Zone 2)</b>
Roger B. Batts North Carolina State University NCSU IR-4 Field Research Center, NCSU Campus Box 7523 Raleigh, NC 27695-7523 Phone: (919) 515-1668 Fax: (919) 513-7226 Email: roger_batts@ncsu.edu	AAFC15-066R-221
<b>TEST SITE MANAGEMENT:</b>	
Michelle Samuel-Foo University of Florida P.O. Box 110720 SW 23rd Drive, Bldg. 685 Gainesville, FL 32611 Phone: (352) 392-1978 x 406 Fax: (352) 392-1988 Email: mfoo@ufl.edu	
<b>PRINCIPAL INVESTIGATOR:</b>	<b>TRIAL ID No. (Zone 5)</b>
Bernard H. Zandstra Dept. of Horticulture Michigan State University A440 Plant & Soil Science Building East Lansing, MI 48824-1325 Phone: (517) 353-6637 Fax: (517) 432-2242 Email: zandstra@msu.edu	AAFC15-066R-215
<b>TEST SITE MANAGEMENT:</b>	
Satoru Miyazaki Michigan State University IR-4 North Central Reg. Res. Ctr. 3815 Technology Boulevard, Suite 1031B Lansing, MI 48910-8396 Phone: (517) 336-4611 Fax: (517) 432-2098 Email: ncrir4@cns-msu.edu	
<b>PRINCIPAL INVESTIGATOR:</b>	<b>TRIAL ID No. (Zone 5)</b>
Dr. Daniel J. Heider University of Wisconsin - IPM Program 1575 Linden Drive Madison, WI 53706 Phone: (608) 262-6491 Fax: (608) 262-4743 Email: djheider@wisc.edu	AAFC15-066R-216

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<b>TEST SITE MANAGEMENT:</b>	
Satoru Miyazaki Michigan State University IR-4 North Central Reg. Res. Ctr. 3815 Technology Boulevard, Suite 1031B Lansing, MI 48910-8396 Phone: (517) 336-4611 Fax: (517) 432-2098 Email: ncrir4@cns-msu.edu	
<b>PRINCIPAL INVESTIGATOR:</b>	<b>TRIAL ID No. (Zone 5)</b>
<i>The Principal Investigator will be included at a later date and added via an amendment.</i>	AAFC15-066R-217
<b>TEST SITE MANAGEMENT:</b>	
<i>The Test Site Management will be included at a later date and added via an amendment.</i>	
<b>PRINCIPAL INVESTIGATOR:</b>	<b>TRIAL ID No. (Zone 10)</b>
Sharon D. Benzen USDA/ARS/Crop Improvement and Protection Research 1636 East Alisal Street Salinas, CA 93905 Phone: (831) 755-2828 Fax: (831) 755-2814 Email: sharon.benzen@ars.usda.gov	AAFC15-066R-218 (decline)
<b>TEST SITE MANAGEMENT:</b>	
Paul H. Schwartz Jr. USDA/ARS/Off. of Minor Use Pesticides 10300 Baltimore Avenue, Rm. 119, Bldg. 308, BARC-E Beltsville, MD 20705 Phone: (301) 504-8256 Fax: (301) 504-5444 Email: paul.schwartz@ars.usda.gov	
<b>PRINCIPAL INVESTIGATOR:</b>	<b>TRIAL ID No. (Zone 10)</b>
Keri Skiles University of California KREC IR-4 Field Research Center 9240 South Riverbend Parlier, CA 93648 Phone: (559) 646-6061 Fax: (559) 646-6015 Email: kmskiles@ucanr.edu	AAFC15-066R-219
<b>TEST SITE MANAGEMENT:</b>	
Rebecca Sisco University of California, Department of Env. Toxicology 1 Shields Avenue, Meyer Hall, Rm. 4218 Davis, CA 95616 Phone: (530) 752-7634 Fax: (530) 752-2866 Cell: (530) 867-1664 Email: rsisco@ucdavis.edu	

