

US EPA ARCHIVE DOCUMENT



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RAB2/HED (7509C)

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RAB2/HED (7509C) *R. Loranger*

This DER was originally prepared under contract by Dynamac Corporation (1910 Sedwick Road, Building 100, Suite B; Durham, NC 27713). It has been reviewed by HED and revised to reflect current OPP policies.

### STUDY REPORT

MRID #46357302. Sandra J. Carringer (2003) *Magnitude of the Residue of TM-444 and its Metabolite in Potato Raw Agricultural and Processed Commodities*. Study #TCI-03-075. Unpublished study prepared by Arvesta Corporation, Morse Laboratories Incorporated, and The National Food Laboratory Incorporated. 420 pages. {OPPTS Residue Chemistry Test Guideline 860.1500}

### EXECUTIVE SUMMARY

Fifteen potato field trials were conducted during 2003 throughout the US. At each trial site, side-by-side tests were conducted comparing the use of clothianidin as either a single in-furrow application at planting (to soil) of a water-soluble granule (WSG) formulation, or as three foliar broadcast applications of a water-dispersible granule (WDG) formulation. The soil application (Belay™ 16WSG) was made at a total rate (essentially 1X the proposed maximum seasonal use rate for soil treatment) of 0.194 to 0.207 pounds of active ingredient per acre (lb ai/A), while the three foliar broadcast applications (Clutch™ 50WDG) were made at a rate of 0.064 to 0.069 lb ai/A per application, for a total rate of 0.196 to 0.204 lb ai/A per season (roughly 1.4X the proposed maximum seasonal use rate for foliar treatment). The foliar applications were made during tuber development with re-treatment intervals (RTIs) of 6 to 8 days; no spray adjuvants were used for any of the applications at any of the field trials. Potato tubers were harvested at commercial maturity, 85 to 134 days after treatment (DAT) from the in-furrow tests, or 13 to 14 DAT from the foliar tests. Additional tuber samples from two trial sites were harvested at 121, 128, and 135 DAT (in-furrow treatment), and at 0, 21, and 28 DAT (foliar treatment) to examine residue decline. Single control and duplicate treated samples were collected from each trial. Samples were stored frozen from collection to analysis for up to 3 months, a duration supported by concurrent storage stability data for potatoes.

The LC/MS/MS methods (Morse Method #Meth-157 and #Meth-164) used to determine residues of clothianidin and its metabolite, TMG, in potatoes were adequately validated in conjunction with the field trial analyses. For both methods, residues are extracted with ACN/water/guanidine-HCl (20:80:1 vol/vol/wt), filtered, and concentrated. Residues of clothianidin and TMG are then cleaned up separately using a ChemElut™ liquid/liquid extraction (LLE) or ENVI-Carb™ solid phase extraction (SPE) columns, respectively. Residues are concentrated, reconstituted in 1% acetic acid, and analyzed by LC/MS/MS. The validated limit of quantitation (LOQ) for both analytes is 0.020 ppm in potatoes, and the limit of detection (LOD) is 0.007 ppm.

Residues of the metabolite, TMG, were non-detectable (less than 0.007 ppm) in all potato samples from all trials using either formulation. For the in-furrow applications using Belay™ 16WSG, residues of clothianidin in tubers harvested 85 to 134 DAT were less than



0.020 ppm (the LOQ) in 21 out of 30 samples, and were 0.020 to 0.033 ppm in the remaining 9 samples. For the foliar applications using Clutch™ 50WDG, residues of clothianidin in tubers harvested 13 to 14 DAT were less than 0.020 ppm (the LOQ) in 27 out of 29 samples, and were 0.020 and 0.021 ppm in the remaining 2 samples. Average residues of clothianidin were 0.014 ppm for the in-furrow applications and 0.011 ppm for the foliar applications, while HAFT residues for clothianidin were 0.031 ppm following the in-furrow application and 0.021 ppm following the foliar application. Because residues of clothianidin were less than LOQ in all samples from the two field trials having repeated sampling intervals, residue decline could not be reliably determined.

**STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS**

Under the conditions and parameters used in the study, the potato field trial residue data are classified as scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the forthcoming US EPA Residue Chemistry Summary Document (DP Barcodes D309473 and D309474).

**COMPLIANCE**

Signed and dated GLP, quality assurance, and data confidentiality statements were provided. No deviations from regulatory requirements were noted that would impact the study results or their interpretation.

**A. BACKGROUND INFORMATION**

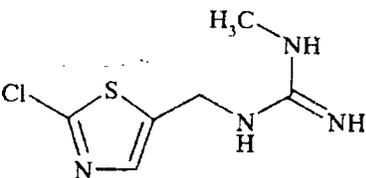
Clothianidin (also known by its development code numbers, TM-444, TI-435, or V-10066) is a systemic insecticide, belonging to the chloronicotinyl (and nitroguanidine) class of chemicals, which enters the transpiration stream through the roots and cotyledons of newly germinating seedlings and protects below- and above-ground plant parts from insect damage. It binds (via ingestion and contact routes) with the nicotinic acetylcholine receptor sites, interfering with transmission of stimuli and eventually inhibiting reproduction of the insect. Clothianidin is a major metabolite of thiamethoxam. It is currently registered (40CFR §180.586) for use on various crops.

Arvesta has submitted a petition (PP#4F6869) requesting the establishment of tolerances for residues of clothianidin in/on grape and potato commodities. The 50% ai water-dispersible granule (WDG) formulation is proposed for foliar applications to grapes and potatoes (Clutch™ 50WDG, EPA Registration #66330-40). The 16% ai water-soluble granule (WSG) formulation is proposed for soil applications to grapes and potatoes (Belay™ 16WSG, EPA Registration #66330-52).

<b>TABLE A.1 Nomenclature of Test Compound and its Metabolite.</b>	
Compound	
Empirical Formula	C <sub>6</sub> H <sub>8</sub> ClN <sub>5</sub> O <sub>2</sub> S



Clothianidin/66330-40 & 66330-52/PC Code 044309/Arvesta Corporation/66330  
 DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3  
 Crop Field Trial - Potato (Tuber)

Common Name	Clothianidin
Company Experimental Names	TM-444, T1-435, V-10066
IUPAC Name	(E)-1-(2-Chloro-1,3-thiazol-5-ylmethyl)-3-methyl-2-nitroguanidine
CAS Name	[C(E)]-N-[(2-Chloro-5-thiazolyl)methyl]-N'-methyl-N''-nitroguanidine
CAS Number	210880-92-5 (formerly 205510-53-8)
Chemical Class	Chloronicotinyl
Known Impurities of Concern	None
End-Use Product (EUP)	Clutch™ 50WDG, EPA Registration #66330-40 Belay™ 16WSG, EPA Registration #66330-52
Metabolite	
Common Name	Metabolite TMG
Company Experimental Name	TMG
CAS Name	[C(E)]-N-[(2-Chloro-5-thiazolyl)methyl]-N'-methylguanidine

Parameter	Value														
Molecular Weight	249.7														
Melting Point (°C)	176.8														
pH at 23°C	6.24 [1% solution/suspension]														
Density (g/cm <sup>3</sup> ) at 20°C	1.61 [PAI], 1.59 [TGAI]														
Water Solubility (g/L) at 20°C	0.327														
Solvent Solubility (g/L) at 25°C	<table border="0"> <tr><td>n-Heptane</td><td>&lt;0.00104</td></tr> <tr><td>Xylene</td><td>0.0128</td></tr> <tr><td>1-Octanol</td><td>0.938</td></tr> <tr><td>Dichloromethane</td><td>1.32</td></tr> <tr><td>Ethyl Acetate</td><td>2.03</td></tr> <tr><td>Methanol</td><td>6.26</td></tr> <tr><td>Acetone</td><td>15.2</td></tr> </table>	n-Heptane	<0.00104	Xylene	0.0128	1-Octanol	0.938	Dichloromethane	1.32	Ethyl Acetate	2.03	Methanol	6.26	Acetone	15.2
n-Heptane	<0.00104														
Xylene	0.0128														
1-Octanol	0.938														
Dichloromethane	1.32														
Ethyl Acetate	2.03														
Methanol	6.26														
Acetone	15.2														
Vapor Pressure (Pa) at 25°C	1.3 x 10 <sup>-10</sup>														
Dissociation Constant (pK <sub>a</sub> ) at 20°C	11.09														
Octanol/Water Partition Coefficient (Log K <sub>ow</sub> ) at 25°C	0.7														
UV/Visible Absorption Spectrum, Maximum (nm)	265.5 [acidic, neutral sol'n's]. 246.0 [basic sol'n].														

