Terrestrial Field Dissipation of Pymetrozine

Report:

Speth, R. M. 1997. Terrestrial field dissipation of CGA-215944 50WP on bare soil in New York. Ciba (Novartis) Laboratory Project 10: 189-95 and ABC Laboratory Project 10: 42962. Unpublished study performed by Ciba (Novartis) Crop Protection, Hudson, NY (field phase), and Ciba (Novartis) Crop Protection, Greensboro, NC and ABC Laboratories, Columbia, MO (analytical phases), and submitted by Novartis Crop Protection, Inc., Greensboro, NC.

Fieser, 1. and R. M. Speth. 1998. Terrestrial field dissipation of CGA-2l5944 50WP on bare soil in New York - Addendum No.1. Ciba (Novartis) Laboratory Project 10: 189-95 and ABC Laboratory Project 10: 42962. Unpublished study performed by ABC Laboratories, Inc., Columbia, MO; and submitted by Novartis Crop Protection, Inc., Greensboro, NC.

Petit, J. B. 1998. Independent laboratory validation of analytical method AG-666: Analytical method for the determination of GS-23199 and CGA-294849, metabolites of CGA-215944, in soil by high performance liquid chromatography with UV detection including validation data. Novartis Laboratory Project 10: 324-97 and CAL Laboratory Project 10: 003-29. Unpublished study performed by Centre Analytical Laboratories, Inc. (CAL), State College, PA; and submitted by Novartis Crop Protection, Inc., Greensboro, NC.

Speth, R.; Gresham, M. (2000) Terrestrial Field Dissipation of CGA-215944 50WP on Bare Soil in New York Addendum No. 2 to Final Report (MRID 44411334): Lab Project Number: 42962: 189-95. Unpublished study prepared by Novartis Crop Protection, Inc. & ABC Labs., Inc. 44 p.

Document No.: MRIDs 44411334, 44647901, 44647902, 45387801

Guideline: OPPTS 835.6100

Classification: This study is classified as **Supplemental**. This study is scientifically valid and provides useful information on terrestrial field dissipation of pymetrozine on bare soil in New York; however, the analytical methods were inadequate for the determination of patterns of formation and decline for the major degradate CGA-249257, and variation in data appear to be a function of inadequate procedural recovery rather than freezer storage instability.

Additional Information This original Data Evaluation Review (DER) (attached) did not include a review of MRID 45387801; therefore, a review is included below as an addendum to the original DER.

PC Code: 101103

Reviewer: Jessica L. O. Joyce, Fate Scientist
U.S. EPA Date: Date: 2018.02.13 16:03:48 -05'00'

Rochelle F.H. Bohaty, Senior Scientist
U.S. EPA Date: 2018.02.08 10:05:47 -05'00'

Introduction

A terrestrial field dissipation study was conducted from June 1995 through December 1996 at a field site in Columbia County, New York to evaluate the mobility and persistence of pymetrozine (CGA-215944) and its major degradation products in soil.

Refer to the original DER (attached) for complete study methodology, results and discussions, and method deficiencies as well as reviewer's comments. The original DER made note that this terrestrial field dissipation study was scientifically valid, but partially fulfilled the Subdivision N Guidelines due to two reasons:

1) The determination of the patterns of formation and decline of the major degradate CGA 249257 was not adequate, due to the instability of the compound;
2) The storage stability data for CGA-180777, CGA-249257, and CGA-359009 were also inadequate.

Study MRID 45387801 is Addendum No. 2 to the final report, and describes the storage stability of CGA 180777, CGA 294849, and GS 23199 under freezer storage conditions, and is discussed below.

Materials and Methods

The formulated product, CGA-215944 50 WP was used as the test substance in this study (described in the final report). All primary stock solutions were stored under freezer conditions (-20 ± 4°C). Calibration and fortification solutions were stored in the refrigerator (6 ± 4°C) or alternatively under freezer conditions (-20 ± 4°C).

CGA-180777

CGA-180777 was analyzed using Ciba Method No. AG-660, entitled: Analytical Method for the Determination of CGA-180777, a Metabolite of CGA-21944, in Soil by HPLC with UV and MS Detection including Validation Data.

Processed bulk homogenized soil from the 0-6-inch layer of the untreated plot was used for preparation of the samples. Thirteen sets of samples were set up to initially cover the storage intervals of 0, 1, 3, 6, 9, 12, 15, 18 months and one additional time point (the remaining four sets would serve as contingency samples).

A total of 65, 20-g samples of soil were weighed. 26 soil samples were fortified with 0.25 ppm of CGA-180777. The remaining 39 samples were labeled to reflect future use as controls and fresh fortifications and all samples were capped and stored at approximately -20 ± 4°C.

At each storage interval, a set of samples (3 controls and 2 fortified samples) were removed from the freezer for analysis.

CGA-294849 and GS-23199

CGA-294849 and GS-23199 were analyzed using Ciba Method No. AG-666, entitled: Analytical Method for the Determination of CGA-294849 and GS-23199, Metabolites of CGA-21944, in Soil by HPLC with UV including Validation Data.

Processed bulk homogenized soil from the 0-6-inch layer of the untreated plot was used for preparation of the samples. Thirteen sets of samples were set up to initially cover the storage intervals of 0, 1, 3, 6, 9, 12, 15, 18, 24, 30 months and one additional time point (the remaining four sets would serve as contingency samples).

A total of 91, 20-g samples of soil were weighed. 26 soil samples (for each analyte) were fortified with 0.1 ppm. The remaining 39 samples were labeled to reflect future use as controls and fresh fortifications and all samples were capped and stored at approximately -20 ± 4°C.

At each storage interval, a set of samples (3 controls and 4 fortified samples) were removed from the freezer for analysis.

Results and Discussion

CGA-180777

The results demonstrate that there are no significant differences in the recoveries of CGA-180777 in the freezer stored samples versus the freshly fortified procedural recovery samples. CGA-180777 was quantitatively recovered from the stored soil throughout the 367-day period. Results are attached.

CGA-294849 and GS-23199

The results demonstrate that there are no significant differences in the recoveries of CGA-294849 and GS-23199 in the freezer stored samples versus the freshly fortified procedural recovery samples. CGA-294849 and GS-23199 were quantitative recovered from stored soil throughout the 992-day period. Results are attached.