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<b>PRINCIPAL INVESTIGATOR:</b>	<b>TRIAL ID No. (Zone 11)</b>
John Harvey USDA-ARS Yakima Agricultural Research Laboratory 5230 Konnowac Pass Road Wapato, WA 98951 Phone: (509) 454-6553 Fax: (509) 454-5646 Email: John.Harvey@ARS.USDA.GOV	AAFC15-066R-220
<b>TEST SITE MANAGEMENT:</b>	
Paul H. Schwartz Jr. USDA/ARS/Off. of Minor Use Pesticides 10300 Baltimore Avenue, Rm. 119, Bldg. 308, BARC-E Beltsville, MD 20705 Phone: (301) 504-8256 Fax: (301) 504-5444 Email: paul.schwartz@ars.usda.gov	
<b>PRINCIPAL INVESTIGATOR:</b>	<b>TRIAL ID No. (Zone 11)</b>
Dan Groenendale Washington State University IAREC 24106 N. Bunn Rd. Prosser, WA 99350-9687 Phone: (509) 786-9365 Fax: (509) 786-9370 Email: dgroenendale@wsu.edu	AAFC15-066R-293
<b>TEST SITE MANAGEMENT:</b>	
Rebecca Sisco University of California, Department of Env. Toxicology 1 Shields Avenue, Meyer Hall, Rm. 4218 Davis, CA 95616 Phone: (530) 752-7634 Fax: (530) 752-2866 Cell: (530) 867-1664 Email: rsisco@ucdavis.edu	

**11. TEST SYSTEM/CROP:**

Asparagus - use a commercial cultivar (*Asparagus officinalis*). At a minimum, record the cultivar name and, if available, the age of the asparagus. Select an asparagus patch that has been established for at least three full growing seasons after transplanting. Field trials will be conducted at the designated sites.

**NOTE:** If a Principal Investigator is assigned more than one trial in this study, the following requirements must be met. Contact the Study Director prior to trial initiation to confirm trial distinction requirements.

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:

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Set	Option	Description
1	A	Trial sites must be separated by at least 32 km (20 miles)
	B	First application or planting date in each trial is separated by at least 30 days
	C	Different crop cultivar (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 cultivars 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 94 L/Ha (10 GPA) difference) Example 1, Trial A has a volume of 187 L/ha (20 US gallons/acre) and Trial B has a volume $\geq$ 281 L/ha (30 US gallons/acre) 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 (Not applicable)	Use of an adjuvant/surfactant (of any suitable type) in the tank mix for one trial vs. <u>no adjuvant/surfactant</u> 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 RDFN or 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 or test items 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

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

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

**12. TRIAL SYSTEM DESIGN and STATISTICAL METHOD:**

Each trial site will consist of one untreated plot and one treated plot. Each individual plot will be established with a minimum area of 100 m<sup>2</sup> (1077 ft<sup>2</sup>) (this includes plot end areas). In the decline trial, the treated plot will be established with a minimum area of 200 m<sup>2</sup> (2153 ft<sup>2</sup>) (this includes plot end areas). If necessary, the plot size should be increased so that sample requirements can be met.

At a minimum, a 5 m (16.4 feet) buffer zone will be employed around the treated and untreated plots to prevent contamination.<sup>1</sup> Since this pesticide use pattern is not registered on this crop, federal law requires that the treated (and untreated if specified in Section 17) crop must be destroyed, or handled in such a way that it is not consumed as a human food or animal feed. Document the crop destruction in the RDFN. If any questions arise regarding crop disposition, contact the Study Director. Mark plots with identifiable markers containing at a minimum the Trial ID # (AAFC15-066R-XXX), and treatment number or treatment name that will persist for the duration of the field research trial or that can be readily replaced. A plot map enabling trial reconstruction by a third party must be created. This study is not designed for the statistical evaluation of field data.

**13. TRIAL SITE PREPARATION AND MAINTENANCE:**

Prepare or select a trial site that has been and will continue to be maintained following local, good agricultural practices for the production of asparagus including fertilization, irrigation and other practices that ensure good crop production. The trial site must be maintained throughout the season to ensure that pests do not interfere with treatment applications or crop maturity.