**B. EXPERIMENTAL DESIGN**

**B.1. Study Site Information**

Fifteen potato field trials were conducted during 2003 comparing an early-season soil application of clothianidin using the Belay™ 16WSG formulation to multiple late-season foliar



Clothianidin/66330-40 & 66330-52/PC Code 044309/Arvesta Corporation/66330  
 DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3  
 Crop Field Trial - Potato (Tuber)

applications using the Clutch™ 50WDG formulation. Side-by-side applications using each formulation were made at each trial site, for a total of 30 tests. For the early-season application, clothianidin (Belay™ 16WSG) was applied at a rate of 0.194 to 0.207 lb ai/A, as a single in-furrow application at planting using ground equipment. For the late-season applications, clothianidin (Clutch™ 50WDG) was applied as three broadcast foliar applications at a rate of 0.064 to 0.069 lb ai/A per application, with RTIs of 6 to 8 days, for total use rates of 0.196 to 0.204 lb ai/A per season. Foliar application were made beginning approximately one month prior to tuber maturity, during tuber development. No adjuvants were added to the spray tank at any of the field trials for either type of application.

Details on soil characteristics were not included in the report; these data should be provided for studies such as MRID #46357302, reviewed in this data evaluation record (DER), which include soil applications. A summary of weather conditions was provided for each field trial site (see Table B.1.1), including average monthly minimum and maximum temperatures and precipitation (rainfall and/or irrigation) during the field trial compared with average historical values for the same months. No unusual meteorological or other conditions were reported that would affect the integrity of the study. The treatment regimes (in-furrow or foliar broadcast) used in the grape field trials are reported in Table B.1.2.

B.1.1 Trial Site Conditions.						
Trial Identification (City, State/Year)	Soil Characteristics				Meteorological Data	
	Type	%OM	pH	CEC	Monthly Precipitation (Inches) <sup>1</sup>	Average Monthly Temperature (°C)
						Min
Sodus, NY/2003	Loamy Very Fine Sand		NR <sup>2</sup>		1.3-4.8	4-17 14-27
North Rose, NY/2003	Sand		NR		1.8-4.7	7-16 17-26
Rose Hill, NC/2003	Loamy Sand		NR		5.8-12.3	7-19 19-30
Winter Garden, FL/2003	Fine Sand		NR		3.1-9.1	11-21 23-32
Geneva, MN/2003	Medium Loam		NR		1.6-7.3	7-18 19-28
Delavan, WI/2003	Silt Loam		NR		2.2-5.3	8-17 19-29
Conklin, MI/2003	Loam		NR		2.1-5.5	7-16 17-27
New Rockford, ND/2003	Loam		NR		0.1-4.4	(-1)-14 13-28
Dillon, MT/2003	Coarse Loam		NR		3.1-5.3	6-10 24-52
Porterville, CA/2003	Sandy Loam		NR		1.4-7.3	3-14 15-32
Payette, ID/2003	Loam		NR		1.0-9.4	3-16 17-38
Ephrata, WA/2003	Sand		NR		0.02-12.8	7-13 21-32
American Falls, ID/2003	Loam		NR		0.8-4.5	5-14 21-32
Ashton, ID/2003	Silt Loam		NR		2.7-5.0	6-10 23-31
Madras, OR/2003	Loam		NR		2.3-9.0	4-11 19-32

1. Rainfall and irrigation are included in the precipitation values.  
 2. NR = Not Reported.


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 Crop Field Trial - Potato (Tuber)