Method Deficiencies and Reviewer's Comments

1) The storage stability results presented in this addendum report demonstrate that there are no significant differences in the recoveries of CGA-180777, CGA-294849, and GS-23199 in the freezer stored soil samples versus their respective freshly fortified procedural recovery samples.
2) CGA-180777 can be quantitatively recovered over a period of 637 days of freezer storage.
3) CGA-294849 and GS-23199 can be quantitatively recovered over a period of 922 days of freezer storage.
4) Variation in data appear to be a function of inadequate procedural recovery rather than freezer storage instability.

Table 2. Average Percent Recovery of CGA-180777 from Soil Following Freezer Storage

	Storage Interval								
Days:	0	33	92	181	273	362	455	546	637
Months:	0	1	3	6	9	12	15	18	21
	Average Percent Recovery [1]								
CGA-180777									
Stored Fortified Soil	75	93	93	82	95	88	88	90	81
Freshly Fortified Soil	75	80	82	73	87	87	87	86	84

[1] The reported average percent recovery values are from analyses of duplicate samples of the stored and freshly fortified soil.

Table 3. Average Percent Recovery of CGA-294849 and GS-23199 from Soil Following Freezer Storage

	Storage Interval										
Days:	0	32	91	182	278	362	458	546	719	909	992
Months:	0	1	3	6	9	12	15	18	24	30	33
	Average Percent Recovery [1]										
CGA-294849											
Stored Fortified Soil	80	64	77	77	78	83	93	64	65	95	75
Freshly Fortified Soil	82	66	70	72	89	88	91	73	93	86	83
GS-23199											
Stored Fortified Soil	81	72	71	72	87	89	97	72	74	83	67
Freshly Fortified Soil	82	92	65	84	93	98	98	67	95	75	86

[1] The reported average percent recovery values are from analyses of duplicate samples of the stored and freshly fortified soil.

Appendix I, Table 1. Recovery of CGA-180777 from Soil Following Freezer Storage

ABC Lab No.	Sample ID	Fort. Conc. µg/g	Date Extracted	Date Analyzed	Residue Found (Wet Basis) µg/g	Residue Found Percent Recovery
Storage Interval: Day 0, Number of days elapsed: 0						
1301	Procedural Control	NA	02/13/97	02/18/97	0.0261	-
1302	Procedural Recovery	0.25	02/13/97	02/18/97	0.1841	74
1303	Procedural Recovery	0.25	02/13/97	02/18/97	0.1866	75
					Average	75
1304	Aged Spike CGA-180777	0.25	02/13/97	02/18/97	0.2002	80
1305	Aged Spike CGA-180777	0.25	02/13/97	02/18/97	0.1726	69
					Average	75
Storage Interval: 1 Month, Number of days elapsed: 33						
1310.1	Procedural Control	NA	03/18/97	03/20/97	0.0285	-
1311.1	Procedural Recovery	0.25	03/18/97	03/20/97	0.2092	84
1312.1	Procedural Recovery	0.25	03/18/97	03/20/97	0.1874	75
					Average	80
1313.1	Aged Spike CGA-180777	0.25	03/18/97	03/20/97	0.2442	98
1314.1	Aged Spike CGA-180777	0.25	03/18/97	03/20/97	0.2183	87
					Average	93

Control raw data values were subtracted from procedural recovery values prior to interpolation from the calibration curve.

Appendix I, Table 1. Recovery of CGA-180777 from Soil Following Freezer Storage (Cont.)

ABC Lab No.	Sample ID	Fort. Conc. µg/g	Date Extracted	Date Analyzed	Residue Found (Wet Basis) µg/g	Residue Found Percent Recovery
Storage Interval: 3 Month, Number of days elapsed: 92						
1319	Procedural Control	NA	05/16/97	05/19/97	0.0343	-
1320	Procedural Recovery	0.25	05/16/97	05/19/97	0.2063	83
1321	Procedural Recovery	0.25	05/16/97	05/19/97	0.1992	80
					Average	82
1322	Aged Spike CGA-180777	0.25	05/16/97	05/19/97	0.2337	93
1323	Aged Spike CGA-180777	0.25	05/16/97	05/19/97	0.2311	92
					Average	93
Storage Interval: 6 Month, Number of days elapsed: 181						
1328	Procedural Control	NA	08/13/97	08/15/97	0.0279	-
1329	Procedural Recovery	0.25	08/13/97	08/15/97	0.1795	72
1330	Procedural Recovery	0.25	08/13/97	08/15/97	0.1832	73
					Average	73
1331	Aged Spike CGA-180777	0.25	08/13/97	08/15/97	0.2092	84
1332	Aged Spike CGA-180777	0.25	08/13/97	08/15/97	0.1998	80
					Average	82

Control raw data values were subtracted from procedural recovery values prior to interpolation from the calibration curve.

Appendix I, Table 1. Recovery of CGA-180777 from Soil Following Freezer Storage (Cont.)

ABC Lab No.	Sample ID	Fort. Conc. µg/g	Date Extracted	Date Analyzed	Residue Found (Wet Basis) µg/g	Residue Found Percent Recovery
Storage Interval: 9 Month, Number of days elapsed: 273						
1337	Procedural Control	NA	11/13/97	11/13/97	0.0299	-
1338	Procedural Recovery	0.25	11/13/97	11/13/97	0.2178	87
1339	Procedural Recovery	0.25	11/13/97	11/13/97	0.2140	86
					Average	87
1340	Aged Spike CGA-180777	0.25	11/13/97	11/13/97	0.2375	95
1341	Aged Spike CGA-180777	0.25	11/13/97	11/13/97	0.2347	94
					Average	95
Storage Interval: 12 Month, Number of days elapsed: 362						
1346	Procedural Control	NA	02/10/98	02/11/98	0.0264	-
1347	Procedural Recovery	0.25	02/10/98	02/11/98	0.2166	87
1348	Procedural Recovery	0.25	02/10/98	02/11/98	0.2138	86
					Average	87
1349	Aged Spike CGA-180777	0.25	02/10/98	02/11/98	0.2102	84
1350	Aged Spike CGA-180777	0.25	02/10/98	02/11/98	0.2265	91
					Average	88

Control raw data values were subtracted from procedural recovery values prior to interpolation from the calibration curve.

Appendix I, Table 1. Recovery of CGA-180777 from Soil Following Freezer Storage (Cont.)