The trial site cannot have been treated with a chemical similar in mode of action to the test item (as outlined in Section 17) for a minimum of 1 year prior to use. The trial site will have a known pesticide history of a minimum of 1 year and **preferably 3 years**.

If needed, before application of the test item, within the trial site asparagus spears may be harvested on a regular basis as per commercial practice to prevent spears from reaching a size too large or in such poor condition as to preclude the harvest of the crop for sampling purposes.

Note: A soil analysis ( $\leq$  5 years old) must be provided for each trial (see Section 21 for details). If an artificial medium is used, provide a detailed description of its composition in place of a soil analysis.

<sup>1</sup> Note that the buffer zone is considered the area outside of the plot and the spray zone that is a conflicting chemistry and test item free zone. The spray zone is an area before and after the treated plot that allows the operator to activate the boom and get up to the appropriate speed before entering the plot; and it is an area after the plot that allows the application to continue outside of the treated plot; which helps to ensure a uniform coverage to the treated plot. It is also an area where the boom can be primed and discharged, as long as contamination of the plot is avoided.

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**14. TEST ITEM:**

Use the Alion Herbicide (suspension concentrate, 200 g a.i./L, equivalent to 1.67 lbs a.i./US gallon) formulation of indaziflam (CAS # 730979-19-8; PCP Reg. No. 30451; US-EPA Reg. No. 264-1106) **that has been characterized to meet GLP standards**. AAFC will arrange procurement of GLP test item from the Registrant. Upon receipt, document the Lot/Batch Number, condition, quantity received and confirmation of GLP characterization. Contact the Study Director if there are any concerns regarding the GLP characterization, expiration date, label identification of the test item (e.g., the name on the bottle or certificate of analysis (CoA) is different from the study plan), etc., and if the CoA does not come with the test item. Store the test item in a secure, clean, dry area at temperature ranges noted in the product label or other references.

**Prior to test item disposal, contact the Study Director for specific instructions.** Unless otherwise specified, the Registrant will archive a retention sample of the test item.

**15. TEST ITEM APPLICATION:**

Each trial requires a unique spray mixture (i.e. do not use the spray mixture from one field trial on another field trial). To ensure the test item is well mixed, agitate during the application if practical. If practical, observe the test item in the spray mixture and provide documentation in the RDFN that the test item was completely dissolved/mixed in the carrier before application. Use application equipment that will provide uniform application of the test item in the required spray volume (see Section 16). Apply the test item as specified (see Section 16), in a manner that represents or simulates the major application technique that is used by area commercial growers. The test item, if applied in a mixture, must be applied to the test system within 2 hours of mixing. The test item must be applied when wind speeds are less than 10 km/hour (6.2 miles/hour) (unless approved by the Study Director), and in a manner to ensure accurate delivery and to prevent contamination to adjacent plots. Avoid application of the test item when heavy rain is forecast and do not apply irrigation within 48 hours of application.

To ensure accurate delivery, calibration for output and speed must be performed. Just prior<sup>2</sup> to the application of test item, calibrate for nozzle or hopper output and speed (equipment or walking speed), by performing a minimum of three, consecutive acceptable checks (within  $\pm 5\%$  of the average output, or  $-5\%$  to  $+10\%$  of the target pass time for speed calibration); or by performing a minimum number of runs for which at least 75% of the total number of checks are acceptable (i.e. 3 acceptable runs out of a total of 4 checks performed. Note in this situation only the values from the 3 acceptable runs will be used for calibration calculation). This is considered a **complete calibration**. Conduct the speed calibration at the edge of the test plot, or on similar terrain. The uncharged spray boom may be held over or directed at the plot.

At a minimum, for multiple applications performed on the same day between trials, a single recheck of the output and speed is necessary for each trial. A single output check must be conducted to confirm consistent delivery ( $\pm 5\%$  of the last complete calibration) just prior to subsequent applications. This is considered a **calibration recheck**. Note: a calibration recheck is only acceptable if application parameters or equipment components have not changed. If the **calibration rechecks** results in an output that differs from the mean output of the **complete calibration** by more than  $\pm 5\%$ , then the equipment must be completely re-calibrated.

