

US EPA ARCHIVE DOCUMENT



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

AUG 16 2000

OFFICE OF  
PREVENTION, PESTICIDES AND  
TOXIC SUBSTANCES

AUG 16 2000

MEMORANDUM

SUBJECT: Experimental Use Permit For Bird Shield Repellent (EPA Reg. No. 66550-EUP-001) Containing 26.4% Methyl Anthranilate As its Active Ingredient In or On Corn, Sunflowers, and Apples. (Chemical No. 128725): Evaluation of Residue Chemistry and Analytical Method. MRID Nos. 450651-01, -02, -03, -04, and -05; Case No. 066100 ; Submission No. S580610; DP Barcodes: D266613

FROM: Freshteh Toghrol, Ph.D., Senior Scientist *F. Toghrol*  
Biochemical Pesticides Branch  
Biopesticides & Pollution Prevention Division

TO: James Downing, Regulatory Action Leader  
Biochemical Pesticides Branch  
Biopesticides & Pollution Prevention Division

ACTION R REQUESTED

The Bird Shield Repellent Corporation is requesting an experimental use permit (EUP) and a temporary tolerance exemption from the requirement of a tolerance for Methyl Anthranilate in or on corn, sunflowers, and apples.

To support this EUP, the registrant has submitted five volumes of residue chemistry data MRID Nos 45065101, 45065102, 45065103, 45065104, and 45065105): two volumes address the method used for determining methyl Anthranilate residues in/on corn, sunflowers, and apples, and three volumes report the results of crop field trials on corn, sunflowers, and apples.

*1/27*

## BPPD CONCLUSIONS AND RECOMMENDATIONS

- 1a. The registrant did not submit a label or a petition for this EUP. However, the registrant has submitted a registered CSF and label for Bird Shield (EPA Reg. No. 66550-1) containing 26.4% methyl anthranilate on cherries, blueberries, and grapes. The registrant must submit a petition and a label for this EUP.
- 1b. Based on submitted residue data (after submission of the petition by the registrant) a temporary exemption from requirement of a tolerance for residues of methyl anthranilate on sweet corn, sunflowers, and apples can be published.
- 1c. The label for this EUP must specify a use pattern consistent with the conducted field trials. Additionally, since no data were provided for residue levels in feedstuffs which are associated with these crops, the label needs to prohibit livestock grazing.
2. Methyl anthranilate is exempt (40 CFR 180.1143) from the requirement of a tolerance on blueberries, cherries, and grapes, when used in accordance with good agricultural practices.
- 3a. For the purpose of this EUP and the temporary exemption from requirement of a tolerance for residues of MA on sunflower, corn, and apples, the submitted analytical method GC/MS detector is acceptable. However, the analytical method used has a very low percent recovery (see conclusion 3b) and also a very large range of percent recovery (see conclusion 3c). In the future for residue studies, the registrant must provide an analytical method with much better range and higher percent recovery. An acceptable analytical method and its detector must be reliable and provide consistent range percent recovery and has recover of 80%-100%.
- 3b. A gas chromatography/mass spectrometry (GC/MS; DB-35MS capillary column; MS detection in scan or SIM modes) was used to determine the residues of MA on surface-treated sweet corn kernels, sunflower seeds, and apples. MA recovery on macerated **sweet corn** samples was 20-27% (depending on the MS detection mode), for surface-washed corn samples was 26-37% in one experiment (one questionable sample had recovery of 22 and 120% for scan and SIM modes, respectively) and 46-72% in a second experiment. Individually treated kernels (off-the cob) had recoveries of 63.31-98.53%. For **sunflowers**, the mean recovery of the treated samples (seeds on whole sunflower heads were treated and washed) was 36.51-37.04%. For **apples**, the mean MA recovery was 72.05%.
- 3c. The analytic method was validated by an laboratory for individually treated sweet corn kernels (off the cob) and sunflower seeds. Sample analysis was by GC/MS (XTI-5 bonded 5% phenyl capillary column; MS in scan mode). The standard deviation of six replicate injections of the 1.01 µg/mL standard (peak areas of 689-1128; mean ± SD is 933 ± 194) and a regression curve derived from the response of analytic standards (1.01-101 µg/mL) were used to determine the LOD and LOQ as 2.09 and 2.91 µg/mL, respectively. For **sweet corn**, recoveries of 46.1-114% were obtained, although the reviewer obtained 68-167% by an alternate calculation. For **sunflowers**, the recoveries of 84.5-125% were obtained (validation was not performed for **apples**).

- 3d. The registrant determined that the recovery of MA from a glass surface (0.1 mg in 20  $\mu$ L; dried 2.5-10 minutes under ambient conditions) was 67-94%, irrespective of drying time (mean  $\pm$  SD was  $81 \pm 14$ ).
- 3e. An analytical method for animal commodities was not addressed by the registrant. This guideline is potentially relevant to this petition because sweet corn, sunflowers, and apples each give rise to animal feedstuffs.
- 3f. The petitioner did not address the potential of the recovery of MA (or any metabolites) by any FDA Multiresidue Methods.
- 4a. Bird Shield Repellent Corporation conducted one sweet corn field trial in 1999. Of the 12 treated plots, 6 received two applications of 0.286 lb ai/A repellent (0.572 lb ai/A/season; 1X recommended label application rate) and the other 6 received two foliar applications of 0.573 lb ai/A repellent (1.144 lb ai/A/season; 2X label rate). The applications were made 10 and 5 days prior to harvest (5-day PHI). Samples for residue analysis (cobs with husks and silk were removed) were collected at both 1 hour and 10 days after the 1<sup>st</sup> application (i.e. at anticipated harvest). Residue levels in all samples were adjusted for 46% method recovery and were corrected for control values. For the 1-hour samples, residue levels for the 1X and 2X treatments ranged from -0.084 to 5.579 ppm and -0.068 to 0.429 ppm, respectively. For the 10-day samples, residues for all the 1X samples and for all but two 2X samples were below control levels.
- 4b. The petitioner conducted one sunflower field trial in 1999; it consisted of two control and 8 treated plots. Four of the treated sites received two foliar applications of 0.286 lb ai/A repellent (0.572 lb ai/A/season; 1X recommended label application rate) and the other 4 received two applications of 0.573 lb ai/A repellent (1.144 lb ai/A/season; 2X label rate). The first application was 14 days prior to harvest and the second was 7 days prior to harvest (7-day PHI). Samples were collected at both one hour and 14 days after the first application (i.e. at anticipated harvest). MA residue levels were adjusted for the 36.78% method recovery and were corrected for control values. Most of the samples had residue levels below those of controls: the 1-hour 1X and 2X samples had residues of -0.053 to 0.058 ppm and -0.048 to 0.099 ppm, respectively, whereas the 14-day 1X and 2X samples had residues -0.140 to 0.170 ppm and -0.091 to 2.002 ppm, respectively.
- 4c. The petitioner conducted one apple field trial in 1999 in which 4 plots received one foliar application of 1.145 lb ai/A (1X recommended label application rate) and the other 4 received one application of 2.290 ai/A (2X label rate). The repellent was applied 14 days prior to the anticipated harvest and samples were collected 5 minutes (or 1 hour; unclear from study) after treatment and again 14 days after treatment. MA residue levels were adjusted for 72.05% method recovery and were corrected for control values. The 5-minute (or 1 hour) 1X and 2X samples had residues of 0.031-1.507 ppm and 1.217-3.348 ppm, respectively, whereas the 14-day 1X and 2X samples were 0.026-0.115 ppm and -0.006 to -0.024 ppm, respectively.
- 5a. The potential of MA residues in processed commodities was not addressed by the registrant. This guideline is potentially relevant to this petition because sunflowers and apples yield processed

commodities of regulatory interest: sunflower meal and oil for sunflowers, and wet apple pomace and apple juice for apples.

- 5b. The registrant did not address the potential for confined/field accumulation of MA residues in rotational crops. The submitted Bird Shield Repellent Concentrate label for cherries, blueberries, and grapes provides no guidelines for rotational crops (possibly because these crops are perennials). Since sweet corn, sunflowers, and apples are annuals, plantback guidelines are needed and the default plantback interval of 12 months needs to be imposed for crops not listed on the label.