**TABLE B.1.2 Study Use Pattern on Potato.**

Location (City, State/Year)	Application									
	EUP	Method <sup>1</sup> ; Timing	Volume (GPA) <sup>2</sup>	Single Rate (lb ai/A) <sup>3</sup>	Number	RTI (Days)	Total Rate (lb ai/A)	Tank Mix Adjuvants		
Sodus, NY/2003	Belay™ 16WSG	In-furrow; at planting.	25	0.200	1	NA <sup>4</sup>	0.200	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	30-31	0.065-0.068	3	7	0.199	None		
North Rose, NY/2003	Belay™ 16WSG	In-furrow; at planting.	20	0.199	1	NA	0.199	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	25	0.064-0.067	3	7	0.196	None		
Rose Hill, NC/2003	Belay™ 16WSG	In-furrow; at planting.	20	0.199	1	NA	0.199	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	17-20	0.066-0.067	3	7-8	0.199	None		
Winter Garden, FL/2003	Belay™ 16WSG	In-furrow; at planting.	20	0.200	1	NA	0.200	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	20	0.066-0.067	3	7	0.199	None		
Geneva, MN/2003	Belay™ 16WSG	In-furrow; at planting.	15	0.198	1	NA	0.198	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	15-16	0.066	3	7	0.199	None		
Delavan, WI/2003	Belay™ 16WSG	In-furrow; at planting.	21	0.206	1	NA	0.206	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	19-20	0.065-0.067	3	6-7	0.198	None		
Conklin, MI/2003	Belay™ 16WSG	In-furrow; at planting.	20	0.198	1	NA	0.198	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	20-21	0.066-0.067	3	7	0.199	None		
New Rockford, ND/2003	Belay™ 16WSG	In-furrow; at planting.	15	0.196	1	NA	0.196	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	15	0.065-0.068	3	7	0.200	None		
Dillon, MT/2003	Belay™ 16WSG	In-furrow; at planting.	13	0.199	1	NA	0.199	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	19-22	0.065-0.069	3	6-7	0.201	None		
Porterville, CA/2003	Belay™ 16WSG	In-furrow; at planting.	20	0.198	1	NA	0.198	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	20	0.067	3	7	0.201	None		



Clothianidin/66330-40 & 66330-52/PC Code 044309/Arvesta Corporation/66330  
 DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIA 8.3.1, 8.3.2, 8.3.3  
 Crop Field Trial - Potato (Tuber)

TABLE B.1.2 Study Use Pattern on Potato.

Location (City, State/Year)	EUP	Method <sup>1</sup> ; Timing	Application					RTI (Days)	Total Rate (lb ai/A) <sup>2</sup>	Tank Mix Adjuvants
			Volume (GPA) <sup>2</sup>	Single Rate (lb ai/A) <sup>2</sup>	Number					
Payette, ID/2003	Belay™ 16WSG	In-furrow; at planting.	15	0.207	1	NA	0.207	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	30-31	0.065-0.069	3	7	0.199	None		
Ephrata, WA/2003	Belay™ 16WSG	In-furrow; at planting.	20	0.197	1	NA	0.197	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	20	0.066	3	7	0.198	None		
American Falls, ID/2003	Belay™ 16WSG	In-furrow; at planting.	20	0.194	1	NA	0.194	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	20-21	0.067-0.068	3	7	0.203	None		
Ashton, ID/2003	Belay™ 16WSG	In-furrow; at planting.	24	0.199	1	NA	0.199	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	20-22	0.067-0.069	3	6-7	0.204	None		
Madras, OR/2003	Belay™ 16WSG	In-furrow; at planting.	20	0.200	1	NA	0.200	None		
	Clutch™ 50WDG	Broadcast foliar; tuber development.	25	0.065	3	7	0.196	None		

- All applications were made using ground equipment. No tank mix adjuvants were used for any applications at any of the field trials.
- GPA = Gallons Per Acre.
- The target single application rate was 0.198 lb ai/A for the in-furrow treatment, and 0.066 lb ai/A for the foliar treatment. The target total application rate was 0.198 lb ai/A for all treatment regimes.
- NA = Not Applicable.