ABC Lab No.	Sample ID	Fort. Conc. µg/g	Date Extracted	Date Analyzed	Residue Found (Wet Basis) µg/g	Residue Found Percent Recovery
Storage Interval: 15 Month, Number of days elapsed: 455						
1355	Procedural Control	NA	05/14/98	05/14/98	0.0325	-
1356	Procedural Recovery	0.25	05/14/98	05/14/98	0.2174	87
1357	Procedural Recovery	0.25	05/14/98	05/14/98	0.2165	87
					Average	87
1358	Aged Spike CGA-180777	0.25	05/14/98	05/14/98	0.2192	88
1359	Aged Spike CGA-180777	0.25	05/14/98	05/14/98	0.2180	87
					Average	88
Storage Interval: 18 Month, Number of days elapsed: 546						
1360	Procedural Control	NA	08/13/98	08/19/98	0.0306	-
1361	Procedural Recovery	0.25	08/13/98	08/19/98	0.2201	88
1362	Procedural Recovery	0.25	08/13/98	08/19/98	0.2102	84
					Average	86
1363	Aged Spike CGA-180777	0.25	08/13/98	08/19/98	0.2280	91
1364	Aged Spike CGA-180777	0.25	08/13/98	08/19/98	0.2229	89
					Average	90

Control raw data values were subtracted from procedural recovery values prior to interpolation from the calibration curve.

Appendix I, Table 1. Recovery of CGA-180777 from Soil Following Freezer Storage (Cont.)

ABC Lab No.	Sample ID	Fort. Conc. µg/g	Date Extracted	Date Analyzed	Residue Found (Wet Basis) µg/g	Residue Found Percent Recovery
Storage Interval: 21 Month, Number of days elapsed: 637						
1365	Procedural Control	NA	11/12/98	11/13/98	0.0254	-
1366	Procedural Recovery	0.25	11/12/98	11/13/98	0.2060	82
1367	Procedural Recovery	0.25	11/12/98	11/13/98	0.2123	85
					Average	84
1368	Aged Spike CGA-180777	0.25	11/12/98	11/13/98	0.2059	82
1369	Aged Spike CGA-180777	0.25	11/12/98	11/13/98	0.1976	79
					Average	81

Control raw data values were subtracted from procedural recovery values prior to interpolation from the calibration curve.

Appendix II, Table 1. Recovery of CGA-294849 and GS-23199 from Soil Following Freezer Storage

ABC Lab No.	Sample ID	Fort. Conc. µg/g	Date Extracted	Date Analyzed	Residue Found CGA-294849 (Wet Basis) µg/g	Residue Found CGA-294849 Percent Recovery	Residue Found GS-23199 (Wet Basis) µg/g	Residue Found GS-23199 Percent Recovery
Timepoint: Day 0, Number of days elapsed: 0								
2301	Procedural Control	NA	04/25/97	04/28/97	0.0000	-	0.0000	-
2302	Procedural Fortification	0.05	04/25/97	04/28/97	0.0379	76	0.0379	76
2303	Procedural Fortification	0.05	04/25/97	04/28/97	0.0433	87	0.0435	87
					Average	82		82
2304	Aged Spike CGA-294849	0.1	04/25/97	04/28/97	0.0832	83	0.0056	-
2305	Aged Spike CGA-294849	0.1	04/25/97	04/28/97	0.0764	76	0.0045	-
					Average	80		
2306	Aged Spike GS-23199	0.1	04/25/97	04/28/97	0.0000	-	0.0821	82
2307	Aged Spike GS-23199	0.1	04/25/97	04/28/97	0.0000	-	0.0790	79
					Average			81
Timepoint: 1 Month, Number of days elapsed: 32								
2310	Procedural Control	NA	05/27/97	06/04/97	0.0000	-	0.0000	-
2311	Procedural Fortification	0.1	05/27/97	06/04/97	0.0650	65	0.0909	91
2312	Procedural Fortification	0.1	05/27/97	06/04/97	0.0655	66	0.0934	93
					Average	66		92
2313	Aged Spike CGA-294849	0.1	05/27/97	06/04/97	0.0613	61	0.0149	-
2314	Aged Spike CGA-294849	0.1	05/27/97	06/04/97	0.0671	67	0.0066	-
					Average	64		
2315	Aged Spike GS-23199	0.1	05/27/97	06/04/97	0.0000	-	0.0577	58
2316	Aged Spike GS-23199	0.1	05/27/97	06/04/97	0.0000	-	0.0852	85
					Average			72

Appendix II, Table 1. Recovery of CGA-294849 and GS-23199 from Soil Following Freezer Storage (Cont.)

ABC Lab No.	Sample ID	Fort. Conc. µg/g	Date Extracted	Date Analyzed	Residue Found: CGA-294849 (Wet Basis) µg/g	CGA-294849 Percent Recovery	GS-23199 (Wet Basis) µg/g	GS-23199 Percent Recovery
Timepoint: 3 Month, Number of days elapsed: 91								
2319	Procedural Control	NA	07/25/97	07/30/97	0.0000	-	0.0000	-
2320	Procedural Fortification	0.1	07/25/97	07/30/97	0.0689	69	0.0704	70
2321	Procedural Fortification	0.1	07/25/97	07/30/97	0.0697	70	0.0600	60
					Average	70		65
2322	Aged Spike CGA-294849	0.1	07/25/97	07/30/97	0.0761	76	0.0060	-
2323	Aged Spike CGA-294849	0.1	07/25/97	07/30/97	0.0769	77	0.0048	-
					Average	77		
2324	Aged Spike GS-23199	0.1	07/25/97	07/30/97	0.0000	-	0.0719	72
2325	Aged Spike GS-23199	0.1	07/25/97	07/30/97	0.0000	-	0.0696	70
					Average			71
Timepoint: 6 Month, Number of days elapsed: 182								
2328	Procedural Control	NA	10/24/97	10/27/97	0.0000	-	0.0000	-
2329	Procedural Fortification	0.1	10/24/97	10/27/97	0.0720	72	0.0857	86
2330	Procedural Fortification	0.1	10/24/97	10/27/97	0.0715	72	0.0824	82
					Average	72		84
2331	Aged Spike CGA-294849	0.1	10/24/97	10/27/97	0.0744	74	0.0098	-
2332	Aged Spike CGA-294849	0.1	10/24/97	10/28/97	0.0791	79	0.0345	-
					Average	77		
2333	Aged Spike GS-23199	0.1	10/24/97	10/28/97	0.0000	-	0.0770	77
2334	Aged Spike GS-23199	0.1	10/24/97	10/28/97	0.0000	-	0.0668	67
					Average			72

Appendix II, Table 1. Recovery of CGA-294849 and GS-23199 from Soil Following Freezer Storage (Cont.)