### RECOMMENDATIONS

Sufficient data have been submitted by the registrant to support an exemption from the requirement of a residue tolerance for methyl anthranilate (MA), the ai in the formulation Bird Shield® Repellent Concentrate, for use on corn, sunflowers, and apples (as intended for human consumption). Consumption of animal feedstuffs arising from these treated crops is prohibited. MA is already exempt from the requirement for a tolerance on blueberries, cherries, and grapes (40 CFR 180.1143), based on a theoretical maximal concentration of 35 ppm MA on cherries, which is comparable to the 33 ppm present naturally in concord grapes (see 1/3/95 memo of G.J. Herndon, PP#2E4071, DP Barcode D209599). The presently submitted field trials with sweet corn (kernels), sunflowers (seeds), and apples yielded MA residues < 33 ppm, even when taking into account the 4.5X and 14X maximum theoretical concentration factors for processed commodities of sunflower seeds and apples, respectively. Therefore, by analogy, sweet corn kernels, sunflower seeds, and apples should also be exempt from the requirement of a tolerance for MA, as formulated in Bird Shield® Repellent. However, because no studies were conducted on residue levels in animal feedstuffs which are associated with these crops, and no animal feeding or metabolism studies were conducted, feeding animals crop commodities treated with MA is prohibited. **The label needs to prohibit livestock grazing and the use of these crops as animal feedstuffs, and to impose a plantback interval of 12 months for crops not listed on the label.** If these restrictions are unenforceable or if the registrant wishes to omit the restrictions and/or to obtain the exemption from a residue tolerance for other crops, additional field trials, processing, animal feeding and/or metabolism studies are required.

DATA EVALUATION REPORT

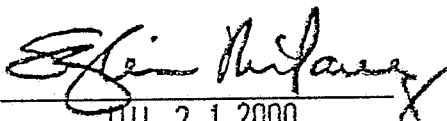
METHYL ANTHRANILATE  
[Bird Shield® Bird Repellent Concentrate]

DP Barcode: D266613      MRIDs: 45065101 through 45065105  
Submission #: S580610      Petition #: none given  
PC Code: 128725      EPA Reg. No.: 066550-EUP-001  
CAS#: 134-20-3


Prepared for  
Biopesticides and Pollution Prevention Division  
Office of Pesticide Programs  
U.S. Environmental Protection Agency  
2800 Crystal Drive  
Arlington, VA 22202

Prepared by  
Chemical Hazard Evaluation Group  
Toxicology and Risk Analysis Section  
Life Sciences Division, Oak Ridge National Laboratory  
Oak Ridge, TN 37830  
Work Assignment No. 38

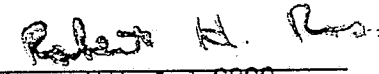
Primary Reviewer:  
Sylvia Milanez, Ph.D., D.A.B.T.

Signature:   
Date: JUL 21 2000

Secondary Reviewers:  
Robin Brothers, Ph.D., D.A.B.T.

Signature:   
Date: JUL 21 2000

Robert H. Ross, M.S., Group Leader

Signature:   
Date: JUL 21 2000

Quality Assurance:  
Eric B. Lewis, M.S.

Signature:   
Date: JUL 21 2000

Disclaimer

This review may have been altered by the BPPD subsequent to signing by Oak Ridge National Laboratory personnel.

Oak Ridge National Laboratory, Managed and Operated by UT-Battelle, LLC., for the U.S. Department of Energy under contract number DE-AC05-00OR22725.

DATA EVALUATION REPORT

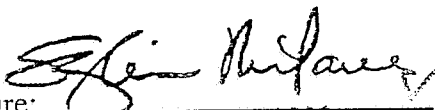
**METHYL ANTHRANILATE**  
[Bird Shield® Bird Repellent Concentrate]

DP Barcode: D266613      MRIDs: 45065101 through 45065105  
Submission #: S580610      Petition #: none given  
PC Code: 128725      EPA Reg. No.: 066550-EUP-001  
CAS#: 134-20-3


Prepared for  
Biopesticides and Pollution Prevention Division  
Office of Pesticide Programs  
U.S. Environmental Protection Agency  
1921 Jefferson Davis Highway  
Arlington, VA 22202

Prepared by  
Chemical Hazard Evaluation Group  
Toxicology and Risk Analysis Section  
Life Sciences Division, Oak Ridge National Laboratory  
Oak Ridge, TN 37830  
Work Assignment No. 38

Primary Reviewer:  
Sylvia Milanez, Ph.D., D.A.B.T.

Signature:   
Date: JUL 21 2000

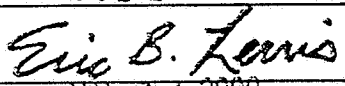
Secondary Reviewers:  
Robin Brothers, Ph.D., D.A.B.T.

Signature:   
Date: JUL 21 2000

Robert H. Ross, M.S., Group Leader

Signature:   
Date: JUL 21 2000

Quality Assurance:  
Eric B. Lewis, M.S.

Signature:   
Date: JUL 21 2000

Disclaimer

This review may have been altered by the BPPD subsequent to signing by Oak Ridge National Laboratory personnel.

Oak Ridge National Laboratory, Managed and Operated by UT-Battelle, LLC., for the U.S. Department of Energy under contract number DE-AC05-00OR22725.

F. Toghrol Date, 7/9/00

**DATA EVALUATION REPORT**

REQUEST FOR AN EXEMPTION FOR THE REQUIREMENT OF A TOLERANCE FOR METHYL ANTHRANILATE IN/ON :

**CORN, SUNFLOWERS, and APPLES**  
(DP Barcode D266613; PC code 128725)

INTRODUCTION

The Bird Shield Repellent Corporation has submitted materials in support of an exemption from the requirement of a residue tolerance for methyl anthranilate (MA) (formulated as Bird Shield® Repellent, 26.4% w/w ai, 2.29 lbs ai/gal, EPA Reg. No. 66550-EUP-001), for use on corn, sunflowers, and apples. The formulation product label states that it is comprised of all food-grade ingredients. Methyl anthranilate is a biochemical pesticide that acts to repel certain bird species without toxic effects, and is found naturally in foods such as concord grapes (a level of 33 ppm is reported in the published literature). MA is listed as GRAS under 21 CFR 182.60 (used as a flavoring agent in foods and as a component of perfumes) and is exempt from the requirement for a tolerance on blueberries, cherries, and grapes under 40 CFR 180.1143, when used in accordance with good agricultural practices. The exemption was granted following a request for registration by Dolphin Trust and the determination that application of the repellent to cherries at the proposed label rate resulted in a worst-case residue level of 35 ppm, which is comparable to MA levels found naturally in concord grapes (see 1/3/95 memo of G.J. Herndon, PP#2E4071, DP Barcode D209599).

Inert ingredients present in the Bird Shield repellent formulation, as listed on the submitted Confidential Statement of Formula (CSF), are: (1) [redacted] (2) [redacted] [redacted] the compound name is misspelled on the CSF], and (3) [redacted] Similar inert ingredients that are exempt from tolerances under 40 CFR 180.1001(c) are (1) [redacted] conforming to 21 CFR 172.860, although insufficient information was provided to determine whether the inert ingredient in the formulation conformed to 21 CFR 172.860 [redacted] are permitted for direct addition to food for human consumption under 21 CFR 172.863); (2) [redacted] (not specified), which is a synonym for [redacted] with a limit of [redacted] of the pesticide formulation [redacted] represents [redacted] of Bird Shield repellent). [redacted]

INERT INGREDIENT INFORMATION IS NOT INCLUDED



INVEST INGREDIENT INFORMATION IS NOT INCALCULATED

[REDACTED] are permitted as direct additives to food for human consumption under 21 CFR 172.120 and 21 CFR 172.135, respectively.

