Lysimeter Study of Pymetrozine

Reports: Coody, P.; Lightle, S. (1997) Soil Dissipation of (carbon 14)CGA-215944 Under Actual Field Conditions in Georgia: Interim Report: Lab Project Number: 1052: 279-96. Unpublished study prepared by PTRL East, Inc. and Hickey's Agri-Services Lab. 269 p.

Lightle, S.; Coody, P.; Atkins, R. (1999) Soil Dissipation of (carbon 14)CGA-215944 Under Actual Field Conditions in Georgia: Lab Project Number: 1052: 279-96. Unpublished study prepared by PTRL East, Inc., Hickey's Agri-Services Laboratory, and Agvise Laboratories, Inc. 571 p.

Lightle, S.; Marsh, J.; Atkins, R. (2000) Frozen Storage Stability of (carbon 14)CGA-215944 in Soil from Georgia: Final Report: Lab Project Number: 1060:321-96. Unpublished study prepared by PTRL East, Inc. 113 p.

Document No.: MRIDs 44411340, 45208704, 45208706

Guideline: Not a guideline study

Classification: A study classification was not provided on the original Data Evaluation Record (attached). This study is not a guideline study, but is classified as **Acceptable**. This study is scientifically valid and provides supplemental information on the terrestrial field dissipation of pymetrozine in lysimeter-enclosed bareground plots of loamy sand soil in Georgia.

This original Data Evaluation Review (DER) (attached) did not include a review of MRIDs 45208704 and 45208706; therefore, a review is included below as an addendum to the original DER.

PC Code: 101103

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Introduction

This study is not a guideline study, but was submitted to supplement the terrestrial field dissipation studies. The original study (MRID 44411340) provides field dissipation data for pymetrozine (CGA-215944) in lysimeter-enclosed bareground plots of loamy sand soil in Georgia, as well as data for degradates CGA-359009, CGA-249257, and CGA-180777. Addendum 1 (MRID 45208704) was submitted after the original review, and discusses the analytical phase and frozen storage stability of GS-23199 and CGA-294849 in soil. MRID 45208706 contains frozen storage stability results for parent only. The following review is for MRIDs 45208704 and 45208706. The original DER for MRID 44411340 is attached for reference.

MRID 45208704

Materials and Methods

The analytical standards for CGA-294849 and GS-23199 were refrigerated at 4 ± 2°C. All soil samples were stored frozen at ≤ -10°C.

Novartis Method AG-666 was used to analyze CGA-294849 and GS-23199 (Appendix A). These two degradates were separated by HPLC and quantitated using an ultra violet detector at an absorbance of 265 nm. The limit of detection for both analytes was 10 ppb. 100 mL of extraction solvent was added to 20 g of soil and refluxed for one hour. The sample was then centrifuged at 9,000 rpm for 10 minutes. The extracting solvent was decanted through filter paper. The sample was concentrated via rotary evaporation until about 20-mL of extract remained. 100 µL of ammonium hydroxide was added and the extract was passed through a preconditioned SAX SPE column and the non-retained eluate was collected. This was then concentrated in a rotary evaporator to approximately 10 mL. 50 µL of formic acid was added, sonicated to mix, and loaded on a preconditioned C_{18} SPE column attached on the back of an SCX column. The non-retained eluate, which contains both analytes was collected and rinsed with the C_{18} rinse solution. The rinse was then added to the same C_{18} and SCX column and the eluate was collected. The columns were then separated and the SCX column was rinsed with methanol and the eluate was also collected. The extract was then concentrated to 3-5 mL in a rotary evaporator. The extract was transferred to a 50 mL concentration tube with the use of excess methanol and concentrated until 2-3 mL of water remained. The final volume was then adjusted to 4 mL with water after the addition of 0.2 mL methanol.

The limit of detection for both analytes was 10 ppb.

The stability of the two degradates were analyzed by first weighing 20 g of control bulk soil into individual bottles. Nine sets of 7 bottles were each weighed out for a total of 63 bottles. All fresh and aged fortifications were done at 1000 ppb. Bottles were capped and stored in the same manner as the study soil samples (freezer at ≤ -10°C). Designated sample sets were removed periodically for analysis according to Novartis Method AG-666. The two fresh fortification samples served as the procedure recovery samples for the set. An analytical set consisted of 4

stored fortified samples, 1 control sample, and 2 procedural recovery samples. The average procedural recovery from the two fresh spikes, if less than 100% was used to correct the results of the stored soil samples.

Results and Discussion

The residues of GS-23199 were detected at levels greater than the Limit of Detection (LOD) at four sampling intervals during the entire study. The residues of CGA-294849 were detected at levels greater than the LOD at eight sampling intervals during the entire study.

The overall average recoveries of CGA-294849 and GS-23199 (n=92) in all three soil depths were 89.1% and 96.7%, respectively. The overall %RSDs were 17.3% and 14.7%, respectively.

Storage stability results indicate that both analytes were stable in soil through the 6-month interval.

MRID 45208706

Summary

The frozen storage stability of pymetrozine (CGA-215944) in Tifton sandy loam soil from Georgia was explored by spiking replicate samples of pymetrozine with both triazine-radiolabels and pyridine-radiolabels, and placing in frozen storage for 555 days (~18 months). Total radiocarbon recovery on average for triazine-labeled samples was 104%. Total radiocarbon recovery on average for pyridine labeled samples was 96%.

Soil extracts 1 and 2 were subjected to chemical characterization to quantitate the chemical species present with the triazine-labeled moiety. Total percent applied radioactivity ranged from 82.1 – 89.2% throughout the 55 days of frozen storage. CGA-359009 was detected between 0.0 – 0.6% if the applied, CGA-294849 was detected between 0.0 – 0.3% of the applied, and CGA-215525 was detected between 0.0 – 1.9% of the applied throughout the 555-day study.

Analytical results from soil treated with pyridine-labeled moiety indicated that on average 98.8% of radiocarbon present in extract 1, and 85.7% is present in extract 2. Total percent of applied radioactivity with the pyridine-labeled moiety was confirmed to range from 74.8% - 82.2% throughout the 555-day study. CGA-359009 was detected between 0.0-0.8%, CGA-300407 was detected between 0.0-2.2% for the duration of the study.

For both the triazine and pyridine labels, an extraction recovery of >90% was achieved. Of the characterized extracted radiocarbon, >97% of Extract 1 radiocarbon and >78% of Extract 2 was identified as parent. No pattern of degradation on frozen soil was observed, indicating that pymetrozine was stable on frozen soil over the 555 days of the study.