ABC Lab No.	Sample ID	Fort. Conc. µg/g	Date Extracted	Date Analyzed	Residue Found: CGA-294849 (Wet Basis) µg/g	CGA-294849 Percent Recovery	GS-23199 (Wet Basis) µg/g	GS-23199 Percent Recovery
Timepoint: 9 Month. Number of days elapsed: 278								
2337	Procedural Control	NA	01/28/98	01/29/98	0.0000	-	0.0000	-
2338	Procedural Fortification	0.1	01/28/98	01/29/98	0.0878	88	0.0858	86
2339	Procedural Fortification	0.1	01/28/98	01/29/98	0.0893	89	0.0993	99
					Average	89		93
2340	Aged Spike CGA-294849	0.1	01/28/98	01/29/98	0.0764	76	0.0040	-
2341	Aged Spike CGA-294849	0.1	01/28/98	01/29/98	0.0800	80	0.0000	-
					Average	78		
2342	Aged Spike GS-23199	0.1	01/28/98	01/29/98	0.0000	-	0.0711	71
2343	Aged Spike GS-23199	0.1	01/28/98	01/29/98	0.0000	-	0.1022	102
					Average			87
Timepoint: 12 Month. Number of days elapsed: 362								
2346	Procedural Control	NA	04/22/98	04/23/98	0.0000	-	0.0000	-
2347	Procedural Fortification	0.1	04/22/98	04/23/98	0.0884	88	0.0972	97
2348	Procedural Fortification	0.1	04/22/98	04/23/98	0.0865	87	0.0980	98
					Average	88		98
2349	Aged Spike CGA-294849	0.1	04/22/98	04/23/98	0.0819	82	0.0095	-
2350	Aged Spike CGA-294849	0.1	04/22/98	04/23/98	0.0834	83	0.0079	-
					Average	83		
2351	Aged Spike GS-23199	0.1	04/22/98	04/23/98	0.0003	-	0.0809	81
2352	Aged Spike GS-23199	0.1	04/22/98	04/23/98	0.0023	-	0.0962	96
					Average			89

Appendix II, Table 1. Recovery of CGA-294849 and GS-23199 from Soil Following Freezer Storage (Cont.)

ABC Lab No.	Sample ID	Fort. Conc. µg/g	Date Extracted	Date Analyzed	Residue Found CGA-294849 (Wet Basis) µg/g	CGA-294849 Percent Recovery	GS-23199 (Wet Basis) µg/g	GS-23199 Percent Recovery
Timepoint: 15 Month. Number of days elapsed: 458								
2360	Procedural Control	NA	07/27/98	07/28/98	0.0000	-	0.0000	-
2361	Procedural Fortification	0.1	07/27/98	07/28/98	0.0923	92	0.0937	94
2362	Procedural Fortification	0.1	07/27/98	07/28/98	0.0894	89	0.1007	101
					Average	91		98
2363	Aged Spike CGA-294849	0.1	07/27/98	07/28/98	0.0887	89	0.0091	-
2364	Aged Spike CGA-294849	0.1	07/27/98	07/28/98	0.0958	96	0.0101	-
					Average	93		
2365	Aged Spike GS-23199	0.1	07/27/98	07/28/98	0.0000	-	0.0973	97
2366	Aged Spike GS-23199	0.1	07/27/98	07/28/98	0.0000	-	0.0955	96
					Average			97
Timepoint: 18 Month. Number of days elapsed: 546								
2367	Procedural Control	NA	10/23/98	10/26/98	0.0061	-	0.0000	-
2368	Procedural Fortification	0.1	10/23/98	10/26/98	0.0663	66	0.0612	61
2369	Procedural Fortification	0.1	10/23/98	10/26/98	0.0793	79	0.0730	73
					Average	73		67
2370	Aged Spike CGA-294849	0.1	10/23/98	10/27/98	0.0604	60	0.0040	-
2371	Aged Spike CGA-294849	0.1	10/23/98	10/27/98	0.0665	67	0.0042	-
					Average	64		
2372	Aged Spike GS-23199	0.1	10/23/98	10/27/98	0.0012	-	0.0734	73
2373	Aged Spike GS-23199	0.1	10/23/98	10/27/98	0.0000	-	0.0707	71
					Average			72

Appendix II, Table 1. Recovery of CGA-294849 and GS-23199 from Soil Following Freezer Storage (Cont.)

ABC Lab No.	Sample ID	Fort. Conc. µg/g	Date Extracted	Date Analyzed	Residue Found: CGA-294849 (Wet Basis) µg/g	CGA-294849 Percent Recovery	GS-23199 (Wet Basis) µg/g	GS-23199 Percent Recovery
Timepoint: 24 Month, Number of days elapsed: 719								
2374	Procedural Control	NA	04/14/99	04/15/99	0.0000	-	0.0012	-
2375	Procedural Fortification	0.1	04/14/99	04/15/99	0.0914	91	0.0931	93
2376	Procedural Fortification	0.1	04/14/99	04/15/99	0.0948	95	0.0967	97
					Average	93		95
2379	Aged Spike CGA-294849	0.1	04/14/99	04/16/99	0.0651	65	0.0035	-
2380	Aged Spike CGA-294849	0.1	04/14/99	04/16/99	0.0640	64	0.0043	-
					Average	65		
2377	Aged Spike GS-23199	0.1	04/14/99	04/16/99	0.0000	-	0.0732	73
2378	Aged Spike GS-23199	0.1	04/14/99	04/16/99	0.0007	-	0.0743	74
					Average			74
Timepoint: 30 Month, Number of days elapsed: 909								
2381	Procedural Control	NA	10/21/99	11/12/99	0.0000	-	0.0438	-
2382	Procedural Fortification	0.1	10/21/99	11/12/99	0.0878	88	0.0729	73
2383	Procedural Fortification	0.1	10/21/99	11/12/99	0.0829	83	0.0760	76
					Average	86		75
2384	Aged Spike GS-23199	0.1	10/21/99	11/12/99	0.0053	-	0.0794	79
2385	Aged Spike GS-23199	0.1	10/21/99	11/12/99	0.0060	-	0.0867	87
							Average	83
2386	Aged Spike CGA-294849	0.1	10/21/99	11/12/99	0.0729	73	0.0361	-
2387	Aged Spike CGA-294849	0.1	10/21/99	11/12/99	0.1166	117	0.0424	-
					Average	95		

Appendix II, Table 1. Recovery of CGA-294849 and GS-23199 from Soil Following Freezer Storage (Cont.)