DP Barcodes D309473 & D309474 / MRID #46357302



Clothianidin/66330-40 & 66330-52/PC Code 044309/Arvesta Corporation/66330  
 DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3  
 Crop Field Trial - Potato (Tuber)

**TABLE B.1.3 Potato Field Trial Numbers and Geographical Locations.**

NAFTA Growing Region <sup>1</sup>	Submitted <sup>2</sup>	Requested	
		Canada	US
1	2	NA <sup>3</sup>	2
2	1	NA	1
3	1	NA	1
4	--	NA	--
5	4	NA	4
6	--	NA	--
7	--	NA	--
8	--	NA	--
9	1	NA	1
10	1	NA	1
11	5	NA	6
12	--	NA	--
<b>Total</b>	<b>15</b>	<b>NA</b>	<b>16</b>

- Regions 13 to 21 and 1A, 5A, 5B, and 7A were not included as the proposed use is for the US only.
- Each field trial included side-by-side tests, using Clutch™ 50WDG as a foliar application and Belay™ 16WSG as an in-furrow application, for a total of 30 tests.
- NA = Not Applicable.

**B.2. Sample Handling and Preparation**

Potato tubers were harvested at commercial maturity 85 to 134 DAT from the in-furrow tests, or 13 to 14 DAT from the foliar tests. Additional tuber samples from two trial sites were harvested at 121, 128, and 135 DAT (in-furrow treatment), and at 0, 21, and 28 DAT (foliar treatment) to examine residue decline. A single control and duplicate treated samples (12 to 24 tubers each) were collected from each trial and placed in frozen (at less than 30°C) storage at the test facility for 0 to 44 days. Samples were then shipped by ACDS freezer truck to the analytical laboratory, Morse Laboratories, in Sacramento, California. At Morse Laboratories, all potato samples were homogenized and stored frozen (-20 ± 5°C) prior to analysis. Samples were stored frozen from collection to analysis for durations of up to 3.4 months.

**B.3. Analytical Methodology**

Potato tubers were analyzed for residues of clothianidin and its metabolite, TMG, using two related LC/MS/MS methods entitled *Determination of TM-444 and TMG in Potatoes* (Morse Method #Meth-157) and *Determination of TM-444 and TMG in Grape and Potato Raw Agricultural and Processed Commodities* (Morse Method #Meth-164). Method #Meth-164 is a more current version of Meth-157 that includes procedures for analysis of grapes and processed potato fractions. For analysis of potato tubers, these methods are essentially identical. A detailed description of Method #Meth-164 is presented in the DER for MRID #46346801, in conjunction with an independent laboratory validation (ILV) of that method.

For both methods, residues of clothianidin and TMG are both extracted with ACN/water/guanidine-HCl (20:80:1 vol/vol/wt) and filtered through Celite. The filtrate is concentrated and diluted with water. Separate aliquots are then taken for further cleanup and determination of clothianidin and TMG. Residues of clothianidin are cleaned up using a



ChemElut™ LLE column eluted with cyclohexane/ethyl acetate (1:1 vol/vol). Residues of TMG are cleaned up using a ENVI-Carb™ SPE cartridge eluted with methanol/water/acetic acid (80:20:1 vol/vol/vol). The purified residues are concentrated and re-dissolved in 1% acetic acid for separate determination by LC/MS/MS. The HPLC system consists of a C<sub>18</sub> column with a mobile phase gradient of water to methanol, each containing 1% formic acid. The retention times for clothianidin and TMG are approximately 7.3 and 4.5 minutes, respectively. The monitored transitions are m/z 250 to 169 for clothianidin and m/z 205 to 132 for TMG. The validated LOQ for each analyte is 0.020 ppm in potato tubers and the LOD is 0.007 ppm.