Table 4. Summary of CGA-294849 and GS-23199 in Georgia Treated Bare Soil
(0-6 inch depth)
(in ppb, uncorrected dry weight basis)

Interval (days)	CAL ID	Ciba ID	Rep.	CGA-294849 (ppb)*	GS-23199 (ppb)*
-1	961249	009	A	< 10	< 10
-1	961257	017	B	< 10	< 10
-1	961265	025	C	< 10	< 10
			Average	< 10	< 10
0	961298	058	A	< 10	< 10
0	961299	059	B	< 10	< 10
0	961300	060	C	< 10	< 10
			Average	< 10	< 10
1	961312	072	A	< 10	< 10
1	931320	080	B	10.7	< 10
1	961328	088	C	<10	< 10
			Average	10.3	< 10
3	961368	128	A	15.3	15.1
3	961376	136	B	12.3	10.9
3	961384	144	C	12.9	10.7
			Average	13.5	12.2
7	961424	184	A	11.3	< 10
7	961432	192	B	14.6	< 10
7	961440	200	C	<10	< 10
			Average	12.0	< 10
14	961480	240	A	<10	< 10
14	961488	248	B	10.2	< 10
14	961496	256	C	14.2	10.8
			Average	11.5	10.3
21	961536	296	A	11.4	<10
21	961544	304	B	<10	<10
21	961552	312	C	12.9	<10
			Average	11.4	<10
28	961592	352	A	<10	<10
28	961600	360	B	10.3	<10
28	961608	368	C	10.0	<10
			Average	10.1	<10
60	961648	408	A	<10	<10
60	961656	416	B	<10	<10
60	961664	424	C	<10	<10
			Average	<10	<10
90	961704	464	A	<10	<10
90	961712	472	B	<10	<10
90	961720	480	C	<10	<10
			Average	<10	<10
120	961757	520	A	<10	<10
120	961765	528	B	<10	<10
120	961773	536	C	<10	<10
			Average	<10	<10

Table 4. Summary of CGA-294849 and GS-23199 in Georgia Treated Bare Soil (0-6 inch depth), continued
(in ppb, uncorrected dry weight basis)

Interval (days)	CAL ID	Ciba ID	Rep.	CGA-294849 (ppb)*	GS-23199 (ppb)*
180	961813	576	A	<10	<10
180	961821	584	B	<10	<10
180	961829	592	C	<10	<10
			Average	<10	<10
270	962728	632	A	<10	<10
270	962736	640	B	<10	<10
270	962744	648	C	<10	<10
			Average	<10	<10
360	964828	688	A	<10	<10
360	964836	696	B	<10	<10
360	964844	704	C	<10	<10
			Average	<10	<10
450	967204	744	A	<10	<10
450	967212	752	B	<10	<10
450	967220	760	C	<10	<10
			Average	<10	<10
540	970929	800	A	<10	<10
540	970937	808	B	<10	<10
540	970945	816	C	<10	<10
			Average	<10	<10

* In the raw data, uncorrected values less than 5 ppb are listed as < 5 ppb and all values > 5 ppb are reported; however, all uncorrected values less than 10 ppb are tabulated here as < 10 ppb, to match the limit of determination.

NOTE: A value of 10 is used in averaging calculations involving a <10 ppb analysis, where one or more Reps are >10 ppb.

Table 5. Summary of CGA-294849 and GS-23199 in Georgia Treated Bare Soil (6-12 inch depth)
(in ppb, uncorrected dry weight basis)

Interval (days)	CAL ID	Ciba ID	Rep.	CGA-294849 (ppb*)	GS-23199 (ppb*)
-1	961250	010	A	<10	<10
-1	961258	018	B	<10	<10
-1	961266	026	C	<10	<10
			Average	<10	<10
1	961313	073	A	<10	<10
1	961321	081	B	<10	<10
1	961329	089	C	<10	<10
			Average	<10	<10
3	961369	129	A	<10	<10
3	961377	137	B	<10	<10
3	961385	145	C	<10	<10
			Average	<10	<10
7	961425	185	A	<10	<10
7	961433	193	B	<10	<10
7	961441	201	C	<10	<10
			Average	<10	<10
14	961481	241	A	<10	<10
14	961489	249	B	<10	<10
14	961497	257	C	<10	<10
			Average	<10	<10
21	961537	297	A	<10	<10
21	961545	305	B	<10	<10
21	961553	313	C	<10	<10
			Average	<10	<10
28	961593	353	A	<10	<10
28	961601	361	B	<10	<10
28	961609	369	C	<10	<10
			Average	<10	<10
60	961649	409	A	<10	<10
60	961657	417	B	<10	<10
60	961665	425	C	<10	<10
			Average	<10	<10
90	961705	465	A	<10	10.12
90	961713	473	B	<10	<10
90	961721	481	C	<10	<10
			Average	<10	10.0
120	961758	521	A	<10	<10
120	961766	529	B	<10	<10
120	961774	537	C	<10	<10
			Average	<10	<10
180	961814	577	A	<10	<10
180	961822	585	B	<10	<10
180	961830	593	C	<10	<10
			Average	<10	<10
270	962729	633	A	<10	<10
270	962737	641	B	<10	<10
270	962745	649	C	<10	<10
			Average	<10	<10

Table 5. Summary of CGA-294849 and GS-23199 in Georgia Treated Bare Soil (6-12 inch depth), continued
(in ppb, uncorrected dry weight basis)

Interval (days)	CAL ID	Ciba ID	Rep.	CGA-294849 (ppb*)	GS-23199 (ppb*)
360	964829	689	A	<10	<10
360	964837	697	B	<10	<10
360	964845	705	C	<10	<10
			Average	<10	<10
450	967205	745	A	<10	<10
450	967213	753	B	<10	<10
450	967221	761	C	<10	<10
			Average	<10	<10
540	970930	801	A	<10	<10
540	970938	809	B	<10	<10
540	970946	817	C	<10	<10
			Average	<10	<10

* In the raw data, uncorrected values less than 5 ppb are listed as < 5 ppb and all values > 5 ppb are reported; however, all uncorrected values less than 10 ppb are tabulated here as < 10 ppb, to match the limit of determination.

NOTE: A value of 10 is used in averaging calculations involving a <10 ppb analysis, where one or more Reps are >10 ppb.

Table 6. Summary of CGA-294849 and GS-23199 in Georgia Treated Bare Soil
(12-18 inch depth)
(in ppb, uncorrected dry weight basis)

Interval (days)	CAL ID	Ciba ID	Rep.	CGA-294849 (ppb*)	GS-23199 (ppb*)
-1	961251	011	A	<10	<10
-1	961259	019	B	<10	<10
-1	961267	027	C	<10	<10
			Average	<10	<10
1	961314	074	A	<10	<10
1	961322	082	B	<10	<10
1	961330	090	C	<10	<10
			Average	<10	<10
3	961370	130	A	<10	<10
3	961378	138	B	<10	<10
3	961386	146	C	<10	<10
			Average	<10	<10
7	961426	186	A	<10	<10
7	961434	194	B	<10	<10
7	961442	202	C	<10	<10
			Average	<10	<10
14	961482	242	A	<10	<10
14	961490	250	B	<10	<10
14	961498	258	C	<10	<10
			Average	<10	<10
21	961538	298	A	<10	<10
21	961546	306	B	<10	<10
21	961554	314	C	<10	<10
			Average	<10	<10
28	961594	354	A	<10	<10
28	961602	362	B	<10	<10
28	961610	370	C	<10	<10
			Average	<10	<10
60	961650	410	A	<10	<10
60	961658	418	B	<10	<10
60	961666	426	C	<10	<10
			Average	<10	<10
90	961706	466	A	<10	<10
90	961714	474	B	<10	<10
90	961722	482	C	<10	<10
			Average	<10	<10
120	961759	522	A	<10	<10
120	961767	530	B	<10	<10
120	961775	538	C	<10	<10
			Average	<10	<10
180	961815	578	A	<10	<10
180	961823	586	B	<10	<10
180	961831	594	C	<10	<10
			Average	<10	<10
270	962730	634	A	<10	<10
270	962738	642	B	<10	<10
270	962746	650	C	<10	<10
			Average	<10	<10

Table 6. Summary of CGA-294849 and GS-23199 in Georgia Treated Bare Soil (12-18 inch depth), continued
(in ppb, uncorrected dry weight basis)

Interval (days)	CAL ID	Ciba ID	Rep.	CGA-294849 (ppb*)	GS-23199 (ppb*)
360	964830	690	A	<10	<10
360	964838	698	B	<10	<10
360	964846	706	C	<10	<10
			Average	<10	<10
450	967206	746	A	<10	<10
450	967214	754	B	<10	<10
450	967222	762	C	<10	<10
			Average	<10	<10
540	970931	802	A	<10	<10
540	970939	810	B	<10	<10
540	970947	818	C	<10	<10
			Average	<10	<10

* In the raw data, uncorrected values less than 5 ppb are listed as < 5 ppb and all values > 5 ppb are reported; however, all uncorrected values less than 10 ppb are tabulated here as < 10 ppb, to match the limit of determination.