Five volumes of residue chemistry data were submitted (MRIDs 45065101, 45065102, 45065103, 45065104, and 45065105): two volumes address the method used for determining methyl anthranilate residues in/on corn, sunflowers, and apples, and three volumes report the results of crop field trials on corn, sunflowers, and apples. Also submitted were a letter from L.R. Askham, president of the Bird Shield Repellent Corporation (MRID 45065100), a CSF for methyl anthranilate, and a product label for use of Bird Shield® on cherries, blueberries; grapes, turf, and non-fishbearing bodies of water.

CONCLUSIONS

OPPTS 830 Series GLNs: Product Properties

- 1. Product chemistry data requirements for MA were not addressed by the registrant in the present submission.

OPPTS GLN 860.1200: Proposed Uses

- 2. The registrant provided neither a petition (Section B) nor a product label for the MA formulation Bird Shield® Repellent use on sweet corn, sunflowers, or apples. The formulation is intended to repel/discourage various bird species from feeding on the crops. A product label was provided for use on cherries, blueberries, grapes, turf, and non-fishbearing bodies of water; this label states that application is to begin when fruit begins to ripen or birds begin feeding on the crop, and is to continue every 6 to 8 days or when odor cannot be detected (reapply if heavy rains wet surfaces within 24 hours after application), with a PHI of 6 to 8 days. The use patterns (i.e. the 1X recommended label application rate) for the submitted field trials were as follows: sweet corn and sunflowers received two applications of 0.286 lb ai/A repellent (0.572 lb ai/A/season), the applications being 10 and 5 days prior to harvest (5-day PHI) for sweet corn and 14 and 7 days prior to harvest (7-day PHI) for sunflowers; apple trees received one application of 1.145 lb ai/A at 14 days prior to harvest. The registrant needs to submit a petition and label for sweet corn, sunflowers, and apples that specifies a use pattern consistent with the conducted field trials. Additionally, the label needs to prohibit livestock grazing and the use of these crops as animal feedstuffs and to impose a plantback interval of 12 months for all crops not listed on the label.

OPPTS GLN 860.1300: Nature of the Residue - Plants

- 3a. The nature of MA residues in plants was not addressed by the registrant. All reported crop residues were for the parent compound.

OPPTS GLN 860.1300: Nature of the Residue - Animals

- 3b. The nature of the residue in animals was not addressed by the registrant. This guideline is potentially relevant to this petition because sweet corn, sunflowers, and apples each give rise to animal feedstuffs.

OPPTS GLN 860.1340: Analytical Methods - Plants

- 4a. The method of residue analysis for MA on surface-treated sweet corn kernels, sunflower seeds, and apples consisted of gas chromatography/mass spectrometry (GC/MS; DB-35MS capillary column; MS detection in scan or SIM modes) analysis of methanol washes of the crop surfaces (macerated corn samples were also evaluated). Prior to the methanol wash, corn leaves and the sunflower outer layer of leaves and pistils were removed. The corn and sunflower methanol washes were additionally filtered (0.2 micron) prior to their application to the GC. Neither a LOQ (limit of quantitation) nor a LOD (limit of detection) were identified. MA recovery on macerated sweet corn samples was 20-27% (depending on the MS detection mode), for surface-washed corn samples was 26-37% in one experiment (one questionable sample had recovery of 22 and 120% for scan and SIM modes, respectively) and 46-72% in a second experiment. Individually treated kernels (off-the cob) had recoveries of 63.31-98.53%. For sunflowers, the mean recovery of the treated samples (seeds on whole sunflower heads were treated and washed) was 36.51-37.04%. For apples, the mean MA recovery was 72.05%.
- 4b. The analytic method was validated by an laboratory for individually treated sweet corn kernels (off the cob) and sunflower seeds. Sample analysis was by GC/MS (XTI-5 bonded 5% phenyl capillary column; MS in scan mode). The standard deviation of six replicate injections of the 1.01 µg/mL standard (peak areas of 689-1128; mean ± SD is 933 ± 194) and a regression curve derived from the response of analytic standards (1.01-101 µg/mL) were used to determine the LOD and LOQ as 2.09 and 2.91 µg/mL, respectively. For sweet corn, recoveries of 46.1-114% were obtained, although the reviewer obtained 68-167% by an alternate calculation. For sunflowers, the recoveries of 84.5-125% were obtained (validation was not performed for apples).
- 4c. The registrant determined that the recovery of MA from a glass surface (0.1 mg in 20 µL; dried 2.5-10 minutes under ambient conditions) was 67-94%, irrespective of drying time (mean ± SD was 81 ± 14).

OPPTS GLN 860.1340: Analytical Methods - Animals

- 4d. An analytical method for animal commodities was not addressed by the registrant. This guideline is potentially relevant to this petition because sweet corn, sunflowers, and apples each give rise to animal feedstuffs.

OPPTS GLN 860.1360: Multiresidue Method

- 4e. The petitioner did not address the potential of the recovery of MA (or any metabolites) by any FDA Multiresidue Methods.

OPPTS GLN 860.1380: Storage Stability Data - Plants

- 5a. Storage stability studies were not conducted for corn, sunflower, or apples. For all three crops, the MA was applied to the crop surface and was subsequently washed off with methanol to evaluate the MA recoveries. The length of time between MA application and the methanol washes was not stated, although GC/MS analysis of the methanol washes took place within approximately 1-3 months of sample application.

OPPTS GLN 860.1380: Storage Stability Data - Animal Commodities

- 5b. Storage stability of animal commodities was not addressed by the registrant. This guideline is potentially relevant to this petition because sweet corn, sunflowers, and apples are each the source of animal feedstuffs.

OPPTS GLN 860.1480: Meat/Milk/Poultry/Eggs

6. Potential MA residues in meat, milk, poultry, and eggs were not addressed by the registrant. This guideline is potentially relevant to this petition because sweet corn, sunflowers, and apples are the source of animal feedstuffs: corn forage, stover, and cannery waste; sunflower meal; and wet apple pomace.

OPPTS GLN 860.1500: Crop Field Trials

- 7a. Bird Shield Repellent Corporation conducted one sweet corn field trial in 1999. Of the 12 treated plots, 6 received two applications of 0.286 lb ai/A repellent (0.572 lb ai/A/season; 1X recommended label application rate) and the other 6 received two foliar applications of 0.573 lb ai/A repellent (1.144 lb ai/A/season; 2X label rate). The applications were made 10 and 5 days prior to harvest (5-day PHI). Samples for residue analysis (cobs with husks and silk were removed) were collected at both 1 hour and 10 days after the 1<sup>st</sup> application (i.e. at anticipated harvest). Residue levels in all samples were adjusted for 46% method recovery and were corrected for control values. For the 1-hour samples, residue levels for the 1X and 2X treatments ranged from -0.084 to 5.579 ppm and -0.068 to 0.429 ppm, respectively. For the 10-day samples, residues for all the 1X samples and for all but two 2X samples were below control levels.
- 7b. The petitioner conducted one sunflower field trial in 1999; it consisted of two control and 8 treated plots. Four of the treated sites received two foliar applications of 0.286 lb ai/A repellent (0.572 lb ai/A/season; 1X recommended label application rate) and the other 4

received two applications of 0.573 lb ai/A repellent (1.144 lb ai/A/season; 2X label rate). The first application was 14 days prior to harvest and the second was 7 days prior to harvest (7-day PHI). Samples were collected at both one hour and 14 days after the first application (i.e. at anticipated harvest). MA residue levels were adjusted for the 36.78% method recovery and were corrected for control values. Most of the samples had residue levels below those of controls: the 1-hour 1X and 2X samples had residues of -0.053 to 0.058 ppm and -0.048 to 0.099 ppm, respectively, whereas the 14-day 1X and 2X samples had residues -0.140 to 0.170 ppm and -0.091 to 2.002 ppm, respectively.