If application parameters (e.g., application type, water volume) or equipment components (e.g., nozzle tips) have changed from the initial calibration, another **complete calibration** (of nozzle output and/or speed, depending on what was modified) must be performed and documented, even if the equipment has been changed back to the parameters of the initial calibration. (Equipment logs should be used to document changes in the equipment parameters).

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

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If the complete calibrations were conducted as part of another trial, a true copy of all complete calibrations referenced along with the required rechecks performed for this trial are to be included in raw data field notebook. **Calculations for the amount of test item to be applied will always be based upon mean output calculated from the most recent complete nozzle output or speed calibration data, not on the recheck results.**

Record actual application pass times in the field notebook and verify the accuracy of the application. The application is considered acceptable if the accuracy is within -5% and +10% of the study plan specified application rate and the spray volume range limits. If the application does not meet this range, the Study Director must be notified of this deviation before proceeding with this trial.

Use application methods that result in maximum coverage. Ensure the targeted spray area receives a consistent spray by starting and ending the application before and after the defined plot area, respectively (this includes the plot ends and guard rows that will not be sampled from). For directed applications, in which the treated area is less than the plot area (row/bed spacing), do not proportionally reduce the application rate (the amount of active ingredient applied per hectare). Direct the entire per-hectare rate into the treated area. If row widths in the research plots are greater than local commercial practices, then the application rate should be calculated using the maximum commercial row width. Contact the Study Director if guidance is needed.

**16. APPLICATION TREATMENTS AND TIMING:**

Trt. #	Treatment	Application Timing	Target Rate of Active Ingredient	Target Rate of Formulated Product <sup>1</sup>	Application Type <sup>2</sup>	Minimum Spray Volume
01	Untreated	Not Applicable	Not Applicable	Not Applicable	Not Applicable	Not Applicable
02	Indaziflam	Pre-emergence	95.2 g a.i./ha (0.085 lbs a.i./ac)	476 mL/ha (193 mL/ac)	Broadcast	93.5 L/ha (10 US gal./acre)

<sup>1</sup> The nominal formulation concentration of the test item will be used in calculating the application rate (see Section 14 for the nominal concentration).

<sup>2</sup> Make one broadcast application of the test item prior to spear emergence (i.e. pre-emergent application). If spears have emerged, make the application after a clean harvest, then cover exposed plants with soil prior to application.

**Harvest asparagus 7 (±1) days after application.**

**Surfactant:** Not applicable

**Note:** for any other additive to the spray solution (such as but not limited to antifoaming agents or pH adjusters) contact the Study Director for approval.

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

**17. SUPPLEMENTAL CROP TREATMENTS:**

The integrity of the field trial should be protected by minimizing damage to the test crop caused by pests. Only registered maintenance pesticides applied according to labelled directions can be used, unless approved by the Study Director. Approval from the Study Director to use non-

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registered pesticides is to be documented in the RDFN. Make identical applications to the untreated and treated plots. If an unregistered maintenance pesticide is used, both treated and untreated plots must be destroyed (Section 12). Document all supplemental crop treatments. **DO NOT USE** pesticides which are similar to the test item, or other chemicals that might interfere with analysis of the test item. If unsure, contact the Study Director.

**18A. RESIDUE SAMPLE COLLECTION**

**(SEE SAMPLE INVENTORY IN STUDY PLAN PART 19A):**

Collect two samples from the untreated plot and two samples from the treated plot. Each sample is to be collected in a manner to ensure a representative, impartial sample. Collect samples at 7 ( $\pm 1$ ) days after the application, when asparagus spear is mature, starting with the untreated plot first, if both plots are being harvested by the same person. Otherwise, the order in which the samples are collected will not be an issue, if contamination between plots is minimized. A minimum of 1 plant from the plot ends must be avoided while sampling. In addition, avoid sampling from areas (i.e. plot edges) that may have not received complete spray coverage of the test item.