NOTE: A value of 10 is used in averaging calculations involving a <10 ppb analysis, where one or more Reps are >10 ppb.

Table 7. Summary of CGA-294849 and GS-23199 in Georgia Treated Cotton Soil (0-6 inch depth)
(in ppb, uncorrected dry weight basis)

Interval (days)	CAL ID	Ciba ID	Rep.	CGA-294849 (ppb)*	GS-23199 (ppb)*
-1	961273	033	A	<10	<10
-1	961281	041	B	<10	<10
-1	961289	049	C	<10	<10
			Average	<10	<10
0	961301	061	A	<10	<10
0	961302	062	B	<10	<10
0	961303	063	C	<10	<10
			Average	<10	<10
1	961336	096	A	<10	<10
1	961344	104	B	<10	<10
1	961352	112	C	<10	<10
			Average	<10	<10
3	961392	152	A	<10	<10
3	961400	160	B	<10	<10
3	961408	168	C	<10	<10
			Average	<10	<10
7	961448	208	A	<10	<10
7	961456	216	B	11.4	<10
7	961464	224	C	<10	<10
			Average	10.5	<10
14	961504	264	A	<10	<10
14	961512	272	B	<10	<10
14	961520	280	C	<10	<10
			Average	<10	<10
21	961560	320	A	11.0	<10
21	961568	328	B	<10	<10
21	961576	336	C	<10	<10
			Average	10.3	<10
28	961616	376	A	<10	<10
28	961624	384	B	<10	<10
28	961632	392	C	<10	<10
			Average	<10	<10
60	961672	432	A	<10	<10
60	961680	440	B	<10	<10
60	961688	448	C	<10	<10
			Average	<10	<10
90	961728	488	A	<10	<10
90	961735	496	B	<10	<10
90	961742	504	C	<10	<10
			Average	<10	<10
120	961781	544	A	<10	<10
120	961789	552	B	<10	<10
120	961797	560	C	<10	<10
			Average	<10	<10

Table 7. Summary of CGA-294849 and GS-23199 in Georgia Treated Cotton Soil (0-6 inch depth), continued
(in ppb, uncorrected dry weight basis)

Interval (days)	CAL ID	Ciba ID	Rep.	CGA-294849 (ppb)*	GS-23199 (ppb)*
180	961837	600	A	<10	<10
180	961845	608	B	<10	<10
180	961853	616	C	<10	<10
			Average	<10	<10
270	962752	656	A	<10	<10
270	962760	664	B	<10	<10
270	962768	672	C	<10	<10
			Average	<10	<10
360	964852	712	A	<10	<10
360	964860	720	B	<10	<10
360	964868	728	C	<10	<10
			Average	<10	<10
450	967228	768	A	<10	<10
450	967236	776	B	<10	<10
450	967244	784	C	<10	<10
			Average	<10	<10
540	970953	824	A	<10	<10
540	970961	832	B	<10	<10
540	970969	840	C	<10	<10
			Average	<10	<10

* In the raw data, uncorrected values less than 5 ppb are listed as < 5 ppb and all values > 5 ppb are reported; however, all uncorrected values less than 10 ppb are tabulated here as < 10 ppb, to match the limit of determination.

NOTE: A value of 10 is used in averaging calculations involving a <10 ppb analysis, where one or more Reps are >10 ppb.

Table 8. Summary of CGA-294849 and GS-23199 in Georgia Treated Cotton Soil (6-12 inch depth)
(in ppb, uncorrected dry weight basis)

Interval (days)	CAL ID	Ciba ID	Rep.	CGA-294849 (ppb*)	GS-23199 (ppb*)
-1	961274	034	A	<10	<10
-1	961282	042	B	<10	<10
-1	961290	050	C	<10	<10
			Average	**<10**	**<10**
1	961337	097	A	<10	<10
1	961345	105	B	<10	<10
1	961353	113	C	<10	<10
			Average	**<10**	**<10**
3	961393	153	A	<10	<10
3	961401	161	B	<10	<10
3	961409	169	C	<10	<10
			Average	**<10**	**<10**
7	961449	209	A	<10	<10
7	961457	217	B	<10	<10
7	961465	225	C	<10	<10
			Average	**<10**	**<10**
14	961505	265	A	<10	<10
14	961513	273	B	<10	<10
14	961521	281	C	<10	<10
			Average	**<10**	**<10**
21	961561	321	A	<10	<10
21	961569	329	B	<10	<10
21	961577	337	C	<10	<10
			Average	**<10**	**<10**
28	961617	377	A	<10	<10
28	961625	385	B	<10	<10
28	961633	393	C	<10	<10
			Average	**<10**	**<10**
60	961673	433	A	<10	20.2
60	961681	441	B	<10	28.7
60	961689	449	C	<10	22.3
			Average	**<10**	**23.7**
90	961729	489	A	<10	<10
90	961736	497	B	<10	<10
90	961743	505	C	<10	<10
			Average	**<10**	**<10**
120	961782	545	A	<10	<10
120	961790	553	B	<10	<10
120	961798	561	C	<10	<10
			Average	**<10**	**<10**
180	961838	601	A	<10	<10
180	961846	609	B	<10	<10
180	961854	617	C	<10	<10
			Average	**<10**	**<10**
270	962753	657	A	<10	<10
270	962761	665	B	<10	<10
270	962769	673	C	<10	<10
			Average	**<10**	**<10**

Table 8. Summary of CGA-294849 and GS-23199 in Georgia Treated Cotton Soil (6-12 inch depth), continued
(in ppb, uncorrected dry weight basis)

Interval (days)	CAL ID	Ciba ID	Rep.	CGA-294849 (ppb*)	CGA-GS-23199 (ppb*)
360	964853	713	A	<10	<10
360	964861	721	B	<10	<10
360	964869	729	C	<10	<10
			Average	<10	<10
450	967229	769	A	<10	<10
450	967237	777	B	<10	<10
450	967245	785	C	<10	<10
			Average	<10	<10
540	970954	825	A	<10	<10
540	970962	833	B	<10	<10
540	970970	841	C	<10	<10
			Average	<10	<10

* In the raw data, uncorrected values less than 5 ppb are listed as < 5 ppb and all values > 5 ppb are reported; however, all uncorrected values less than 10 ppb are tabulated here as < 10 ppb, to match the limit of determination.