ABC Lab No.	Sample ID	Fort. Conc. µg/g	Date Extracted	Date Analyzed	Residue Found: CGA-294849 (Wet Basis) µg/g	CGA-294849 Percent Recovery	GS-23199 (Wet Basis) µg/g	GS-23199 Percent Recovery
Timepoint: 33 Month, Number of days elapsed: 992								
2395	Procedural Control	NA	01/12/00	01/14/00	0.0000	-	0.0004	-
2396	Procedural Fortification	0.1	01/12/00	01/14/00	0.0807	81	0.0838	84
2397	Procedural Fortification	0.1	01/12/00	01/14/00	0.0840	84	0.0883	88
					Average	83		86
2398	Aged Spike GS-23199	0.1	01/12/00	01/14/00	0.0000	-	0.0653	65
2399	Aged Spike GS-23199	0.1	01/12/00	01/14/00	0.0000	-	0.0684	68
							Average	67
2400	Aged Spike CGA-294849	0.1	01/12/00	01/14/00	0.0780	78	0.0073	-
2401	Aged Spike CGA-294849	0.1	01/12/00	01/14/00	0.0718	72	0.0187	-
					Average	75		

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DATA EVALUATION RECORD

STUDY 8

CHEM 101103 Pymetrozine (CGA-215944) §164-1
CAS No. 123312-89-0
FORMULATION--06--WETTABLE POWDER

STUDY ID 44411334
Speth, R. M. 1997. Terrestrial field dissipation of CGA-215944 50WP on bare soil in New York. Ciba (Novartis) Laboratory Project ID: 189-95 and ABC Laboratory Project ID: 42962. Unpublished study performed by Ciba (Novartis) Crop Protection, Hudson, NY (field phase), and Ciba (Novartis) Crop Protection, Greensboro, NC and ABC Laboratories, Columbia, MO (analytical phases), and submitted by Novartis Crop Protection, Inc., Greensboro, NC.

STUDY ID 44647901
Fieser, J. and R. M. Speth. 1998. Terrestrial field dissipation of CGA-215944 50WP on bare soil in New York - Addendum No. 1. Ciba (Novartis) Laboratory Project ID: 189-95 and ABC Laboratory Project ID: 42962. Unpublished study performed by ABC Laboratories, Inc., Columbia, MO; and submitted by Novartis Crop Protection, Inc., Greensboro, NC.

STUDY ID 44647902
Petit, J. B. 1998. Independent laboratory validation of analytical method AG-666: "Analytical method for the determination of GS-23199 and CGA-294849, metabolites of CGA-215944, in soil by high performance liquid chromatography with UV detection including validation data." Novartis Laboratory Project ID: 324-97 and CAL Laboratory Project ID: 003-29. Unpublished study performed by Centre Analytical Laboratories, Inc. (CAL), State College, PA; and submitted by Novartis Crop Protection, Inc., Greensboro, NC.

DIRECT REVIEW TIME = 86.5 Hours

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CONCLUSIONS

Field Dissipation - Terrestrial

1. This study *(MRID# 44471703)* and its addendum *(MRID# 44647904)* can be used to partially fulfil Subdivision N Guidelines of EPA data requirements on terrestrial field dissipation, by providing scientifically valid useful information on the field dissipation of pymetrozine and its degradates on bareground and cotton plots of loamy sand soil in Georgia. However, note that:

   (i) the determination of the patterns of formation and decline of the major degradate CGA-249257 is not adequate, due to the instability of the compound;
   (ii) the storage stability data for CGA-180777, CGA249257 and CGA359009 were also inadequate.

   EFED believes that these issues will not negatively impact the fate and risk assessment of pymetrozine, as presence of these degradates in the field should not be significant, based on the laboratory studies.

2. Pymetrozine (50 WP), broadcast applied at a nominal rate of 748 g a.i./A, dissipated with a registrant-calculated half-life of 22 days ($r^2 = 0.82$; 0-56 days data) on a bareground plot of loam soil in New York. However, the graphical half-life of the parent was 3 days. All reported concentration values are the mean of three replicates unless otherwise noted. Degradate data are reported in parent equivalents. Pymetrozine was initially present in the 0- to 6-inch depth at 0.73 ppm, decreased to 0.36 ppm by 3 days posttreatment and to 0.092 by 56 days and was 0.024 ppm at 532 days; parent compound was not detected below the 0- to 6-inch depth. The major degradate CGA-359009 was present in the 0- to 6-inch depth at 0.040 ppm at day 0, increased to a maximum of 0.086 ppm by 7 days, and was last detected at 0.029 ppm at 369 days posttreatment; CGA-359009 was not detected below the 0- to 6-inch depth. The major degradate CGA-249257 was present in one replicate from the 6- to 12-inch depth at 0.018 ppm at 1 day posttreatment. CGA-249257 was not recovered from field fortified soil samples and was not stable in frozen storage. The major degradate CGA-180777 (niacin, a naturally occurring compound) was present in the 0- to 6-inch depth at 0.015 ppm 1 day prior to application, was a maximum (day 1) of 0.032 ppm, and was <0.010-0.025 ppm from 3 to 532 days; in the 6- to 12-inch depth, CGA-180777 was a maximum of 0.022 ppm (day 1) and was <0.010-0.015 ppm from 3 to 532 days. CGA-180777 was detected in one replicate from the 12- to 18-inch depth, at 0.011 ppm (day 7). Residue data for the degradates CGA-294849 and GS-23199 were reported in a separate study (MRID 44647904); data were reported as means of three replicates unless otherwise noted. The reviewer reported concentrations from one replicate or the mean of two replicates when concentrations were less than the limit of quantitation. The major degradate CGA-294849 was initially detected in the 0- to 6-inch depth at 0.014 ppm (one replicate) at 1 day posttreatment, was a maximum of 0.016 ppm at 7 days, and was last detected at 0.015 ppm (two replicates) at 28 days; CGA-294849 was detected in the 6- to 12-inch depth only at 172 days, at 0.011 ppm (one replicate), and was

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not detected at any other sampling interval or depth. The major degradate GS-23199 was detected in the 0- to 6-inch depth at 0.011 ppm at 7 and 14 (two replicates) days posttreatment; GS-23199 was detected in the 6- to 12-inch depth at 0.012 ppm (one replicate) at 172 days posttreatment and was not detected at any other sampling interval or depth.