- 7c. The petitioner conducted one apple field trial in 1999 in which 4 plots received one foliar application of 1.145 lb ai/A (1X recommended label application rate) and the other 4 received one application of 2.290 ai/A (2X label rate). The repellent was applied 14 days prior to the anticipated harvest and samples were collected 5 minutes (or 1 hour; unclear from study) after treatment and again 14 days after treatment. MA residue levels were adjusted for 72.05% method recovery and were corrected for control values. The 5-minute (or 1 hour) 1X and 2X samples had residues of 0.031-1.507 ppm and 1.217-3.348 ppm, respectively, whereas the 14-day 1X and 2X samples were 0.026-0.115 ppm and -0.006 to -0.024 ppm, respectively.

#### OPPTS GLN 860.1520: Processed Food/Feed

8. The potential of MA residues in processed commodities was not addressed by the registrant. This guideline is potentially relevant to this petition because sunflowers and apples yield processed commodities of regulatory interest: sunflower meal and oil for sunflowers, and wet apple pomace and apple juice for apples.

#### OPPTS GLN 860.1850 and 860.1900: Confined/Field Accumulation in Rotational Crops

9. The registrant did not address the potential for confined/field accumulation of MA residues in rotational crops. The submitted Bird Shield Repellent Concentrate label for cherries, blueberries, and grapes provides no guidelines for rotational crops (possibly because these crops are perennials). Since sweet corn, sunflowers, and apples are annuals, plantback guidelines are needed and the default plantback interval of 12 months needs to be imposed for crops not listed on the label.

### RECOMMENDATIONS

Sufficient data have been submitted by the registrant to support an exemption from the requirement of a residue tolerance for methyl anthranilate (MA), the ai in the formulation Bird Shield® Repellent Concentrate, for use on corn, sunflowers, and apples (as intended for human consumption). Consumption of animal feedstuffs arising from these treated crops is prohibited.

MA is GRAS under 21 CFR 182.60 (food flavoring agent, perfume component). The

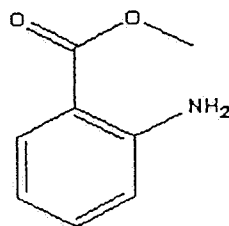
formulation inerts, as listed on the submitted CSF, are reasonably similar to chemicals exempt from tolerances under 40 CFR 180.1001(c), and permitted for direct addition to food for human consumption under 21 CFR 172. [The formulation product label states that it is comprised of all food-grade ingredients.] MA is already exempt from the requirement for a tolerance on blueberries, cherries, and grapes (40 CFR 180.1143), based on a theoretical maximal concentration of 35 ppm MA on cherries, which is comparable to the 33 ppm present naturally in concord grapes (see 1/3/95 memo of G.J. Herndon, PP#2E4071, DP Barcode D209599). The presently submitted field trials with sweet corn (kernels), sunflowers (seeds), and apples yielded MA residues < 33 ppm, even when taking into account the 4.5X and 14X maximum theoretical concentration factors for processed commodities of sunflower seeds and apples, respectively. Therefore, by analogy, sweet corn kernels, sunflower seeds, and apples should also be exempt from the requirement of a tolerance for MA, as formulated in Bird Shield® Repellent. However, because no studies were conducted on residue levels in animal feedstuffs which are associated with these crops, and no animal feeding or metabolism studies were conducted, feeding animals crop commodities treated with MA is prohibited.

The registrant needs to submit a petition and label for sweet corn, sunflowers, and apples that specifies a use pattern consistent with the conducted field trials. Additionally, the label needs to prohibit livestock grazing and the use of these crops as animal feedstuffs, and to impose a plantback interval of 12 months for crops not listed on the label. If these restrictions are unenforceable or if the registrant wishes to omit the restrictions and/or to obtain the exemption from a residue tolerance for other crops, additional field trials, processing, animal feeding and/or metabolism studies are required.

### DETAILED CONSIDERATIONS

#### OPPTS 830 Series GLNs: Product Properties

The chemical structure of MA is as follows:



Product chemistry data for methyl anthranilate were neither submitted nor addressed by the registrant. MA is a biochemical pesticide that acts to repel certain bird species without toxic effects, and is found naturally in foods such as concord grapes (a level of 33 ppm is reported in the published literature). Bird Shield Repellent is a liquid concentrate end-use product (EPA Reg. No. 66550-R) that contains, based on a CSF dated

6/24/99, 26.41% w/w MA (CAS No. 134-20-3) as the ai and the inert ingredients (1) [REDACTED] (CAS No. - none) (2) [REDACTED] [the compound name is misspelled on the CSF], (no CAS No. given) and (3) [REDACTED]. The Bird Shield Repellent Concentrate product label states that the product contains 26.4% w/w ai, is 2.29 lbs ai/gal, and is EPA, Reg. No. 66550-1). [In the submitted residue studies, the EPA Reg. No. of the formulation is given alternatively as 66550-EUP-1.]

OPPTS GLN 860.1200: Proposed Uses

The registrant provided neither a petition (Section B) nor a product label for use of Bird Shield® Repellent (26.4% ai by weight; 9.17 pounds of ai per gallon for the pure active ingredient) on sweet corn, sunflowers, or apples. The formulation is intended to repel/discourage various bird species from feeding on the crops or from using the turf and water bodies. A product label was provided for use on cherries, blueberries, grapes, turf, and non-fishbearing bodies of water. Use directions given for blueberries, cherries, and table grapes state that 1 part repellent is to be mixed with 99 parts water (v/v) and applied at a rate of 0.5-2.7 gallons (1.15-6.18 lb ai) per acre, but a seasonal total was not stated. Aerial application is prohibited in New York. Application is to begin when fruit begins to ripen or birds begin feeding on crop, and is to continue every 6 to 8 days or when odor cannot be detected (reapply if heavy rains wet surfaces within 24 hours after application). All fruit and foliage is to be thoroughly wetted until solution runs off surfaces. Harvest 6 to 8 days after last treatment, or after all odor of the product has dissipated, whichever occurs last.

*Sweet corn.* In the submitted field trial, sweet corn received two applications of 0.286 lb ai/A repellent (0.572 lb ai/A/season; 1X recommended label application rate). Application was by fixed wing aircraft at 5 gallons of tank mix per acre. The first application was 10 days prior to the anticipated harvest and the second application was 5 days after the first (5-day PHI).

*Sunflowers.* The repellent treatment (1X recommended label application rate) of sunflowers consisted of two applications of 0.286 lb ai/A (0.572 lb ai/A/season) by fixed wing aircraft at 5 gallons of tank mix per acre. The first applications was 14 days prior to the anticipated harvest and the second application was 7 days prior to harvest (7-day PHI).

*Apples.* Apple trees received one application of 1.145 lb ai/A as the 1X recommended label treatment rate. Application was in 100 gallons of water using a Rears air blast pull sprayer, and occurred when the fruit was mature and birds had begun to feed on it, 14 days prior to the anticipated harvest.

The registrant needs to submit a petition and label for sweet corn, sunflowers, and apples that specifies a use pattern consistent with the conducted field trials, that prohibits livestock grazing and the use of these crops as animal feedstuffs, and that imposes a plantback interval of 12 months for crops not listed on the label. Additionally, no information was provided by the registrant of their intent to restrict aerial application in New York for sweet corn, sunflowers, and

apples, as was done on the label previously submitted for cherries, etc. This issue needs to be addressed in the future label for corn, sunflowers, and apples.

#### OPPTS GLN 860.1300: Nature of the Residue - Plants

The metabolism of MA in plants and the nature of the residue in corn, sunflower seeds, and apples was not addressed by the registrant. All reported crop residues were for the parent compound.

#### OPPTS GLN 860.1300: Nature of the Residue - Animals

The nature of the residue in animals was not addressed by the registrant. This guideline is potentially relevant to this petition because sweet corn, sunflowers, and apples each give rise to animal feedstuffs.

#### OPPTS GLN 860.1340: Analytical Methods - Plants

##### Method used by Bird Shield Repellent Corporation for corn, sunflowers, and apples

The petitioner submitted a method for MA residue analysis on sweet corn, sunflowers, and apples that was conducted by the Laboratory for Bioanalysis and Biotechnology, Unit 2 (LBB2) at Washington State University, Pullman, WA. The study results are described in:

MRID 45065101. Askham, L.R. (1999) Method for determining the recovery of methyl anthranilate, formulated as Bird Shield Repellent Concentrate, from corn, sunflowers, and apples. Bird Shield Repellent Corporation. P.O. Box 785, Pullman, WA 99163. Study No. BSRC990401, December 15, 1999. Unpublished.