NOTE: A value of 10 is used in averaging calculations involving a <10 ppb analysis, where one or more Reps are >10 ppb.

Table 9. Summary of CGA-294849 and GS-23199 in Georgia Treated Cotton Soil (12-18 inch depth)
(in ppb, uncorrected dry weight basis)

Interval (days)	CAL ID	Ciba ID	Rep.	CGA-294849 (ppb*)	GS-23199 (ppb*)
-1	961275	035	A	<10	<10
-1	961283	043	B	<10	<10
-1	961291	051	C	<10	<10
			Average	<10	<10
1	961338	098	A	<10	<10
1	961346	106	B	<10	<10
1	961354	114	C	<10	<10
			Average	<10	<10
3	961394	154	A	<10	<10
3	961402	162	B	<10	<10
3	961410	170	C	<10	<10
			Average	<10	<10
7	961450	210	A	<10	<10
7	961458	218	B	<10	<10
7	961466	226	C	<10	<10
			Average	<10	<10
14	961506	266	A	<10	<10
14	961514	274	B	<10	<10
14	961522	282	C	<10	<10
			Average	<10	<10
21	961562	322	A	<10	<10
21	961570	330	B	<10	<10
21	961578	338	C	<10	<10
			Average	<10	<10
28	961618	378	A	<10	<10
28	961626	386	B	<10	<10
28	961634	394	C	<10	<10
			Average	<10	<10
60	961674	434	A	<10	<10
60	961682	442	B	<10	<10
60	961690	450	C	<10	<10
			Average	<10	<10
90	961730	490	A	<10	<10
90	961737	498	B	<10	<10
90	961744	506	C	<10	<10
			Average	<10	<10
120	961783	546	A	<10	<10
120	961791	554	B	<10	<10
120	961799	562	C	<10	<10
			Average	<10	<10
180	961839	602	A	<10	<10
180	961847	610	B	<10	<10
180	961855	618	C	<10	<10
			Average	<10	<10
270	962754	658	A	<10	<10
270	962762	666	B	<10	<10
270	962770	674	C	<10	<10
			Average	<10	<10

Table 9. Summary of CGA-294849 and GS-23199 in Georgia Treated Cotton Soil (12-18 inch depth), continued
(in ppb, uncorrected dry weight basis)

Interval (days)	CAL ID	Ciba ID	Rep.	CGA-294849 (ppb*)	GS-23199 (ppb*)
360	964854	714	A	<10	<10
360	964862	722	B	<10	<10
360	964870	730	C	<10	<10
			Average	<10	<10
450	967230	770	A	<10	<10
450	967238	778	B	<10	<10
450	967246	786	C	<10	<10
			Average	<10	<10
540	970955	826	A	<10	<10
540	970963	834	B	<10	<10
540	970971	842	C	<10	<10
			Average	<10	<10

* In the raw data, uncorrected values less than 5 ppb are listed as < 5 ppb and all values > 5 ppb are reported; however, all uncorrected values less than 10 ppb are tabulated here as < 10 ppb, to match the limit of determination.

NOTE: A value of 10 is used in averaging calculations involving a <10 ppb analysis, where one or more Reps are >10 ppb.

Table X. Summary of Characterization of Soil Samples Treated with Triazine[^{14}C]CGA-215944.

Days of Frozen Storage	Sample	Soil Extract	% of Applied as CGA-215944	% of Applied as CGA-359009	% of Applied as CGA-294849	% of Applied as CGA-215525	Total % of Applied Dose
0	1060-41	1	70.0	0.0	0.0	0.0	70.0
		2	10.1	0.0	0.0	0.8	10.9
		Total	**80.1**	**0.0**	**0.0**	**0.8**	**80.9**
	1060-55	1	74.4	0.0	0.0	0.0	74.4
		2	9.7	0.0	0.0	1.2	10.9
		Total	**84.1**	**0.0**	**0.0**	**1.2**	**85.3**
		Average	**82.1**	**0.0**	**0.0**	**1.0**	**83.1**
18	1060-35	1	78.4	0.0	0.0	0.0	78.4
		2	11.4	0.0	0.0	0.0	11.4
		Total	**89.8**	**0.0**	**0.0**	**0.0**	**89.8**
	1060-60	1	77.6	0.0	0.0	0.0	77.6
		2	9.9	0.0	0.0	1.3	11.1
		Total	**87.5**	**0.0**	**0.0**	**1.3**	**88.8**
		Average	**88.6**	**0.0**	**0.0**	**0.6**	**89.3**
32	1060-46	1	75.7	0.0	0.0	0.0	75.7
		2	9.2	0.0	0.0	0.0	9.2
		Total	**84.9**	**0.0**	**0.0**	**0.0**	**84.9**
	1060-61	1	81.2	0.0	0.0	0.0	81.2
		2	9.7	0.0	0.0	1.3	11.0
		Total	**90.9**	**0.0**	**0.0**	**1.3**	**92.2**
		Average	**87.9**	**0.0**	**0.0**	**0.7**	**88.6**
60	1060-33	1	76.5	0.0	0.0	0.0	76.5
		2	9.2	0.0	0.0	0.0	9.2
		Total	**85.7**	**0.0**	**0.0**	**0.0**	**85.7**
	1060-52	1	80.1	0.5	0.0	0.0	80.6
		2	8.1	0.0	0.0	1.3	9.5
		Total	**88.2**	**0.5**	**0.0**	**1.3**	**90.1**
		Average	**87.0**	**0.3**	**0.0**	**0.7**	**87.9**
120	1060-39	1	75.8	0.6	0.0	0.0	76.4
		2	9.3	0.0	0.0	0.0	9.3
		Total	**85.2**	**0.6**	**0.0**	**0.0**	**85.8**
	1060-51	1	81.7	0.0	0.0	0.0	81.7
		2	10.9	0.0	0.0	0.0	10.9
		Total	**92.6**	**0.0**	**0.0**	**0.0**	**92.6**
		Average	**88.9**	**0.3**	**0.0**	**0.0**	**89.2**

Table X (Continued). Summary of Characterization of Soil Samples Treated with Triazine[^{14}C]CGA-215944.