### C. RESULTS AND DISCUSSION

The number and geographic representation of the potato field trials are adequate. A total of 15 potato field trials were conducted during 2003. At each trial site, side-by-side tests were conducted comparing the use of clothianidin as either a single in-furrow application at planting, using the Belay™ 16WSG formulation at a rate of 0.194 to 0.207 lb ai/A (roughly 1X the proposed maximum seasonal use rate of 0.2 lb ai/A for soil treatment), or as three foliar broadcast applications using the Clutch™ 50WDG formulation at a rate of 0.064 of 0.069 lb ai/A per application, for a total use rate of 0.196 to 0.204 lb ai/A per season (roughly 1.4X the proposed maximum seasonal use rate of 0.14 lb ai/A for foliar treatment). No adjuvants were added to the spray tank at any of the field trials for either type of application. The foliar applications were made during tuber development with RTIs of 6 to 8 days. Potato tubers were harvested at commercial maturity 85 to 134 DAT for the in-furrow treatment, or 13 to 14 DAT for the foliar treatment. Additional tuber samples from two trial sites were harvested at 121, 128, and 135 DAT (in-furrow treatment), and at 0, 21, and 28 DAT (foliar treatment) to examine residue decline. Single control and duplicate treated samples were collected from each trial.

The LC/MS/MS methods (Morse Methods #Meth-157 and #Meth-164) used to determine residues of clothianidin and metabolite TMG in potato tubers are adequate for data collection. Average concurrent method recoveries were  $84 \pm 5\%$  for the 21 potato tuber samples fortified with clothianidin at 0.020 to 0.500 ppm and  $89 \pm 9\%$  for the 21 potato tuber samples fortified with metabolite TMG at 0.020 to 0.500 ppm (see Table C.1). Apparent residues of clothianidin were less than the LOD in all control samples. The validated LOQ for clothianidin residues is 0.020 ppm for potato tubers; the LOD is 0.007 ppm. Adequate sample calculations and chromatograms were provided.

Samples were stored frozen from collection to analysis for durations of up to approximately 3½ months (see Table C.2). Storage stability data (MRID #46357301) are available indicating that clothianidin and TMG are stable in frozen potatoes stored for intervals of up to 6 months. These data will support the current potato field trials.

For the in-furrow applications using the Belay™ 16WSG formulation, residues of clothianidin in tubers harvested 85 to 134 DAT were less than 0.020 ppm (the LOQ) in 21 out of 30 samples; residues were 0.020 to 0.033 ppm in the remaining 9 tuber samples. For the foliar applications using the Clutch™ 50WDG formulation, residues of clothianidin in tubers harvested 13 to 14 DAT were less than 0.020 ppm (the LOQ) in 27 out of 29 samples; residues were 0.020 and 0.021 ppm in the remaining 2 samples from one trial (see Table C.3). Average residues of clothianidin were 0.014 ppm for the in-furrow applications and 0.011 ppm for the foliar applications (see Table C.4). HAFT residues for clothianidin were 0.031 ppm following the in-furrow application and 0.021 ppm following the foliar applications. Residues of TMG were non-



detectable (less than 0.007 ppm) in all potato samples from all trials using either formulation.

Residue decline could not be reliably determined in the two field trials having repeated sampling because residues in all samples from these 4 tests were less than the LOQ.

Common cultural practices were used to maintain plants, and the weather conditions and the maintenance chemicals and fertilizer used in the study did not have a notable impact on the residue data.

TABLE C.1 Summary of Method Validation and Concurrent Recovery Results for the LC/MS/MS Methods from Potatoes.					
Analyte	Crop [Matrix]	Spiking Level (mg/kg)	Sample Size	Recoveries (%)	Mean Recovery ± Std Dev (%)
<b>Method Validation Recoveries</b>					
Clothianidin	Potato [Tuber]	0.020	3	83-96	86 ± 8
		0.500	3	79-82	
TMG	Potato [Tuber]	0.020	3	87-113	96 ± 11
		0.500	3	83-100	
<b>Concurrent Method Validation Recoveries</b>					
Clothianidin	Potato [Tuber]	0.020	8	73-91	84 ± 5
		0.050	8	79-95	
		0.100	4	81-92	
		0.500	1	82	
TMG	Potato [Tuber]	0.020	8	73-101	89 ± 9
		0.050	8	82-97	
		0.100	4	82-98	
		0.500	1	105	

TABLE C.2 Summary of Freezer Storage Conditions.			
Potato Matrix	Storage Temperature (°C)	Actual Storage Duration (Months) <sup>1</sup>	Limit of Demonstrated Storage Stability (Months) <sup>2</sup>
Tuber	-20 ± 5	~3½	6

1. Extracts were stored frozen for 0 to 14 days prior to analysis.

2. Storage stability data are available indicating that clothianidin is stable under frozen conditions in potato for intervals of up to 6 months (MRID #46357301).