Harvest a minimum of 24 spears by cutting or snapping the asparagus at the soil surface from at least 24 separate plants per sample randomly from the entire plot to achieve a representative sample while avoiding sampling from the plot ends. **Each sample should weigh a minimum of 2 kg (4.4 lbs)**, but preferably not more than 3 kg (6.6 lbs). If loose soil adheres to the asparagus spear, remove it by lightly brushing it off with a dry, clean soft brush or cloth (document what is used to remove the soil or debris), prior to placement into the sample bag. If necessary, lightly rinse off with a minimal amount of clean water (do not scrub). Pat lightly while drying with clean paper towels. **DO NOT RUB WHILE RINSING OR DRYING THE ASPARAGUS SPEARS.**

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 one plot before proceeding to the next. Store all samples in plastic-lined cloth bags or bags approved by the Study Director. See Section 20 for residue sample handling directions. Document how sampling was conducted in the RDFN. Identify each bag of samples as follows:

**Trial ID No.** - enter Trial ID Number (AAFC15-066R-XXX); **Commodity (Crop)** - enter crop fraction; **Chemical** - enter common chemical name and formulation; **Sample ID No.** - enter sample ID; **Date Sampled** - enter harvest/sampling dates; **Application Rate (g a.i./ha)** - enter application rate, or not applicable (for untreated sample only) and **Investigator**- enter name of Principal Investigator.

**NOTE:** An extra set of samples may be collected if deemed necessary (i.e. for shipping assurance) by the PI, or Study Director. Document the collection, labelling and disposal procedures for these samples, in the RDFN. If extra samples are taken, then identify each sample according to instructions outlined in the paragraph above, with the addition of the word 'DUPLICATE' or 'EXTRA' beside the sample ID No. Contact the Study Director for approval regarding disposal of extra samples.

**18B. SAMPLE COLLECTION (FOR DECLINE TRIAL ID# 218 outlined in Section 10 ONLY):**  
**(SEE SAMPLE INVENTORY IN STUDY PLAN PART 19B):**

In addition to the samples required in Section 18A (i.e. 7 ( $\pm 1$ ) day), collect two samples from the treated plot at 4 ( $\pm 1$ ), 10 ( $\pm 1$ ) and 13 ( $\pm 1$ ) days from the application. Follow the sampling method described above.

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**19A. RESIDUE SAMPLE INVENTORY:**

Sample ID	TRT #	Treatment	Days After Application	Minimum Quantity <sup>2</sup>	Crop Fraction
A	01	Untreated	N/A <sup>1</sup>	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear
B	01	Untreated	N/A <sup>1</sup>	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear
C	02	Indaziflam	7 (±1)	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear
D	02	Indaziflam	7 (±1)	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear

<sup>1</sup> The days after application is not applicable (NA) since the asparagus were not treated. However, these samples should be targeted for collection at 7 (±1) days after the application.

<sup>2</sup> Collect a minimum of 24 asparagus spears from at least 24 separate plants from the entire plot to achieve a representative sample weighing a minimum of 2 kg (4.4 lbs).

**19B. DECLINE SAMPLE INVENTORY****(FOR DECLINE TRIAL ID# 218 as outlined in Section 10 ONLY):**

Sample ID	TRT #	Treatment	Days After Application	Minimum Quantity <sup>2</sup>	Crop Fraction
A	01	Untreated	N/A <sup>1</sup>	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear
B	01	Untreated	N/A <sup>1</sup>	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear
C	02	Indaziflam	7 (±1)	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear
D	02	Indaziflam	7 (±1)	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear
E <sup>3</sup>	02	Indaziflam	4 (±1)	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear
F <sup>3</sup>	02	Indaziflam	4 (±1)	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear
G	02	Indaziflam	10 (±1)	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear
H	02	Indaziflam	10 (±1)	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear
I	02	Indaziflam	13 (±1)	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear

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J	02	Indaziflam	13 ( $\pm 1$ )	24 spears from at least 24 separate plants AND min. 2 kg or 4.4 lbs	Spear
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<sup>1</sup>The days after application is not applicable (NA) since the asparagus were not treated. However, these samples should be targeted for collection at 7 ( $\pm 1$ ) days after the application.