Days of Frozen Storage	Sample	Soil Extract	% of Applied as CGA-215944	% of Applied as CGA-359009	% of Applied as CGA-294849	% of Applied as CGA-215525	Total % of Applied Dose
179	1060-57	1	79.5	0.3	0.0	0.0	79.8
		2	10.7	0.0	0.0	0.0	10.7
		Total	**90.1**	**0.3**	**0.0**	**0.0**	90.5
	1060-58	1	78.6	0.3	0.0	0.0	78.9
		2	9.0	0.0	0.0	0.0	9.0
		Total	**87.6**	**0.3**	**0.0**	**0.0**	87.9
		Average	**88.9**	**0.3**	**0.0**	**0.0**	89.2
270	1060-49	1	75.3	0.0	0.0	0.0	75.3
		2	10.0	0.0	0.0	1.7	11.7
		Total	**86.3**	**0.0**	**0.0**	**1.7**	88.0
	1060-59	1	76.6	0.8	0.0	0.0	77.4
		2	9.9	0.0	0.0	1.1	11.0
		Total	**86.5**	**0.8**	**0.0**	**1.1**	88.4
		Average	**86.4**	**0.4**	**0.0**	**1.4**	88.2
365	1060-37	1	77.7	0.5	0.3	0.0	78.5
		2	10.1	0.0	0.0	0.5	10.6
		Total	**87.8**	**0.5**	**0.3**	**0.5**	89.1
	1060-44	1	71.9	0.5	0.3	0.0	72.7
		2	9.7	0.0	0.0	0.8	10.6
		Total	**81.6**	**0.5**	**0.3**	**0.8**	83.3
		Average	**84.7**	**0.5**	**0.3**	**0.7**	86.2
450	1060-32	1	68.4	0.5	0.0	0.0	68.9
		2	10.9	0.0	0.0	1.9	12.8
		Total	**79.3**	**0.5**	**0.0**	**1.9**	81.7
	1060-38	1	88.5	0.6	0.3	0.0	89.4
		2	10.6	0.0	0.0	1.9	12.5
		Total	**99.1**	**0.6**	**0.3**	**1.9**	101.9
		Average	**89.2**	**0.6**	**0.1**	**1.9**	91.8
555	1060-47	1	72.6	0.3	0.0	0.0	72.9
		2	11.8	0.0	0.0	0.0	11.8
		Total	**84.5**	**0.3**	**0.0**	**0.0**	84.8
	1060-50	1	73.9	0.3	0.0	0.0	74.2
		2	14.0	0.0	0.0	0.0	14.0
		Total	**87.9**	**0.3**	**0.0**	**0.0**	88.2
		Average	**86.2**	**0.3**	**0.0**	**0.0**	86.5

Table XI. Summary of Characterization of Soil Samples Treated with Pyridine[^{14}C]CGA-215944.

Days of Frozen Storage	Sample	Soil Extract	% of Applied as CGA-215944	% of Applied as CGA-359009	% of Applied as CGA-300407	Total % of Applied Dose
0	1060-16	1	72.2	0.0	0.0	72.2
		2	8.7	0.0	1.7	10.3
		Total	**80.9**	**0.0**	**1.7**	82.6
	1060-17	1	73.4	0.0	0.0	73.4
		2	8.2	0.0	1.9	10.2
		Total	**81.6**	**0.0**	**1.9**	83.5
		Average	**81.3**	**0.0**	**1.8**	83.1
18	1060-6	1	66.5	0.0	0.0	66.5
		2	9.8	0.0	1.0	10.8
		Total	**76.3**	**0.0**	**1.0**	77.3
	1060-29	1	67.5	0.0	0.0	67.5
		2	9.8	0.0	0.8	10.6
		Total	**77.3**	**0.0**	**0.8**	78.1
		Average	**76.8**	**0.0**	**0.9**	77.7
32	1060-13	1	71.7	0.0	0.0	71.7
		2	8.7	0.0	0.0	8.7
		Total	**80.4**	**0.0**	**0.0**	80.4
	1060-20	1	71.3	0.0	0.0	71.3
		2	8.6	0.0	0.0	8.6
		Total	**79.9**	**0.0**	**0.0**	79.9
		Average	**80.2**	**0.0**	**0.0**	80.2
60	1060-8	1	74.9	0.7	0.0	75.5
		2	8.1	0.0	1.2	9.3
		Total	**83.0**	**0.7**	**1.2**	84.9
	1060-28	1	73.4	0.0	0.0	73.4
		2	8.1	0.0	1.4	9.5
		Total	**81.5**	**0.0**	**1.4**	82.9
		Average	**82.2**	**0.3**	**1.3**	83.9
120	1060-2	1	69.3	0.6	0.0	69.9
		2	9.1	0.0	1.3	10.4
		Total	**78.4**	**0.6**	**1.3**	80.3
	1060-22	1	69.1	0.0	0.0	69.1
		2	6.5	0.0	0.9	7.5
		Total	**75.6**	**0.0**	**0.9**	76.6
		Average	**77.0**	**0.3**	**1.1**	78.5

Table XI (Continued). Summary of Characterization of Soil Samples Treated with Pyridine[^{14}C]CGA-215944.

Days of Frozen Storage	Sample	Soil Extract	% of Applied as CGA-215944	% of Applied as CGA-359009	% of Applied as CGA-300407	Total % of Applied Dose
179	1060-11	1	68.3	0.7	0.0	69.0
		2	8.3	0.0	1.4	9.7
		Total	**76.6**	**0.7**	**1.4**	**78.7**
	1060-27	1	73.6	0.7	0.0	74.3
		2	8.0	0.0	1.1	9.1
		Total	**81.6**	**0.7**	**1.1**	**83.4**
		Average	**79.1**	**0.7**	**1.3**	**81.1**
270	1060-7	1	72.2	0.8	0.0	73.0
		2	8.5	0.0	1.7	10.2
		Total	**80.7**	**0.8**	**1.7**	**83.2**
	1060-31	1	71.1	0.7	0.0	71.9
		2	8.7	0.0	1.8	10.5
		Total	**79.8**	**0.7**	**1.8**	**82.4**
		Average	**80.3**	**0.8**	**1.8**	**82.8**
365	1060-15	1	70.5	0.0	0.0	70.5
		2	8.8	0.0	1.1	9.9
		Total	**79.3**	**0.0**	**1.1**	**80.4**
	1060-26	1	69.0	0.8	0.5	70.3
		2	10.2	0.0	1.5	11.7
		Total	**79.2**	**0.8**	**2.0**	**82.0**
		Average	**79.2**	**0.4**	**1.5**	**81.2**
450	1060-21	1	68.8	0.7	0.0	69.5
		2	9.9	0.0	2.0	11.9
		Total	**78.7**	**0.7**	**2.0**	**81.4**
	1060-23	1	70.1	0.9	0.0	71.0
		2	9.6	0.0	2.3	11.9
		Total	**79.7**	**0.9**	**2.3**	**82.9**
		Average	**79.2**	**0.8**	**2.2**	**82.2**
555	1060-9	1	66.7	0.7	0.2	67.7
		2	10.3	0.0	1.5	11.8
		Total	**77.0**	**0.7**	**1.7**	**79.5**
	1060-25	1	63.4	0.6	0.0	64.0
		2	9.3	0.0	1.7	11.0
		Total	**72.6**	**0.6**	**1.7**	**75.0**
		Average	**74.8**	**0.7**	**1.7**	**77.2**

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DATA EVALUATION RECORD

STUDY 11

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

STUDY ID 44411340
Coody, P. N. and S. Lightle. 1997. Soil dissipation of [^{14}C]CGA-215944 under actual field conditions in Georgia. Interim report. PTRL Project ID: 1052. Novartis Study No.: 279-96. Unpublished study performed by PTRL East, Inc., Richmond, KY (analytical phase); and Hickey's Agri-Services Laboratory, Camilla, GA (in-life phase); and submitted by Novartis Crop Protection, Inc., Greensboro, NC.

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CONCLUSIONS

Field Dissipation - Terrestrial

1. This study is scientifically valid and provides supplemental information on the terrestrial field dissipation of pymetrozine (CGA-215944) in lysimeter-enclosed bareground plots of loamy sand soil in Georgia.

2. This study was not submitted as a guideline study, but as a supplemental study in addition to studies conducted according to Subdivision N, Series 164-1 Guidelines. Additionally, the reviewed study was an interim report and did not contain complete data sets; the anticipated length of the full study was 18 months.