METHODOLOGY

Pymetrozine (50 WP) was broadcast applied once as a spray at a nominal application rate of 748 g a.i./A onto a bareground plot (93 × 75 ft. with three subplots of 27 × 75 ft., <1.0% slope; pp. 21; Figures 3-5, pp. 54-56) of Knickerbocker loam soil (39% sand, 42% silt, 18% clay, 2.4% organic matter, pH 5.8, CEC 7.8 meq/100 g; Table 1, p. 36) in Columbia County, New York. An untreated control plot (93 × 30 ft.) was located 100 ft. from the treated plot. A single application was made using a tractor-mounted spray boom 17 inches above the soil surface (p. 22). A five-year plot history indicated no prior use of pymetrozine or related compounds (Table 2, p. 37). The test plot and control plot were treated three times during the study with Roundup® (glyphosate, 1.5 lb. a.i./A; Table 2, p. 37) to control weeds. The depth to the water table was greater than 6 feet (p. 21). Precipitation was supplemented with irrigation as needed; the total water input (77.6 inches) was 132% of the 30-year average (p. 28; Table 3, p. 38). Pan evaporation data were not reported.

Soils samples from the treated and untreated plots were collected 1 day prior to the application and at 0, 1, 3, 7, 14, 28, 56, 90, 123, 172, 279, 369, 452 and 532 days posttreatment. Five soil cores were randomly collected from each treated subplot (total of 15 soil cores) to a depth of 6 inches immediately following application of the pesticide and to a depth of 48 inches at each subsequent sampling interval; samples were also collected from the control plots. (p. 25). A three-stage (Concord) hydraulic probe with an acetate liner was used to collect the 0- to 6-inch depth soil cores (2.25 inch i.d.) and 6- to 48-inch depth soil cores (1.75 inch i.d.). Soil cores were sectioned into 6-inch increments to the 36-inch depth, separated according to subplot and depth increment (five samples per bundle), and shipped frozen (-20°C) to the analytical laboratory where they were composited and homogenized (p. 27).

The tank mix solution was analyzed for the parent by HPLC (Nucleosil C18 column) with an isocratic mobile phase of acetonitrile:water:phosphate buffer (pH 7; 11:77:12, v:v:v) and UV detection (p. 86). The mean concentration of the parent in the tank mix solution was 3.9 ± 0.12 mg/mL immediately following application, which was 102% of the expected concentration (3.78 mg/mL) based on the target application rate (p. 29; Table 6, p. 41). The tank mix solution was not analyzed prior to application.
The application rate was verified using application monitoring pads composed of filter paper (15 cm diameter) and contained in Petri dishes; fifteen pads were utilized per treated plot and five pads were utilized on the control plot to measure spray drift (p. 24).

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Immediately following pesticide application, the pads were sealed inside Petri dishes and shipped frozen to the analytical laboratory. The parent was extracted from the application monitoring pads by shaking with methanol for 30 minutes (p. 88). The extract was diluted with HPLC mobile phase solution (acetonitrile:water:phosphate buffer, pH 7; 11:77:12, v:v:v) and analyzed by HPLC as described above. Mean recovery of the parent from the pads was 16 ± 3.1 $\mu g/cm^2$ (85 ± 17% of expected; equivalent to 648 ± 125 g a.i./A, reviewer calculated; Table 7, p. 42). The initial concentration of the parent in soil (0.73 ppm) was 88% of the expected concentration (reviewer calculated) based on mean recovery from application monitoring pads, and 76% of the expected concentration based on the target application rate.

Soil samples were analyzed for the parent and two degradates: 1,2,4-triazine-3,5(2H,4H)-dione, dihydro-6-methyl-4-[(3-pyridinylmethylene)amino]- (CGA-359009) and 1,2,4-triazin-3(2H)-one, 4,5-dihydro-6-methyl- (CGA-249257). Soil samples were extracted three times by shaking with water:methanol with 1% ammonium hydroxide (1:9, v:v) followed by centrifugation (p. 233). The combined extracts were concentrated by rotary evaporation and redissolved in methanol:water (1:9, v:v) prior to analysis by HPLC (YMC ODS-AQ column) with a mobile phase gradient of 0.02 M ammonium acetate buffer (pH 5.8) in water:methanol (90:10, v:v; System A):0.02 M ammonium acetate buffer (pH 5.8) in water:acetonitrile (50:50, v:v; System B):methanol (System C; A:B:C 100:0:0 to 0:100:0 to 0:0:100) with mass selective detection in the positive ion mode (pp. 257, 258); the limit of quantitation was 0.01 ppm (p. 233).

Soil samples were also analyzed for the degradate 3-pyridinecarboxylic acid (CGA-180777; niacin). Soil samples were extracted twice by shaking with water:methanol with 1% ammonium hydroxide (1:9, v:v; p. 325). The methanol was evaporated, and the aqueous phase was acidified and passed through a solid phase extraction column (SPE; C18) and cation exchange column; the analyte was eluted from the cation exchange column with ammonium hydroxide:methanol (1:99, v:v). The eluate was reduced to dryness, and the residue was dissolved in HPLC mobile phase solution (methanol:water with 0.1% acetic acid; 25:75, v:v) and analyzed by HPLC (Zorbax SCX column) with an isocratic mobile phase and UV detection (265 nm; p. 347); the limit of quantitation was 0.01 ppm. Confirmatory analysis was performed using HPLC with mass selective detection in the positive ion mode (p. 348).

In a method validation study, soil samples from California, Georgia and New York, were fortified separately with the parent and the degradates CGA-359009, CGA-249257 or CGA-180777 at 0.01-1.00 ppm. Mean recoveries of pymetrozine from the California, Georgia and New York soils were 80 ± 8.5%, 82 ± 13.1% and 85 ± 7.2%, respectively; mean recoveries of CGA-359009 were 81 ± 9.2%, 83 ± 13.5% and 81 ± 3.5%, respectively; mean recoveries of CGA-249257 were 82 ± 9.0%, 80 ± 12.0% and 87 ± 3.3%, respectively (p. 252; see Comment #6). Mean recoveries of CGA-180777 from the California, Georgia and New York soils using HPLC/UV analysis were 83.4 ± 5.6%, 72.7 ± 4.7% and 85.5 ± 14.9%, respectively (p. 343); mean recoveries using HPLC/MS analysis were 77.3 ± 8.1%, 76.3 ± 6.7% and 89.8 ± 14.7%, respectively.