Sample analysis was by a gas chromatography/mass spectrometry (GC/MS) system consisting of a Hewlett-Packard (HP) 5890 Series II gas chromatograph with a HP 5989A mass spec. engine. The GC was fitted with a J & W Scientific DB-35MS capillary column (0.25mm x 15m). A 1  $\mu$ L sample was injected at a flow rate of 1.0 mL/min onto the GC column. The MS setting was 50-500 scan mode or 92, 119, and 152 m/z SIM mode. Test sample recoveries were determined by comparison to a linear regression of serially diluted analytic standards (0.025, 0.25, 2.5 and 25 ppm). The time between sample application (or sample harvest) was not provided, although it was stated that the experimental phase of the study was conducted from 7/13/99 - 12/30/99.

The glass surface recovery of MA was examined by drying 20  $\mu$ L MA (0.1 mg) for 2.5-10 minutes under ambient conditions, washing it off with methanol, and measuring the recovery. The recovery ranged from 67-94%, irrespective of drying time (mean  $\pm$  SD was 81  $\pm$  14).

*Sweet corn.* The petitioner examined recoveries of MA applied directly to the surface of corn kernels on or off the cob, as measured in subsequent surface washes with methanol (i.e.

“irrigation”), as well as the recovery of MA from spiked macerated corn samples. Recoveries were the poorest from the macerated corn samples and were the best from individually treated off-the-cob kernels, as described below. Neither a LOQ nor a LOD were identified.

Macerated corn samples were prepared by blending 50 g corn removed from ears in a 10-speed Osterizer blender for 2 minutes with 50 mL methanol (spiked with 1.00 mg MA diluted in methanol, if needed). The extract was filtered through 4 layers of cheesecloth, centrifuged in a microfuge for 2 minutes, and a 1.5 mL aliquot was filtered through a 0.2 micron millex-GP Millipore Luer Lock filter and placed into a glass GC sampling vial prior to analysis by GC/MS. Recoveries of duplicate analyses of a 20.0 ng (nominal) spiked sample in the scan and SIM modes ranged from 20-27% (ratio of SIM/scan areas = 21), as shown in Table 1.

The recovery of MA (aqueous dilution of the Bird Shield Concentrate formulation) was examined in fresh ears of corn (with silk and husks removed) to which 0.1 mg (20  $\mu$ L) was applied on the surface center. The MA was irrigated (i.e., washed) off the corn with 50 mL methanol, 50 mL ethyl acetate, or twice with 10 mL methanol. An aliquot of 1.5 mL was filtered through a 0.2 micron millex-GP Millipore Luer Lock filter and placed into a glass GC sampling vial prior to analysis by GC/MS. A sample of MA (0.1 mg in 20  $\mu$ L, diluted in 50 mL methanol) was also evaluated for reference. The analytic recoveries of these samples ranged from 26-42%, as shown in Table 1. The recoveries for a second sample treated with two 10 mL methanol washes was reported as 22% in the scan mode and 120% in the SIM mode in the discussion of the study results on p. 15 of MRID 45065101, although these results were not presented with the others in Table 5 on p. 11 of MRID 45065101. No clear differences were seen between the different sample irrigation methods or between methanol and ethyl acetate washes.



Table 1: Recovery of methyl anthranilate (MA) from fresh corn kernels (on the cob or removed and macerated) to which diluted Bird Shield Repellent Concentrate or MA analytic standard were applied.			
Sample	Area; % Recovery		Ratio of SIM/Scan areas
	Scan mode	SIM mode	
<b>MA (analytic standard) applied to macerated corn kernels</b>			
Macerated corn spiked with 1.00 mg MA	–	6097770; 20%	–
Repeat of above	390900 (27%)	8064060; 25%	21
<b>MA (diluted formulation) applied to corn kernels attached to the cob</b>			
Untreated macerated control	0	0	–
0.1 mg MA only	77150 (100%)	2137260 (100%)	28
0.1 mg MA on corn washed w/ 50 mL methanol	28340; 37%	558350; 26%	20
0.1 mg MA on corn washed w/ 50 mL ethyl acetate	32660; 42%	689110; 32%	21
0.1 mg MA on corn washed w/ 10 + 10 mL methanol	22% <sup>1</sup>	120% <sup>1</sup>	NA
Repeat of above	68900 <sup>2</sup> ; 36%	–	

Data from pp. 9 and 11, MRID 45065101.

<sup>1</sup>The recoveries were presented on p. 15 in the results discussion but were not presented in Table 5 on p. 11 of 45065101. The actual areas of the signals were not given (NA).

<sup>2</sup>Adjusted for lower volume of washes.

Another analysis compared residue recoveries between individual corn kernels on or off the cob that were treated with MA (diluted formulation) and irrigated with methanol. Low and high standards (0.05 or 0.10 mg MA in 25 mL untreated corn methanol wash) were used for reference. Three samples were prepared each for (a) untreated whole corn on the cob washes (one 5 mL and three 10 mL methanol washes of 15 kernels attached to cob (solution was decanted into a 25 mL glass flask and 1.5 mL was filtered with a 0.2 micron Millex-GP Millipore Luer Lock filter into 1.5 mL glass GC sampling vials); (b) as (a) but three kernels on the cob were treated with 20 µL MA (0.1 mg) and the inclusive surrounding area of 15 kernels was irrigated with methanol; (c) single kernels off the cob were treated individually with 20 µL MA (0.1 mg) and irrigated and filtered as in (a). Results, as shown in Table 2, indicate that recoveries from individual off-the-cob kernels (63.31-98.53%) were somewhat greater than those from on-the-cob samples (46.43-72.27%). Note that the reviewer found several inconsistencies between information presented in the study tables and the study text (e.g. results summary on p. 15 and results presented in Tables on pp. 9 and 11 of MRID 45065101), as well some miscalculations (e.g. p. 12 of MRID 45065101).

**Table 2: Recovery of methyl anthranilate (MA) from fresh corn kernels (on or off the cob) to which diluted Bird Shield Repellent Concentrate was applied.**

Sample	Uncorrected area (mean of 3 replicates)	Area; % Corrected recovery <sup>1</sup>	
		Comparison to low standard	Comparison to high standard
Low standard (0.05 mg in untreated corn wash)	442240.00 ± 13562.00	(100%)	—
High standard (0.10 mg in untreated corn wash)	681180.00 ± 34606.62	—	(100%)
Untreated corn washes <sup>2</sup>	12,766.67 ± 4086.19	—	—
Kernel off cob application	435935.56 ± 161903.40	98.53%	63.31%
Kernel on cob application	323135.56 ± 131821.98	72.27%	46.43%

Data from p. 12, MRID 45065101.

<sup>1</sup>Percent recovery = [corrected (for untreated sample) area ÷ corrected (for untreated sample) area of the low or high standards] x 100.

<sup>2</sup>The text (p. 12, MRID 45065101) states that the mean area of the untreated samples is 17,022.22, but the reviewer calculated the mean (of 11,850.00, 17,233.33, and 9,216.67) to be 12,766.67 and used this value to correct the areas of the MA (treated corn and spike standard) samples.

**Sunflowers.** Residue recovery was evaluated in sunflower seed heads that were treated directly with 20 µL (0.1 mg diluted formulation) MA. The seed heads were irrigated with 20 mL methanol and a 1.5 mL aliquot was filtered through a 0.2 micron millex-GP Millipore Luer Lock filter and placed into a glass GC sampling vial prior to analysis by GC/MS. Two controls were also assayed: a sample of MA (0.1 mg in 20 µL water, diluted in 25 mL “mix 3”, which was not defined but was probably a methanol wash of untreated sunflower seeds) as a spike standard, and an untreated control, which was an untreated sunflower head that was irrigated and filtered. The mean corrected (for untreated control) analytic recovery of the treated sunflower samples was 36.78%, as shown in Table 3.