3. Uniformly triazine ring-labeled [^{14}C]pymetrozine (50 WP) and uniformly pyridine ring-labeled [^{14}C]pymetrozine, applied at a nominal rate of 748 g a.i./A to lysimeter-enclosed bareground plots of Tifton loamy sand soil in Georgia, dissipated with registrant-calculated half-lives of 10.7 days (0 to 14 days data; $r^2 = 0.74$) and 7.9 days (0 to 14 days data; $r^2 = 0.95$), respectively; half-lives were determined from parent compound detected in the 0- to 3-inch depth only, which may have affected half-life estimations. The degradation of the compound (both labels) was biphasic with degradation occurring more slowly in the second phase, for which registrant-calculated half-lives of 114 days (14 to 180 days data; $r^2 = 0.96$) for the triazine label and 103 days (14 to 180 days data; $r^2 = 1.0$) for the pyridine label were determined.

   Uniformly triazine ring-labeled [^{14}C]pymetrozine was initially present in the 0- to 3-inch depth at 1.5 ppm, decreased to 0.79 ppm by 7 days posttreatment, and was 0.19 ppm at 180 days posttreatment. Residues detected below the 3-inch depth were not characterized. Degradate data are reported in parent equivalents. In the 0- to 3-inch depth, the major degradate CGA-359009 was a maximum of 0.065 ppm at 3 days posttreatment and was 0.019 ppm at 180 days. The major degradate CGA-294849 was a maximum of 0.020 ppm at 7 days posttreatment and was last detected at 0.004 ppm at 90 days. The major degradate GS-23199 was a maximum of 0.015 ppm (day 7) and was last detected at 0.003 ppm at 29 days. The major degradate CGA-215525 was present at 0.003-0.017 ppm at 0-90 days posttreatment. Two unidentified major degradates were present at maximums of 0.010 ppm and 0.025 ppm at 90 days posttreatment. Total [^{14}C]residues (in parent equivalents) were present in the 3- to 6-inch, 6- to 12-inch, 12- to 18-inch, 18- to 24-inch, and 24- to 30-inch depths at maximums of 0.16 ppm (1 day), 0.021 ppm (60 days), 0.017 ppm (29 days), 0.007 ppm (one replicate; 60 days), and 0.006 ppm (one replicate; 90 days), respectively.

   Uniformly pyridine ring-labeled [^{14}C]pymetrozine was initially present in the 0- to 3-inch depth at 1.6 ppm, decreased to 0.96 ppm by 3 days posttreatment and 0.44 ppm by 14 days,

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and was 0.15 ppm at 180 days. Residues detected below the 3-inch depth were not characterized. Degradate data are reported in parent equivalents. In the 0- to 3-inch depth, the major degradate CGA-359009 was a maximum of 0.086 ppm at 3 days posttreatment and was 0.021 ppm at 180 days. The major degradate CGA-300407 was a maximum of 0.029 ppm at 3 days posttreatment and was 0.011 ppm at 180 days. An unidentified major degradate was a maximum of 0.014 ppm (one replicate) at 60 days posttreatment. Total [^{14}C]residues (in parent equivalents) were present in the 3- to 6-inch and 6- to 12-inch depths at maximums of 0.012 ppm (3 days) and 0.008 ppm (one replicate; 60 days), respectively; [^{14}C]residues were not detected below the 6- to 12-inch depth.

METHODOLOGY

Uniformly triazine ring-labeled [^{14}C]pymetrozine (50 WP; radiochemical purity 98.7%, specific activity 12.1 µCi/mg; Figure 1, p. 72) and uniformly pyridine ring-labeled [^{14}C]pymetrozine (radiochemical purity 97.5%; specific activity 12.4 µCi/mg) were applied once at a nominal rate of 748 g a.i./A (5.99 mg) to 8-inch diameter steel lysimeter-enclosed bareground plots (4% slope; p. 18) of Tifton loamy sand soil (characteristics not reported) in Mitchell County, GA. The plot contained 57 cylindrical, steel lysimeters (38-inch length; 8-inch i.d.) inserted vertically into the soil to a target depth of 36 inches (leaving the rim two inches above the soil surface). The lysimeters were inserted along an access trench (5.5 feet x 55 feet x 4 feet), and a glass leachate collection apparatus was placed under the soil columns. The lysimeters remained untreated for approximately 23 days prior to the initiation of the study (p. 19). An overflow collection apparatus, consisting of a tube inserted through the rim (one inch above the soil surface) connected to an aluminum-foil covered glass jar was used to collect water that pooled on the top of the soil suface as a result of heavy rainfall. The soil surfaces within 42 lysimeters were individually treated drop-wise around the inner six-inch area with triazine or pyridine ring-labeled test solution using a glass Pasteur pipette (p. 21). Of the remaining lysimeters, three were utilized as untreated controls and four were treated with 972 mg (178 lb Br^-/A) KBr, dissolved in water, to monitor the wetting front associated with the compound treatment (p. 22). Maintenance pesticides were not applied during the in-life phase of the study. The plot history for the preceding five years was not reported. Environmental data were collected on-site (Appendix 3, pp. 99-111). The depth to the water table was not reported. Precipitation was supplemented by irrigation; total water input via precipitation and irrigation was 29.63 inches (Appendix 3, p. 99-111; see Comment #11). Pan evaporation data were not reported.

Soil lysimeters were collected at 0, 1, 3, 7, 14, 29, 60, 90, and 180 days posttreatment for the triazine label study and at 0, 3, 14, 60, and 180 days posttreatment for the pyridine label study (Table I, p. 55). Control lysimeters were collected at 1 and 60 days posttreatment. Soil samples were collected by removing the entire lysimeter from the test plot (p. 23). For the 0 day samples, the 0- to 3-inch depth was removed from the lysimeter in the field and frozen prior to shipping. All other lysimeters were sealed (top and bottom) intact and shipped frozen to the analytical laboratory. The soil columns within the lysimeters were

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divided into 3-inch (to 6-inches) or 6-inch (6- to 36-inches) sections and homogenized (pp. 23, 26). The soil samples were placed into bags and stored frozen until analysis. Prior to extraction, three subsamples of soil from each sampling interval and depth were collected and analyzed for total radioactivity by LSC following combustion; the limits of detection and quantitation were 2 ppb (pp. 14, 27). Soil samples were homogenized again if the coefficient of variation between the three soil aliquots was greater than 15% following LSC analysis. Control soil cores were collected and used to quantify background radioactivity and to determine the gravimetric water content in each soil layer (p. 24).

Soil samples (100 g) were extracted three times by shaking with acetonitrile:0.5 N ammonium hydroxide (4:1, v:v) and centrifuged (p. 28; Figure 4, p. 78). The supernatants were decanted and combined, and triplicate aliquots were analyzed for total radioactivity by LSC. The extracts were concentrated, filtered, and analyzed by reverse-phase HPLC (Alltima C18 column; p. 18) using a mobile phase gradient of 1% acetonitrile and 0.1% trifluoroacetic acid in water (pH 2):0.05% trifluoroacetic acid in methanol (100:0 to 93:7 to 88:12 to 82:18 to 50:50 to 0:100, v:v) with radioactive flow detection (pp. 30, 31); eluate fractions were collected and analyzed for total radioactivity by LSC. Samples were co-chromatographed with nonradiolabeled reference standards which were located by UV light (220 nm). Soil samples for which recoveries were less than 90% of the soil radioactivity following the previous extraction were further extracted by shaking with acetone:1 N acetic acid (3:1, v:v) and centrifuged (p. 29); supernatants were decanted and combined. Aliquots were analyzed by LSC and HPLC as previously described. Soil samples for which recoveries were less than 90% of the total radioactivity remaining in the soil following the previous extraction were further extracted by refluxing with acetonitrile:1 N ammonium hydroxide (4:1, v:v) and centrifuged. Triplicate aliquots of the extracts were analyzed for total radioactivity by LSC. Subsamples of post-extracted soil were air dried, ground to powder, and analyzed for total radioactivity by LSC following combustion.