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In a concurrent recovery study, mean recoveries from soil samples fortified with pymetrozine and the degradates CGA-359009, CGA-249257 and CGA-180777 at 0.01-1.0 ppm were 97 ± 15% (5 of 30 samples outside 70-120% recovery; p. 30; Table 4, pp. 171, 172), 98 ± 15% (2 of 30 samples >120%), 97 ± 10% (1 of 30 samples >120%), and 85 ± 14% (3 of 36 samples <70%; Table 5, pp. 173, 174), respectively.

To determine stability of the parent and degradates during field handling and shipping, duplicate soil samples were fortified separately with pymetrozine, CGA-359009, CGA-249257, CGA-294849 or CGA-23199 at 0.10 ppm and stored frozen for 131 days prior to extraction (pp. 26, 31, 156), or fortified with CGA-180777 at 0.01-0.10 ppm and stored frozen for 264-288 days prior to extraction (p. 165). Recoveries of pymetrozine, CGA-359009 and CGA-249257 from soil samples were 81%, 75% and 0%, respectively (p. 165; see Comment #1); recoveries of CGA-294849 and CGA-23199 were reported in MRID 44647901. Recovery of CGA-180777 from soil samples was 32-33%.

A frozen storage stability study was conducted by fortifying duplicate soil samples (10 g) separately with parent pymetrozine, CGA-359009 or CGA-249257 at 0.10 ppm, or with CGA-180777 at 0.25 ppm (p. 156). The treated samples were stored frozen for up to 181 days (CGA-180777) or 353 days (pymetrozine, CGA-359009 and CGA-249257) under conditions similar to those used for the field sample storage. Recovery of pymetrozine was 78-97% and did not exhibit a clear pattern of degradation by 353 days of frozen storage (p. 164). Recovery of CGA-359009 was 124-132% from 0 to 93 days posttreatment and 86-93% from 184 to 353 days. Recovery of CGA-249257 was initially 102%, decreased to 57% by 44 days posttreatment, and was 20-30% from 184 to 353 days posttreatment. Recovery of CGA-180777 was 75-93% and did not exhibit a clear pattern of degradation by 181 days of frozen storage (see Comment #2). Field samples were stored frozen for up to 318 days prior to extraction of pymetrozine, CGA-359009 and CGA-249257 (p. 31); samples were stored frozen for up to 608 days prior to extraction of CGA-180777.

MRID 44647901

A separate study was submitted for the determination of two additional degradates from soil samples of the original field study (MRID 44411334). Soil samples were analyzed for the degradates 1,2,4-triazine-3,5(2H, 4H)-dione, 4-amino-6-methyl- (CGA-294849) and 1,2,4-triazine-3,5(2H, 4H)-dione, 6-methyl- (GS-23199). Soil samples were extracted by refluxing with water:methanol (20:80, v:v) and centrifuged (pp. 19, 20; Appendix 1, pp. 73-77). The supernatant was decanted, filtered and concentrated by rotary evaporation. Ammonium hydroxide was added and the extract was concentrated using solid phase extraction (SPE; SAX column). The eluate was collected, concentrated and mixed with formic acid. The solution was sonicated and concentrated using SPE (C18 column attached to the top of a SCX column). The eluate was collected, concentrated and brought to volume with water following the addition of methanol. The extract was analyzed by HPLC (YMC ODS-AQ column) using a mobile phase gradient of

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(A) methanol:water (3:97, v:v) and (B) acetonitrile:water (70:30, v:v; A:B, 100:0 to 0:100, v:v) with UV (265 nm) detection (Appendix 1, Table II, p. 91); the limit of quantitation was 10 ppb (Appendix 1, 67).

In a method validation study, mean recoveries from soil samples (0- to 6-inch depth) fortified separately with CGA-294849 and GS-23199 at 10 ppb and 100 ppb were 83% ± 9.2% and 86% ± 5.1%, respectively (p. 20; Table 3, p. 28). For both compounds, recoveries were greater from samples fortified at 10 ppb (90% recovery for each) than from samples fortified at 100 ppb (75% and 81% recoveries, respectively).

Mean concurrent recoveries from soil samples (0- to 24-inch depths) fortified separately with CGA-294849 and GS-23199 at 10 ppb and 100 ppb were 79% ± 11% (7 of 35 samples <70%) and 90% ± 12% (1 of 35 samples <70%), respectively (p. 21; Table 5, pp. 30, 31).

In a handling and transit stability study of field-fortified samples (described in MRID 44411334), recoveries of CGA-294849 and GS-23199, stored frozen for up to 626 days (p. 22), were 49-119% (2 of 4 samples <70% recovery) and 70-90%, respectively; respective concurrent recoveries were 72-87% and 87-94% (Table 12, p. 52).

In a storage stability study, soil samples fortified at 100 ppb were placed in frozen storage (-14 ± 8 °C) for up to 15 months (pp. 18, 22; see Comment #2). Mean recoveries were 64-93% (1 of 7 samples <70% recovery) and 71-97% for CGA-294849 and GS-23199, respectively (Table 11, pp. 48-51). Respective concurrent recoveries were 66-91% (1 of 7 samples <70%) and 70-97% and generally demonstrated a similar pattern to the recoveries observed in the fortified, stored soil samples, indicating that decreased recoveries were due to procedural error rather than instability of the compounds in storage.

Independent Method Validation MRID 44647902

Five soil samples (20 g) were fortified separately with CGA-294849 and GS-23199 at 10 ppb and 100 ppb and analyzed as described previously for MRID 44647901 (p. 13). Mean recoveries from soil fortified with CGA-294849 at 10 ppb and 100 ppb were 76.5% ± 11.0% (1 of 5 samples <70%) and 87.8% ± 11.5% (1 of 5 samples <70%), respectively; and were 87.9% ± 10.8% and 86.5% ± 11.6% (1 of 5 samples <70%), respectively, from soil fortified with GS-23199 at 10 ppb and 100 ppb (p. 20; Table I, p. 23).

DATA SUMMARY

MRID 44411334

Pymetrozine (50 WP), broadcast applied at a nominal application rate of 748 g a.i./A, dissipated with a registrant-calculated half-life of 22 days ($r^2 = 0.82$; 0 to 56 days posttreatment) on a bareground plot of loam soil (Figure 7, p. 58; Table 14, p. 49). However, the apparent half-life of the parent was 3 days (p. 32; see Comment #3).