TABLE C.3 Residue Data from Potato Field Trials with Clothianidin.								
Trial ID (City, State/Year)	EPA Region	Variety	Potato Matrix	Treatment Regime	Total Rate (lb ai/A)	PHI <sup>1</sup> (Days)	Residues (ppm) <sup>2</sup>	
							Clothianidin	TMG
Sodus, NY/2003	j	Monona	Tuber	In-furrow	0.200	108	0.020, 0.021	ND <sup>3</sup> , ND
				Foliar	0.199	14	ND, ND	ND, ND
North Rose, NY/2003	l	Green Mountain	Tuber	In-furrow	0.199	112	(0.009, 0.007] <sup>4</sup>	ND, ND
				Foliar	0.196	14	ND, ND	ND, ND



Clothianidin/66330-40 & 66330-52/PC Code 044309/Arvesta Corporation/66330  
 DACO 7.4.1/OPPTS 860.1500/OECD HIA 6.3.1, 6.3.2, 6.3.3 and HIA 8.3.1, 8.3.2, 8.3.3  
 Crop Field Trial - Potato (Tuber)

TABLE C.3 Residue Data from Potato Field Trials with Clothianidin.								
Trial ID (City, State/Year)	EPA Region	Variety	Potato Matrix	Treat- ment Regime	Total Rate (lb ai/A)	PHI <sup>1</sup> (Days)	Residues (ppm) <sup>2</sup>	
							Clothianidin	TMG
Rose Hill, NC/2003	2	Red Pontiac	Tuber	In-furrow	0.199	85	[0.007, 0.007]	ND, ND
				Foliar	0.199	13	ND, ND	ND, ND
Winter Garden, FL/2003	3	Red Pontiac	Tuber	In-furrow	0.200	88	[0.009, 0.010]	ND, ND
				Foliar	0.199	14	ND, ND	ND, ND
Geneva, MN/2003	5	Cascade	Tuber	In-furrow	0.198	105	ND, ND	ND, ND
				Foliar	0.199	14	ND, ND	ND, ND
Delavan, WI/2003	5	Superior	Tuber	In-furrow	0.206	99	ND, ND	ND, ND
				Foliar	0.198	14	ND, ND	ND, ND
Conklin, MI/2003	5	Norland Dark Red	Tuber	In-furrow	0.198	89	0.020, 0.020	ND, ND
				Foliar	0.199	14	ND, ND	ND, ND
New Rockford, ND/2003	5	Viking	Tuber	In-furrow	0.196	114	ND, ND	ND, ND
						128	[0.010], ND	ND, ND
						135	[0.007, 0.007]	ND, ND
						142	[0.008, 0.010]	ND, ND
				Foliar	0.200	0	ND, ND	ND, ND
						14	ND <sup>5</sup>	ND, ND
						21	ND, ND	ND, ND
						28	ND, ND	ND, ND
Dillon, MT/2003	9	A 7961-2	Tuber	In-furrow	0.199	87	[0.007], ND	ND, ND
				Foliar	0.201	14	0.021, 0.020	ND, ND
Porterville, CA/2003	10	Russett	Tuber	In-furrow	0.198	119	0.033, 0.029	ND, ND
				Foliar	0.201	14	ND, ND	ND, ND
Payette, ID/2003	11	Shepody	Tuber	In-furrow	0.207	124	ND, ND	ND, ND
				Foliar	0.199	14	ND, ND	ND, ND
Ephrata, WA/2003	11	Russett Ranger	Tuber	In-furrow	0.197	120	[0.010, 0.010]	ND, ND
				Foliar	0.198	14	ND, [0.007]	ND, ND