To confirm compound identities, selected soil extracts from the first and second extraction procedures were analyzed using two-dimensional TLC on cellulose and silica gel plates, respectively, developed perpendicularly with chloroform:methanol:14.8 N ammonium hydroxide:water (80:30:2:4, v:v:v:v) followed by ethyl acetate:methanol: water (75:15:10, v:v:v); radiolabeled compounds were detected with a radioactive imaging scanner (p. 32). Sample extracts were co-chromatographed with nonradiolabeled reference standards which were located with UV light (wavelength unspecified). Areas of radioactivity were scraped from the plates, dissolved in methanol, and analyzed for total radioactivity by LSC.

Leachate accumulations were monitored at 3-day intervals and collected when leachate was present (p. 24). The jars were sealed and shipped frozen to the analytical laboratory. Triplicate aliquots were analyzed for total radioactivity by LSC; the limit of detection was 0.0005 ppm (p. 14). Overflow collection jars were also monitored at 3-day intervals and after heavy precipitation, and collected when overflow water was present (p. 25). The jars were sealed and shipped frozen to the analytical laboratory. Triplicate aliquots were

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analyzed for total radioactivity by LSC (p. 28). Leachate and overflow water samples were concentrated and analyzed by HPLC as previously described for the soil samples (p. 29). Water present on top of the soil surface at the time of sampling was collected, shipped frozen to the analytical laboratory and analyzed for total radioactivity by LSC (p. 25).

DATA SUMMARY

Uniformly triazine ring-labeled [^{14}C]pymetrozine (50 WP; radiochemical purity 98.7%) and uniformly pyridine ring-labeled [^{14}C]pymetrozine (radiochemical purity 97.5%), applied at a nominal rate of 748 g a.i./A to lysimeter-enclosed bareground plots of Tifton loamy sand soil in Georgia, dissipated with registrant-calculated half-lives of 10.7 days (0 to 14 days data; $r^2 = 0.74$) and 7.9 days (0 to 14 days data; $r^2 = 0.95$), respectively (Figure 11, pp. 85, 86); half-lives were determined from parent compound detected in the 0- to 3-inch depth only, which may have affected half-life estimations. The degradation of the compound (both labels) was biphasic with degradation occurring more slowly in the second phase, for which registrant-calculated half-lives of 114 days (14 to 180 days data; $r^2 = 0.96$) for the triazine label and 103 days (14 to 180 days data; $r^2 = 1.0$) for the pyridine label were determined.

Uniformly triazine ring-labeled [^{14}C]pymetrozine

The parent compound was initially present in the 0- to 3-inch depth at 1.5 ppm, decreased with variability to 0.79 ppm by 7 days posttreatment, and was 0.19 ppm at 180 days posttreatment (Table XI, pp. 65-67). Residues detected below the 3-inch depth were not characterized. Degradate data are reported in parent equivalents. In the 0- to 3-inch depth, the major degradate

CGA-359009 (chemical name not reported; chemical structure presented in Figure 1, p. 72)

was initially present at 0.032 ppm, was a maximum concentration of 0.065 ppm at 3 days posttreatment, and was 0.019 ppm at 180 days posttreatment. The major degradate

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

was initially present at 0.007 ppm at 3 days posttreatment, increased to a maximum of 0.020 ppm by 7 days posttreatment and was last detected at 0.008 ppm (one replicate) at 90 days posttreatment. The major degradate

GS-23199 (chemical name not reported; chemical structure presented in Figure 1, p. 73)

was initially present at a maximum of 0.015 ppm at 7 days posttreatment and was last detected at 0.005 ppm (one replicate) at 29 days posttreatment. The major degradate

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CGA-215525 (chemical name not reported; chemical structure presented in Figure 1, p. 72)

was sporadically present, at 0.003-0.017 ppm (single replicates), from 0 to 90 days posttreatment. Two unidentified major degradates were present at maximums of 0.010 ppm and 0.025 ppm at 90 days posttreatment. Total [^{14}C]residues (in parent equivalents) were present in the 3- to 6-inch, 6- to 12-inch, 12- to 18-inch, 18- to 24-inch, and 24- to 30-inch depths at maximums of 0.16 ppm (1 day), 0.021 ppm (60 days), 0.017 ppm (29 days), 0.007 ppm (one replicate; 60 days), and 0.006 ppm (one replicate; 90 days), respectively (Table V, p. 59).

Total [^{14}C]residues detected in the leachate were 0.01-0.44% of the applied from 14 to 90 days posttreatment (Table VII, p. 61). Characterization data for the leachate were reported for samples collected at 11, 15, 18, 29, 33, 43, 48, 60 and 85 days posttreatment. Parent compound was detected at 0.81-6.5 ppb at 11, 29, 43, 48 and 85 days posttreatment. The major degradates CGA-294849 and GS-23199, and an unidentified major degradate were detected at 2.7-27.4 ppb (11-15 and 29-85 days), 1.2-20.6 ppb (11-85 days) and 0.42-11.7 ppb (11-15 and 29-85 days), respectively (Appendix 22, pp. 230-231). The minor degradates CGA-249257, CGA-359009 and CGA-329520 were detected at 0.45-2.3 ppb (11-15, 29 and 60 days), 0.80-8.8 ppb (11 and 29-60 days) and 0.88-5.8 ppb (15, 29-33 and 48-60 days), respectively.

Total [^{14}C]residues detected in the overflow water samples were 0.03-5.3% of the applied from 60 to 180 days posttreatment (Table VII, p. 61). Characterization data for the overflow water samples were reported for samples collected at 33 and 68 days posttreatment. Parent compound was detected at 1.8-39.8 ppb at 33-68 days posttreatment. The major degradates CGA-294849, CGA-249257 and GS-23199, and an unidentified major degradate were detected at 0.84-14.6 ppb (33-68 days), 2.5-10.6 ppb (33 days), 6.0-25.8 ppb (33-68 days) and 0.55-47.3 ppb, respectively (Appendix 23, pp. 233, 234). The minor degradates CGA-215525, CGA-359009 and CGA-329520 were detected at 3.2-8.1 ppb (33-68 days), 0.67-5.5 ppb (33-68 days) and 1.3-2.4 ppb (33-68 days), respectively.

Total [^{14}C]residues (in parent equivalents) detected in the water samples collected from the soil surface at the time of sampling were 262.8-263.8 ppb, 31.2-35.0 ppb and 6.8-9.3 ppb at 14, 180 and 270 days posttreatment, respectively (Appendix 12, p. 162); [^{14}C]residues were not characterized.