<sup>2</sup>Collect a minimum of 24 asparagus spears from at least 24 separate plants from the entire plot to achieve a representative sample weighing a minimum of 2 kg (4.4 lbs).

<sup>3</sup>Note: Samples E and F are to be collected at 4 ( $\pm 1$ ) days after the application.

**20. RESIDUE SAMPLE HANDLING AND SHIPMENT:**

Place the samples into a freezer. If the samples cannot be placed into a freezer within approximately one hour after collection, an appropriate method of cooling samples must be used to maintain sample integrity and container temperatures must be monitored. The methods used in harvest, sample handling, and storage will be outlined generally in SOPs, and must be described in raw data. For pre-shipment storage, the samples will be held frozen at temperatures generally less than  $-18^{\circ}\text{C}$  ( $0^{\circ}\text{F}$ ), allowing for normal variations due to freezer cycling, sample movement, etc. Freezer logs must be used to document all sample additions to and removals from freezer storage. All storage temperatures must be monitored and documented. Shipment of frozen samples must be by freezer truck or "express" shipment, unless approved by the Study Director. Samples must be frozen prior to shipment. Shipments sent via express shipment (overnight carriers such as Federal Express or Purolator) 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, raw data field notebook, communication note, etc.

Send samples to the laboratory identified in table below, as soon as practical. For samples packed with dry ice, avoid shipments from Thursday through Sunday. Note: Section 24 needs to identify a PI prior to sample shipment. If this information is not completed, please contact the Study Director.

Trial ID No.	Ship to: (Trial ID No., Contact and Shipping Address)
All trial ID No. listed in Section 10	AAFC15-066R-222  <i>The shipping address will be identified at a later date, at which time the shipping information will be added by amendment</i> See Section 24 for responsible person for this trial ID No.

**21. FIELD DOCUMENTATION AND RECORD KEEPING:**

All operations, data and observations appropriate to this study should be recorded directly and **promptly** into the RDFN. The content of the RDFN should be sufficiently detailed to completely reconstruct the field trial. At a minimum, collect and maintain the following raw data:

- Names of all personnel conducting specific research functions
- Study plan amendments relevant to the field trial
- Deviations from study plan and standard operating procedures
- Trial site information, including historic pesticide use
- Plot maps
- Test item receipt, use and disposition records
- Test item storage conditions (including minimum and maximum temperatures)

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- Data regarding calibration and use of application equipment
- Treatment application
- Crop maintenance pesticides, crop production and cultural practices
- Residue sample identification, collection, storage conditions and handling
- Residue sample shipping information
- Description of crop destruction, or explanation for lack of destruction
- Meteorological/Irrigation records<sup>3</sup>
- Pass times (if applicable) and other data to confirm amount of material applied to plots
- Other applicable data requested in the RDFN<sup>4</sup> that are needed to prove that the conduct of the study was in accordance with the study plan.

**22. STUDY PLAN/SOP MODIFICATIONS - FIELD RESEARCH:**

Consult with the Study Director regarding desired changes to the study plan prior to occurrence. If appropriate an amendment will be issued. Any deviations to the study plan or to a standard operating procedure will require the Principal Investigator or Study Director to complete a deviation form. Any deviation should be communicated to the Study Director either verbally, by fax or email within **48 hours** (document in communication log) and in writing on the form provided, within **7 days** of occurrence or recognition. The Study Director will assess the impact of the deviation on the study and act accordingly.

**23. RAW DATA FIELD NOTEBOOK/ARCHIVING:**

The Principal Investigator will ensure that the completed **original** RDFN is forwarded to the GLP Admin after sample shipment and appropriate review. The Principal Investigator will maintain a complete certified true copy of these field documents.