Clothianidin/66330-40 & 66330-52/PC Code 044309/Arvesta Corporation/66330  
 DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3  
 Crop Field Trial - Potato (Tuber)

**TABLE C.3 Residue Data from Potato Field Trials with Clothianidin.**

Trial ID (City, State/Year)	EPA Region	Variety	Potato Matrix	Treat- ment Regime	Total Rate (lb ai/A)	PHI <sup>1</sup> (Days)	Residues (ppm) <sup>2</sup>	
							Clothianidin	TMG
American Falls, ID/2003	11	Russett Burbank	Tuber	In-furrow	0.194	107	[0.007, 0.009]	ND, ND
						121	[0.009, 0.008]	ND, ND
						128	[0.007], ND	ND, ND
						135	ND, ND	ND, ND
				Foliar	0.203	0	ND, ND	ND, ND
						14	ND, ND	ND, ND
						21	ND, ND	ND, ND
						28	ND, ND	ND, ND
Ashton, ID/2003	11	Russett Burbank	Tuber	In-furrow	0.199	87	0.029, 0.027	ND, ND
				Foliar	0.204	14	ND, ND	ND, ND
Madras, OR/2003	11	Russett Burbank	Tuber	In-furrow	0.200	134	[0.010], 0.020	ND, ND
				Foliar	0.196	14	ND, ND	ND, ND

1. PHI = Pre-Harvest Interval.
2. The LOQ is 0.020 ppm and the LOD is 0.007 ppm.
3. ND = Not Detected (less than the LOD).
4. Values in brackets are between the LOD and the LOQ.
5. Only one sample was analyzed for residues, as the second sample thawed at the field trial site.

**TABLE C.4 Summary of Residue Data for Potato Field Trials with Soil (In-Furrow) or Foliar (Broadcast) Applications of Clothianidin.**

Crop [Matrix]	Treatment	Total Rate (lb ai/A)	PHI (Days)	Residue Levels (ppm) <sup>1</sup>					
				n	Min.	Max.	HAFT <sup>2</sup>	Mean	Std. Dev.
<b>Clothianidin Residues</b>									
Potato [Tuber]	In-furrow	0.194-0.207	84-134	30	<0.020	0.033	0.031	0.014	0.007
	Foliar	0.196-0.204	13-14	29	<0.020	0.021	0.021	0.011	0.004
<b>TMG Residues</b>									
Potato [Tuber]	In-furrow	0.194-0.207	84-134	30	<0.020	<0.020	<0.020	<0.020	0
	Foliar	0.196-0.204	13-14	29	<0.020	<0.020	<0.020	<0.020	0

1. The LOQ is 0.020 ppm and the LOD is 0.007 ppm. For calculation of the mean and standard deviation (when some of the sample residues were above the LOQ), half of the LOQ was used as the residue level for samples with residues less than the LOQ.
2. HAFT = Highest Average Field Trial.

**D. CONCLUSION**

The potato field trial data are adequate. These data support the use of clothianidin on potatoes as either a single in-furrow application of the Belay™ 16WSG formulation (at planting) at a rate of 0.2 lb ai/A (1X the proposed maximum seasonal use rate), or the use of up to 3 broadcast foliar applications using the Clutch™ 50WDG formulation (during tuber development) at a rate of 0.066 lb ai/A per application, for a total rate of roughly 0.2 lb ai/A per



season. For the late-season foliar application, the data support a PHI of 14 days. Although residues of clothianidin were low following either type of application, the in-furrow application at planting resulted in higher residues in potato tubers than the late season foliar applications.

#### **E. REFERENCES**

Subject: *Independent Laboratory Validation for the Determination of TM-444 and TMG in Grapes*, Laboratory Study ID #Arvesta-1506

Author: Diane E. Reed

Dated: 2004

MRID: 46346801

#### **F. DOCUMENT TRACKING**

RDI: W. T. Drew (8/19/2005), R. A. Loranger (12/28/2005)

Petition Number: 4F6869

DP Barcode: D309473 and D309474

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