**Table 3: Recovery of methyl anthranilate (MA) from sunflower seeds (on the heads) to which diluted Bird Shield Repellent Concentrate was applied.**

Sample	Area	Sample concentration (ppm)		Recovery (%) (mean ± SD)
		Uncorrected	Corrected	
Untreated control	87070	0.125	0.001	0.00
Spike standard (0.10 mg)	71682680	9.246	9.121	91.21
Treated samples (0.10 mg)	366850	0.161	0.036	0.039 <sup>1</sup>
	26224910	3.455	3.330	36.51
	26587210	3.502	3.378	37.04
				(36.78 ± 0.37)

Data from p. 13, MRID 45065101.

<sup>1</sup>This sample was labeled as being defective and was not used to calculate the recovery mean.

*Apples.* Recovery of residues from apples treated directly with 5 µL (0.1 mg in water) MA were determined by GS/MS. The samples analyzed were (a) an untreated control sample (aliquot of untreated apple surface 50 mL methanol wash, of which 1.5 mL was transferred into a glass GC sampling vial); (b) a spiked standard (0.025 mg MA diluted in 50 mL methanol and placed in 1.5 mL glass GC sampling vial); and (c) treated samples (as (a) but 0.025 mg MA was placed on the apple surface prior to irrigation). The mean corrected (for untreated control) recovery of the treated apple samples was 72.05%, as shown in Table 4.

Sample	Area	Sample concentration (ppm)		Recovery (%) (mean ± SD)
		Uncorrected	Corrected -	
Untreated control	36360	0.054	0.003	0.00
Spike standard (0.025 mg)	1608220	0.289	0.235	100.00
Treated samples (0.025 mg)	1039110	0.204	0.150	65.66
	1254450	0.236	0.182	78.44 (72.05 ± 9.04)

Data from p. 13, MRID 45065101.

Method validation for analysis of residues on corn and sunflowers

The analytical method for MA was validated for sweet corn and sunflowers by another laboratory, Genesis Laboratories, Inc., Wellington, CO. The study results are described in:

MRID 45065102. Harkrader, R.J. (2000) Method for the analysis of Bird Shield Repellent Concentrate on corn and sunflowers. Genesis Laboratories, Inc., 10122 N.E. Frontage Road, Wellington, CO 80549. Study No. 99048, March 3, 2000. Unpublished.

Sweet corn samples were prepared by applying 20 µL (56.2 ng) diluted Bird Shield Repellent Concentrate to a single kernel of corn (6 kernels total) removed from fresh corn ears (locally purchased), and washing the kernel with methanol (10 mL total). Sunflower seed samples were prepared by removing a single seed from whole sunflowers (supplied by the Bird Shield Repellent Corporation and kept frozen until use) and applying to it 20 µL (56.2 ng) diluted Bird Shield Repellent Concentrate, followed by a 9 mL methanol wash (10 mL total); a total of 6 seeds were treated. Untreated control sweet corn and sunflower seeds (blanks) were prepared similarly to the treated samples except no test compound was applied. The length of time between either MA application or sample collection and sample analysis was not provided; the experimental phase of the study occurred 12/29/99 - 3/1/00.

Sample analysis was by GC/MS using a HP 5890 Series II gas chromatograph with a HP 5972 mass selective detector. The GC column was a Restek Corporation XTI-5 (bonded 5% phenyl)

capillary column (0.25 mm x 30 m); 1  $\mu$ L samples were injected at a flow rate of 1.0 mL/min. The MS setting was 45-500 scan mode. The standard deviation of six replicate injections of the 1.01  $\mu$ g/mL standard (peak areas of 689-1128; mean  $\pm$  SD is  $933 \pm 194$ ) and a regression curve derived from the response of analytic standards (1.01-101  $\mu$ g/mL) were used to determine the LOD and LOQ as 3X the SD (i.e.  $3 \times 194 = 582$ , which corresponded to 2.09  $\mu$ g/mL) and 10X the SD (i.e.  $10 \times 194 = 1940$ , which corresponded to 2.91  $\mu$ g/mL), respectively. MA concentration of the individual treated corn and sunflower seed samples were determined by comparison of their response to that of 5.05  $\mu$ g/mL analytic MA standards run before and after each sample. Residue recoveries were calculated by comparison of the MA response from treated crop samples to a standard curve obtained using analytic standards (1.01-101  $\mu$ g/mL).

The MA concentrations in the Bird Shield Repellent formulation experimentally measured by Genesis Laboratories were greater than predicted by the product label (10.2 and 9.7  $\mu$ g/mL MA in corn and sunflower assays vs. 5.6  $\mu$ g/mL theoretical). Based on the adjusted formulation concentration, residue recoveries of the corn and sunflower seed samples ( $\pm$  SD) were, respectively  $80.0 \pm 25.7\%$  and  $101.2 \pm 15.1\%$ , as shown in Table 5. However, it appeared to the reviewer that an error was made in determining the MA concentration of the formulation used for the corn samples, and using the provided sample calculation (p. 12, MRID 45065102) yields 6.94  $\mu$ g/mL and not 10.2  $\mu$ g/mL for the MA concentration. Based on a formulation concentration of 6.94  $\mu$ g/mL, the percent recoveries for the corn samples ranged from 68-167% (mean  $\pm$  SD of  $117 \pm 38$ ). The recoveries of MA in both corn and sunflower samples was associated with a great deal of uncertainty, however, since there was a 2-fold range of response (i.e. peak area) associated with the standard 5.05  $\mu$ g/mL solution used to determine individual sample concentrations.

Table 5: Method validation of the recovery of methyl anthranilate (MA) from sweet corn and sunflower seeds to the surface of which diluted Bird Shield Repellent Concentrate was applied.				
Sample	MA area (response)	Sample concentration (µg/mL)		Recovery (%) (mean ± SD)
		Applied	Recovered	
<b>Sweet corn</b>				
MA-treated sweet corn	14414	10.2	11.6	114
	11366	10.2	8.6	84.3
	15647	10.2	8.7	85.3
	10634	10.2	5.5	53.9
	8927	10.2	4.7	46.1
	16075	10.2	9.8	96.1
Untreated sweet corn (blank)	0, 0, 0	0, 0, 0	0, 0, 0	- 0
<b>Sunflower seeds</b>				
MA-treated sunflower seeds	17020	9.7	10.7	110
	18239	9.7	12.1	125
	14849	9.7	8.2	84.5
	16821	9.7	8.7	89.7
	17381	9.7	9.0	92.8
	18150	9.7	10.2	105
Untreated sunflower seed (blank)	0, 0, 0	0, 0, 0	0, 0, 0	- 0

Data from pp. 13-14, MRID 45065102.

**Conclusions:** A wide range of recoveries were obtained for surface-treated sweet corn using the various methods submitted by the petitioner. MA recovery on macerated corn samples was 20-27% (depending on the MS detection mode), for surface-washed corn samples was 26-37% in one experiment (one questionable sample had recovery of 22 and 120% for scan and SIM modes, respectively) and 46-72% in a second experiment. Individually treated kernels (off-the-cob) had recoveries of 63.31-98.53%. Method validation by Genesis Laboratories, Inc. using off-the-cob corn kernels yielded recoveries of 46.1-114%, although the reviewer obtained 68-167% by an alternate calculation. **For sunflowers**, the mean recovery of the treated samples (seeds on whole sunflower heads were treated and washed) was 36.51-37.04%, but the validation laboratory obtained much greater recoveries (84.5-125%) when the MA was applied to individual sunflower seeds. **For apples**, the mean MA recovery was 72.05%; validation was not performed. The registrant noted (MRID 4506501) that MA interacts with active sites on the GC column (DB-35MS), and repeated use causes the columns to degrade quickly. This may partially account for the variable recoveries of this analytic method. Due to this variability, the method would not be adequate as an enforcement method, although per OPPTS guideline 860.1550, p. 2, an analytic method for enforcement purposes is rarely needed for pesticides that are exempt from tolerances.