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All reported concentration values are the mean of three replicates unless otherwise noted. Degradate data are reported in parent equivalents. The parent was initially present in the 0- to 6-inch depth at 0.73 ppm, decreased to 0.36 ppm by 3 days posttreatment and to 0.092 ppm by 56 days, and was 0.024 ppm at 532 days posttreatment (Table 8, p. 43). Pymetrozine was not detected below the 0- to 6-inch depth (Tables 10, 12; pp. 45, 47). The major degradate

1,2,4-triazine-3,5(2H,4H)-dione, dihydro-6-methyl-4-[(3-pyridinylmethylene)= amino]- (CGA-359009)

was present in the 0- to 6-inch depth at 0.040 ppm at day 0, increased to a maximum of 0.086 ppm by 7 days posttreatment, was 0.026-0.038 ppm from 56 to 369 days posttreatment and was not detected by 452 days posttreatment; CGA-359009 was not detected below the 0- to 6-inch depth. The major degradate

1,2,4-triazin-3(2H)-one, 4,5-dihydro-6-methyl- (CGA-249257)

was present in one replicate from the 6- to 12-inch depth, at 0.018 ppm, at 1 day posttreatment (Table 11, p. 46). However, in stability studies, CGA-249257 was not recovered from field-fortified soil samples and was not stable in test soil in frozen storage (see Comment #1). The major degradate

3-pyridinecarboxylic acid (CGA-180777)

or niacin, a naturally occurring compound, was present in the 0- to 6-inch depth 1 day prior to application of the parent at 0.015 ppm (Table 9, p. 44). CGA-180777 was present in the 0- to 6-inch depth at a maximum of 0.032 ppm at 1 day posttreatment and was <0.010-0.025 ppm from 3 to 532 days posttreatment. CGA-180777 was present in the 6- to 12-inch depth at a maximum of 0.022 ppm at 1 day posttreatment and was <0.010-0.015 ppm from 3 to 532 days posttreatment. CGA-180777 was detected in one replicate in the 12- to 18-inch depth at 0.011 ppm at 7 days posttreatment (Table 13, p. 48).

MRID 44647901

Residue data were reported as means of three replicates unless otherwise noted. The reviewer reported concentrations from one replicate or the mean of two replicates when concentrations were less than the limit of quantitation. The major degradate

4-amino-6-methyl-1,2,4-triazine-3,5(2H, 4H)-dione (CGA-294849)

was initially detected in the 0- to 6-inch depth at 0.014 ppm (one replicate) at 1 day posttreatment, was a maximum of 0.016 ppm at 7 days posttreatment, and was last detected at 0.015 ppm (mean of two replicates) at 28 days posttreatment (Table 6, p. 32); CGA-294849 was detected in the 6- to 12-inch depth at 0.011 ppm (one replicate) at 172

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days posttreatment, and was not detected at any other sampling interval or depth (Table 7, 8; pp. 33, 34). The major degradate

6-methyl-1,2,4-triazine-3,5(2H, 4H)-dione (GS-23199)

was detected in the 0- to 6-inch depth at 0.011 ppm at 7 and 14 (two replicates) days posttreatment; GS-23199 was detected in the 6- to 12-inch depth at 0.012 ppm (one replicate) at 172 days posttreatment and and was not detected at any other sampling interval or depth.

COMMENTS

1. The analytical methods were inadequate for the determination of the patterns of formation and decline of the major degradate CGA-249257. The degradate CGA-249257 was only detected in one replicate from the 6- to 12-inch depth at one sampling interval. Unacceptably low recoveries of the degradate CGA-249257 were observed in a frozen storage stability study (20-30% recovery) following 184-353 days of storage prior to extraction (p. 164). Additionally, the degradate CGA-249257 was not recovered (0% recovery) from soil samples fortified in the field at 0.10 ppm and stored frozen for 131 days prior to extraction (p. 165). Field samples were stored frozen for up to 318 days prior to extraction.

2. The storage stability studies were inadequate. The study author reported that the frozen storage stability study of CGA-180777 was on-going, and that results were only available through 181 days posttreatment; the study author stated that complete results would be reported in an addendum to the study (p. 31). Field samples were stored frozen for up to 608 days prior to extraction of CGA-180777. In addition, recovery of CGA-180777 from field-fortified soil samples stored frozen for 264-288 days prior to extraction was unacceptably low, at 32-33%. [The registrant noted that CGA-180777 (niacin) is naturally occurring compound that is present in all living cells (p. 33).] Additionally, in MRID 44647901, soil samples were stored frozen for up to 990 days prior to extraction (p. 22). However, in the corresponding storage stability study, fortified samples were analyzed following a maximum of 452 days. The study authors stated in MRID 44647901 that storage stability assessments for CGA-294849 and GS-23199 was ongoing and would continue until the maximum storage holding time had been reached.

3. The half-life determination of the parent was inadequate. The registrant calculated a half-life of 22 days ($r^2 = 0.82$; 0 to 56 days posttreatment) for the parent compound. However, based on reported data, the apparent half-life was 3 days. The reviewer noted a biphasic decline of the parent over the 0- to 56-day period which was initially very rapid (0 to 3 days) followed by a relatively slower decline (3 to 56 days). The reviewer calculated half-lives of 3 days ($r^2 = 0.84$; 0 to 3 days) and 30 days ($r^2 = 0.92$; 3 to 56 days) for the two phases based on data presented in Table 8 (p. 43). The reviewer noted that residues of the parent were present from 90 to 532 days posttreatment.

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4. The maximum annual application rate of the parent is two applications at a maximum rate of 340 g a.i./A with a 3-7 day interval between applications. The application rate in the present study was 748 g a.i./A, which was 110% of the maximum annual label rate for tree crops (p. 19). The study author stated that a single application was made at slightly above the maximum annual rate in order to simulate a worst case scenario. The reviewer notes, however, that the target rate was not achieved (based on method validation recovery data).

5. The reviewer notes that CGA-23199 was referred to as GS-23199 in MRIDs 44647901 and 44647902.

6. The reviewer noted that the method validation data reported on pages 252 and 343 (MRID 4441134) closely resemble the data reported in MRID 44411339. However, while the reported mean recoveries are the same for each soil/method/analyte combination, the reported standard deviations differ between the two studies. It was unclear to the reviewer whether the data represented the same method validation study and, if so, why this discrepancy (i.e., different standard deviations reported) occurred.