**24. LABORATORY PERSONNEL/TRIAL ID NO.:**

(Responsible for Sections 25-35)

The Principal Investigator and test site management must sign the GLP Acceptance form (Appendix A) and return as directed.

**PRINCIPAL INVESTIGATOR:**

*The PI will be indicated at a later date and added via an amendment.  
The PI must be identified before sample shipment.*

**TRIAL ID No.**

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**TEST SITE MANAGEMENT:**

*The Test Site Management will be indicated at a later date and added via an amendment.*

*The laboratory will be identified at a later date, at which time the appropriate information will be added by amendment.*

**25. LABORATORY SAMPLE INVENTORY:**

Treated and untreated crop samples will be received from the field sites outlined in Section 20 (for responsible persons see Section 10). Notify the appropriate Principal Investigator and Study Director of sample receipt by returning (by fax, email, or mail) a copy of the completed Chain of

<sup>3</sup> Weather/irrigation records for the entire month 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. Daily climatic records for the trial year (growing period) and at a minimum the 10 year mean data, rainfall and temperature (if possible with standard deviations) must be provided.

<sup>4</sup> Report soil information from a soil analysis that is  $\leq 5$  years old (organic matter, pH, Cationic Exchange Capacity, textural fractions, and preferably soil texture) or from any official documents necessary, such as a Soil Survey or US-NRCS soil web data, to accurately document the requested information for this trial. The nature of this study is such that soil characteristics do not need to be determined under GLP standards. If an artificial medium is used, provide a detailed description of its composition, in place of a soil analysis.

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Custody form or a similar laboratory form used for sample arrival confirmation.

**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 (Note: use of the field sample identification number is acceptable). A cross-reference must be maintained between the assigned laboratory sample number and the identification utilized in the Sample Chain of Custody Form received from the field sites. Both identification numbers must be reported in the Final Analytical Report.

**27. LABORATORY SAMPLE STORAGE/PREPARATION:**

Store samples in a limited access area at temperatures that will maintain frozen sample integrity (generally less than  $-18^{\circ}\text{C}$  ( $0^{\circ}\text{F}$ )), allowing for normal variations due to freezer cycling, sample movement, etc. until extraction. The samples may be stored whole or macerated, depending on the standard procedure of the analytical laboratory. Note: The entire sample is to be macerated prior to taking a sample for analysis **and samples are not to be composited**. Contact Study Director if guidance is needed. All storage temperatures, conditions and location of sample storage must be monitored and documented.

*Information for Sections 28 – 35 will be identified at a later date, at which time the appropriate information will be added by amendment.*

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**APPENDIX A**

GLP Acceptance Form

**Trial ID #:** AAFC15-066R-XXX\_\_\_\_\_ (add unique three digit number)

I acknowledge that I have read, and understood, the material contained in the assigned sections of this study plan. The research will be conducted in accordance with this study plan and the OECD GLP Principle of Good Laboratory Practices (revision 1997). Any work conducted in the USA will be conducted according to EPA Good Laboratory Practice standards, 40 CFR Part 160, amended as effective Oct. 16, 1989, which are acceptable to OECD standards. In addition, I will cooperate with the Quality Assurance Personnel in scheduling needed inspections and documenting responses to QA audit reports.

Principal Investigator (PI):

Printed Name	Signature	Date
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Acknowledged by Test Site Manager:

Printed Name	Signature	Date
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*The following Individual or Company will be responsible for the Quality Assurance for this trial*

\_\_\_\_\_  
Name of Quality Assurance (Print)

***Form Completion and Return Instructions:*** At a minimum, the PI is to sign this form prior to performing any experimental work. Once the form has been completed, a true copy of the form needs to be sent to the individual identified below and the original retained in the RDFN or lab raw data by the PI.

**GLP Admin**  
AAFC Minor Use Pesticide Program  
Building 57, Central Experimental Farm  
960 Carling Avenue  
Ottawa, ON, Canada  
K1A 0C6  
Fax: 613-759-1400  
Email: GLPArchivist@agr.gc.ca