#### OPPTS GLN 860.1340: Analytical Methods - Animals

An analytical method for animal commodities was not addressed by the registrant. This guideline is potentially relevant to this petition because sweet corn, sunflowers, and apples each give rise to animal feedstuffs.

#### OPPTS GLN 860.1360: Multiresidue Method

The petitioner did not address the potential of the recovery of MA (or any metabolites) by any FDA Multiresidue Methods.

#### OPPTS GLN 860.1380: Storage Stability Data -Plants

Storage stability studies were not conducted for the corn, sunflower, or apple samples. For all three crops, the MA was applied to the crop surface and was subsequently washed off with methanol (and filtered for corn and some sunflower samples) to evaluate the MA recoveries. The length of time between application and the washes was not stated, although GC/MS analysis of sample MA residues took place within approximately 1-3 months of sample application.

#### OPPTS GLN 860.1380: Storage Stability Data - Animal Commodities

Storage stability of animal commodities was not addressed by the registrant. This guideline is potentially relevant to this petition because sweet corn, sunflowers, and apples each give rise to animal feedstuffs.

#### OPPTS GLN 860.1500: Crop Field Trials

##### Sweet corn

Bird Shield Repellent Corporation conducted one field trial in 1999 to support an exemption from the requirement of a residue tolerance for MA (formulated as Bird Shield® Repellent Concentrate, 26.4% w/w ai, EPA Reg. No. 66550-EUP-001) for use on sweet corn. The results are reported in:

MRID 45065103. Askham, L.R. (1999) Residue chemistry: crop field trials. Sweet corn. Bird Shield Repellent Corporation. P.O. Box 785, Pullman, WA 99163. Study No. BSRC990612, December 15, 1999. Unpublished.

The field trial was conducted near Grand Junction, CO (EPA crop region 9), and consisted of two control and 12 treated plots. Six of the sites received two applications of 0.286 lb ai/A repellent (0.572 lb ai/A/season; 1X recommended label application rate) and the other 6 received two applications of 0.573 lb ai/A repellent (1.144 lb ai/A/season; 2X label rate). The sweet corn was treated by fixed wing aircraft at 5 gallons of tank mix per acre. The first application was 10 days

prior to the anticipated harvest and the second application was 5 days after the first (5-day PHI). Two samples (3 ears of corn per sample) were collected at each of the 14 test sites at both one hour and 10 days after the first application and were placed in cold storage until sample analysis. The husks and silk were removed from the ears of corn prior to analysis for MA (in filtered methanol washes of the corn surface). The period of time between sample application (or harvest) and sample analysis was not stated; sample application was 8/12/99 and 8/17/99, sample collection was 8/12/99 (1-hour) and 8/22/99 (10-day), and the GC/MS chromatograms were dated 11/29/99-12/01/99. Sample analysis was by a GC/MS method and was conducted by LBB2 Analytical Services Laboratory at Washington State University, which provided sufficient sample chromatograms and calibration information.

MA residue levels in all samples were adjusted for 46% method recovery; treated samples were also corrected for background levels found in the controls, as shown in Table 6. For the 1 hour post application samples, residue levels for the 1X and 2X treatments ranged from -0.084 to 5.579 ppm and -0.068 to 0.429 ppm, respectively. For the 10-day samples, residue levels were below control levels for all the 1X treatment samples and for all but two 2X treatment samples. Neither a LOQ nor a LOD (limit of detection) were identified. Table 6 summarizes the residue levels of MA on sweet corn.

Table 6: Residues of methyl anthranilate (MA) on sweet corn following two applications of Bird Shield® Repellent with a re-treatment interval of 5 days and a PHI of 5 days (study conducted near Grand Junction, CO, EPA crop region 9).

Sample	Application rate per treatment (lb ai/A)	Corrected residue levels (ppm) <sup>1</sup>	
		1 hr. after 1 <sup>st</sup> application (Mean ± SD)	10 days after 1 <sup>st</sup> application (Mean ± SD)
Control samples: 1.1.1, 1.2.1 2.1.1, 2.2.1	0	(0.092±0.098) 0.235, 0.043 0.074, 0.017	(0.203 ± 0.036) 1.704 <sup>2</sup> , 0.198 0.241, 0.170
Treated samples (1X): 7.1.1, 7.2.1 8.1.1, 8.2.1 9.1.1, 9.2.1 10.1.1, 10.2.1 11.1.1, 11.2.1 12.1.1, 12.2.1	0.286 (total of 0.572)	0.108, -0.071 -0.051, -0.066 -0.014, 0.642 5.579, -0.071 0.134, -0.008 0.108, -0.084	-0.090, -0.105 -0.029, -0.094 -0.125, -0.114 -0.125, -0.129 -0.120, -0.112 -0.116, -0.086
Treated samples (2X): 1.1.1, 1.2.1 2.1.1, 2.2.1 3.1.1, 3.2.1 4.1.1, 4.2.1 5.1.1, 5.2.1 6.1.1, 6.2.1	0.573 (total of 1.144)	0.429, -0.068 0.001, 0.192 0.036, -0.038 0.264, 0.358 0.066, 0.055 0.418, -0.064	0.388, -0.009 -0.181, 0.001 -0.046, 0.008, -0.040, -0.020 -0.122, -0.107 -0.125, -0.120

Data from p.13 of MRID 45065103.

A limit of quantitation (LOQ) for sweet corn was not identified.

<sup>1</sup>For treated samples, background residue levels (i.e. mean of untreated controls) were subtracted from the detected concentration and the resulting values were corrected for 46% method recovery.

<sup>2</sup>Number rejected because it falls outside of Q test for 90% acceptance.



## Sunflowers

Bird Shield Repellent Corporation conducted one field trial in 1999 to support an exemption from the requirement of a residue tolerance for MA (formulated as Bird Shield® Repellent Concentrate, 26.4% w/w ai, EPA Reg. No. 66550-EUP-001) for use on sunflowers. The results are reported in:

MRID 45065104. Askham, L.R. (1999) Residue chemistry: crop field trials. Sunflowers. Bird Shield Repellent Corporation. P.O. Box 785, Pullman, WA 99163. Study No. BSRC980401, December 15, 1999. Unpublished.

The field trial was conducted near Fargo, ND (EPA crop region 5), and consisted of two control and 8 treated plots. Four of the treated sites received two applications of 0.286 lb ai/A repellent (0.572 lb ai/A/season; 1X recommended label rate) and the other 4 received two applications of 0.573 lb ai/A repellent (1.144 lb ai/A/season; 2X label rate). The sunflowers were treated by fixed wing aircraft at 5 gallons of tank mix per acre. The first application was 14 days prior to the anticipated harvest and the second application was 7 days prior to harvest (7-day PHI). Two samples (3 sunflowers per sample) were collected at each test site one hour and again 14 days after the first application and were placed in cold storage 1-4 hours after collection. The outer layers of leaves and the pistils were removed to expose all the seeds of each flower. Samples were analyzed for MA by a GC/MS method; the analyzed samples consisted of filtered methanol washes of the sunflower seed surfaces. The time between either sample application or collection and sample analysis was not specified; treatment occurred 10/17/99 and 10/24/99, harvest was 10/17/99 and 10/24/99, and the GC/MS chromatograms were dated 12/1/99 and 12/3/99. Sample analysis was conducted by LBB2 Analytical Services Laboratory at Washington State University, which provided sufficient sample chromatograms and calibration information.

MA residue levels in all samples were adjusted for 36.78% method recovery; treated samples were also corrected for background levels found in the controls. Most of the samples had residue levels below those of controls: the 1-hour 1X and 2X treatment samples had residues of -0.053 to 0.058 ppm and -0.048 to 0.099 ppm, respectively, whereas the 14-day 1X and 2X samples had residues of -0.140 to 0.170 ppm and -0.091 to 2.002 ppm, respectively. Neither a LOQ nor a LOD were identified. Table 7 summarizes the residue levels of MA on sunflowers.

Table 7: Residues of methyl anthranilate (MA) on sunflowers following two applications of Bird Shield® Repellent with a re-treatment interval of 7 days and a PHI of 7 days (study conducted near Fargo, ND, EPA crop region 5).			
Sample	Application rate (lb ai/A)	Corrected residue levels (ppm) <sup>1</sup>	
		1 hr. after 1 <sup>st</sup> application (Mean ± SD)	14 days after 1 <sup>st</sup> application (Mean ± SD)
Control samples: 1.1.1, 1.2.1 2.1.1, 2.2.1	0 0	(0.083±0.049) 0.117, 0.038 0.133, 0.044	(0.137 ± 0.038) 0.098, 0.158 0.114, 0.179
Treated samples (1X): 1.1.1, 1.2.1 2.1.1, 2.2.1 3.1.1, 3.2.1 4.1.1, 4.2.1	0.286 (total of 0.572)	0.058, -0.020 0.020, 0.004 0.010, -0.007 -0.053, -0.012	-0.012, 0.124 0.170, -0.042 -0.058, 0.004 -0.140, 0.015
Treated samples (2X): 5.1.1, 5.2.1 6.1.1, 6.2.1 7.1.1, 7.2.1 8.1.1, 8.2.1	0.573 (total of 1.144)	0.061, -0.023 0.099, 0.094 -0.042, -0.048 -0.031, -0.020	2.002, -0.029 -0.012, -0.050 -0.050, 0.012, -0.091, 0.080

Data from p.13 of MRID 45065104.

A limit of quantitation (LOQ) for sunflowers was not identified.

<sup>1</sup>For treated samples, background (control) residue levels were subtracted from the detected concentration and the resulting values were corrected for 36.78% method recovery.

## Apples

Bird Shield Repellent Corporation conducted one field trial in 1999 to support an exemption from the requirement of a residue tolerance for MA (formulated as Bird Shield® Repellent Concentrate, 26.4% w/w ai, EPA Reg. No. 66550-EUP-001) for use on apples. The results are reported in:

MRID 45065105. Askham, L.R. (1999) Residue chemistry: crop field trials. Apples. Bird Shield Repellent Corporation. P.O. Box 785, Pullman, WA 99163. Study No. BSRC991017, December 15, 1999. Unpublished.

The field trial was conducted in Orondo, WA (EPA crop region 11), and consisted of three control and 8 treated plots. Four of the treated sites received one application of 1.145 lb ai/A (1X recommended label application rate) and the other 4 received one application of 2.290 ai/A (2X label rate). The repellent was applied in 100 gallons of solution using a Rears air blast pull sprayer. The application was made when the fruit was mature and birds had begun to feed on it, 14 days prior to the anticipated harvest. One sample (3 apples per sample) was collected at each test plot 5 minutes after treatment and again 14 days after treatment; the initial collection was reported alternately as being 1 hour after treatment. Collected samples were placed on ice for

transport to the analytical facility, and were analyzed for MA within 18 hours of collection. The analyzed samples consisted of filtered methanol washes of the apple surface. The time period between either sample application or collection and sample analysis was not specified; crops were treated 10/17/99 and harvest took place 10/17/99 and 10/31/99, and the GC/MS chromatograms were dated 11/16/99 and 11/17/99 (controls 12/5/99). Sample analysis was conducted by a GC/MS method by LBB2 Analytical Services Laboratory at Washington State University, which provided sufficient sample chromatograms and calibration information.

MA residue levels in all samples were adjusted for 72.05% method recovery; treated samples were also corrected for background levels found in the controls. The 5 minute (or 1 hour?) 1X and 2X treatment samples had residues of 0.031-1.507 ppm and 1.217-3.348 ppm, respectively, whereas the post-14 day 1X and 2X samples were 0.026-0.115 ppm and -0.006 to -0.024 ppm, respectively. Neither a LOQ nor a LOD (limit of detection) were identified. Table 8 summarizes the residue levels of MA on apples.

Table 8: Residues of methyl anthranilate (MA) on apples following one application of Bird Shield® Repellent with a PHI of 14 days (study conducted in Orondo, Wa, EPA crop region 11).			
Sample	Application rate (lb ai/A)	Corrected residue levels (ppm) <sup>1</sup>	
		5 minutes (1 hr.?) <sup>2</sup> after application (Mean ± SD)	14 days after application (Mean ± SD)
Control samples: 1.2.1, 1.1.1, 2.1.1	0	(0.092±0.125) 0.262, 0.087, 0.019	(0.016 ± 0.000) 0.021, 0.021, 0.021
Treated samples (1X): 2.1.1 2.2.1 2.3.1 2.4.1	1.145	0.346 1.507 0.306 0.031	0.115 0.051 0.026 0.027
Treated samples (2X): 1.1.1 1.2.1 1.3.1 1.4.1	2.29	1.223 3.348 1.217 1.261	-0.006 -0.024 -0.024 -0.024

Data from p. 12 of MRID 45065105.

A limit of quantitation (LOQ) for apples was not identified.

<sup>1</sup>For treated samples, background residue levels (i.e. mean of untreated controls) were subtracted from the detected concentration and the resulting values were corrected for 72.05% method recovery.

<sup>2</sup>Collection time was given in the study text alternatively as 5 minutes and 1 hour after treatment.

**Conclusions:** MA residue levels on sweet corn, sunflower seeds, and apples that received 1X the label application rate were low at anticipated harvest. Maximum residue levels for sweet corn were below controls and were 0.170 and 0.115 ppm for sunflower seeds and apples, respectively. Residue levels were greater 1 hour after treatment (or possibly 5 minutes after treatment for

apples); maximum levels of 5.579, 0.058, and 1.507 ppm were found on sweet corn, sunflower seeds, and apples, respectively. This demonstrates that residues declined with time for all three crops, as is anticipated based on the volatility of the compound and its reported instability in sunlight.

For all three crops, sampling after either time period yielded residue levels much lower than the 35 ppm calculated theoretical maximal concentration of MA on cherries, which was determined immediately after the cherries were dipped in MA solution (see 1/3/95 memo of G.J. Herndon, PP#2E4071, DP Barcode D209599). Cherries (and blueberries and grapes), however, do not have any processed commodities and possible concentration of the residue was not a factor in estimating human dietary levels. Sunflower seeds and apples, however, each have processed commodities, with maximum concentration factors of 4.5X and 14X, respectively. Taking these into account, the maximum residue levels for sunflower processed commodities that received 1X the proposed application rate would be 0.086 ppm and 0.29 ppm for the 1-hour and 14-day sample collection (using the highest average field trial (HAFT) of 0.019 ppm and 0.064 ppm, respectively). For apples, the maximum residues in processed commodities (1X application) would be 21.1 ppm and 1.6 ppm for the 5 minute (or 1 hour?) and 14-day samples, respectively. Thus, the field trial data indicate that the human MA dietary burden contributed by sweet corn, sunflower seeds, and apples and their processed commodities would not exceed that contributed by cherries, which were granted an exemption from the requirement of a tolerance, and therefore, by analogy, sweet corn, sunflower seeds, and apples should also be exempt from the requirement of a tolerance for MA. This exemption, however, is *limited* to human consumption of the sweet corn kernels, sunflower seeds, and apples; consumption of animal feedstuffs arising from these treated crops is prohibited due to a lack of relevant crop residue data and animal metabolism and feeding studies.

#### OPPTS GLN 860.1520: Processed Food/Feed

The potential of MA residues in processed commodities was not addressed by the registrant. This guideline is potentially relevant to this petition because sunflowers and apples yield processed commodities of regulatory interest: sunflower meal and oil for sunflowers, and wet apple pomace and apple juice for apples.

#### OPPTS GLN 860.1850 and 860.1900: Confined/Field Accumulation in Rotational Crops

The registrant did not address the potential for confined/field accumulation of MA residues in rotational crops. The submitted Bird Shield Repellent Concentrate label for cherries, blueberries, and grapes provides no guidelines for rotational crops (possibly because these crops are perennials). Since sweet corn, sunflowers, and apples are annuals, plantback guidelines are needed and due to the absence of other data, the default plantback of 12 months needs to be imposed for crops not listed on the label.