Uniformly pyridine ring-labeled [^{14}C]pymetrozine

The parent compound was initially present in the 0- to 3-inch depth at 1.6 ppm, decreased to 0.96 ppm by 3 days posttreatment and 0.44 ppm by 14 days posttreatment, and was 0.15 ppm at 180 days posttreatment (Table XII, pp. 68, 69). Residues detected below the 3-inch depth were not characterized. Degradate data are reported in parent equivalents.

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In the 0- to 3-inch depth, the major degradate

CGA-359009

was initially present at 0.069 ppm at day 0, increased to a maximum of 0.086 ppm by 3 days posttreatment, and was 0.021 ppm at 180 days posttreatment. The major degradate

CGA-300407

was initially present at 0.022 ppm at day 0, increased to a maximum of 0.029 ppm by 3 days posttreatment and decreased with variability to 0.011 ppm by 180 days posttreatment. An unidentified major degradate was detected sporadically and was present at a maximum of 0.014 ppm (one replicate) at 60 days posttreatment. An unidentified minor degradate was present at ≤0.005 ppm at 60 and 180 days posttreatment. Total [^{14}C]residues (in parent equivalents) were present in the 3- to 6-inch and 6- to 12-inch depths at maximums of 0.012 ppm (3 days posttreatment) and 0.008 ppm (one replicate; 60 days posttreatment), respectively; [^{14}C]residues were not detected below the 6- to 12-inch depth (Table VI, p. 60).

Total [^{14}C]residues detected in the leachate were 0.13% (one replicate) and 0.01% (one replicate) of the applied at 14 and 180 days posttreatment (Table VIII, p. 62). Total [^{14}C]residues detected in the overflow water samples were 0.39-0.41% of the applied from 60 (one replicate) to 180 days posttreatment. Characterization data were not reported for the leachate and overflow samples.
Total [^{14}C]residues (in parent equivalents) detected in the water samples collected from the soil surface at the time of sampling were 37.8 ppb at 70 days posttreatment (Appendix 13, p. 164); [^{14}C]residues were not characterized.

COMMENTS

1. This study was not submitted as a guideline study, but as a supplemental study in addition to studies conducted according to Subdivision N, Series 164-1 Guidelines. Additionally, the reviewed study was an interim report and did not contain complete data sets; the anticipated length of the full study was 18 months. The reviewer notes, however, that the pesticide was applied at an exaggerated use rate which is generally not considered to be valid for the determination of the half-life of the parent compound under field conditions.

2. The study was not conducted under typical use conditions. The lysimeters (8-inch inner diameter; p. 18) were too small to be representative of actual use conditions. The method of application was atypical (glass pipette) and only limited areas of the plot (the inner 6 inches of the lysimeter) were treated (p. 21).

3. A reasonable attempt was not made to identify all degradates present at ≥0.01 ppm. In the

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triazine label study, two unidentified major degradates were present at maximums of 0.010 ppm and 0.025 ppm at 90 days posttreatment (Table XI, pp. 65-67). In the pyridine label study, an unidentified major degradate was present at a maximum of 0.014 ppm (one replicate) at 60 days posttreatment (Table XII, pp. 68-69). In the leachate and overflow samples of the triazine label study, unidentified major degradates were detected at maximums of 11.7 ppb (43 days) and 47.3 ppb (33 days), respectively (Appendices 22-23, pp. 230-231, 233-234). Under Subdivision N Guidelines, the registrant must demonstrate that a reasonable attempt was made to identify all degradates present at ≥0.01 ppm.

4. Complete characteristics of the Tifton loamy sand soil were not reported. Subdivision N Guidelines require that the test soil be characterized according to the USDA classification system. The reviewer noted that a soil series description was reported in Figure 3 (pp. 76, 77). The study authors stated that complete physical/chemical characteristics of the test site soil will be included in the final study (p. 38).

5. Limits of quantitation were not reported for LSC analyses of water samples, and limits of detection and quantitation were not reported for HPLC and TLC analyses. Both limits of detection and quantitation should be reported to allow the reviewer to evaluate the adequacy of the method for the determination of the test compound and its degradates.

6. It could not be determined whether monitoring adequately detected all major degradates identified in the aerobic metabolism studies. Two aerobic soil metabolism studies (MRIDs 44024965 and 44024966) were submitted; however, an adequate attempt was not made to extract and characterize degradates in those studies. Monitoring adequately detected all major degradates identified in two anaerobic aquatic metabolism studies (MRIDs 44024967 and 44024968) that were submitted.

7. A plot history was not provided. Additionally, no pretreatment samples were taken to confirm that the test site was not contaminated with the test substance or other related pesticides prior to application of the test substance. Subdivision N guidelines require that the site have no previous use history involving the test compound or related compounds.

8. The maximum proposed label rate for pymetrozine (CGA-215944) was reported to be 680 g a.i./A for tree crops and 170 g a.i./A for row crops (MRID 44411334).

9. The study authors stated that the samples were shipped frozen to the analytical laboratory and placed in frozen storage until being processed for analysis (p. 23). The maximum duration for which the samples were stored and the storage temperature were not reported. The study authors stated that the frozen storage stability of the parent compound in the test soil was being evaluated in a separate study.

10. The slope (4%) of the test plot was relatively steep for a typical field dissipation study (p. 18). However, because lysimeters were utilized and the steel walls extended above the soil surface, the slope was not likely to decrease the vertical infiltrate (which might have otherwise led to an underestimated dissipation half-life) as would have occurred if

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horizontal leaching and/or overland flow had been allowed.

11. Historical average precipitation data corresponding to the months of the year during which the study was conducted were not reported; therefore, total water input during the study as a percentage of the historical average for those months could not be calculated. The average annual rainfall (10 years) for the test area was reported to be 54.64 inches (p. 38). The reported total water input (63.93 inches) via precipitation and irrigation for the first year of the study was reported to be 117% of the 10-year average annual rainfall (Appendix 3, pp. 99-111); for the 180-day period described in this study, the total water input was 54% of the 10-year average annual rainfall.

12. The study authors reported [^{14}C]residue data in units of concentration and as percentages of the applied radioactivity. The percentage data represented the percentage of the applied radioactivity detected in the entire soil segment and was calculated by multiplying the residue soil concentration (µg/g) by the mass of the soil segment. Because the mass of soil segments varied between samples, the patterns of formation and decline observed in the concentration data were different those observed in the percentage data (which were considered to inaccurately represent the residue concentrations). Therefore, the reviewer reported only the concentration data.

13. Evaporative losses from the leachate and overflow collection jars were quantified (p. 25); however, data were not reported. The study authors stated that the available results demonstrated that evaporation from the collection jars, which were covered with perforated lids (overflow jars) or collection funnels (leachate jars), occurred during the short residence time (approximately 3 days) when aqueous samples were in the field. The reported leachate and overflow data were not adjusted for evaporation.

14. Material balances are not required for terrestrial field dissipation studies and are generally not reported. Because this study was conducted with radiolabeled test compounds applied in lysimeters, material balances were reported. Material balances for the triazine and pyridine label studies were 71.6-118.6% and 35.8-116.1% of the applied radioactivity, respectively, with decreasing material balances observed with time (Tables VII-VIII, pp. 61-62). Due to the rapid loss of radioactivity in the pyridine label study, an additional study was conducted in Georgia, to determine the amount of $^{14}CO_2$ produced. "Mini-lysimeters" containing Tifton sandy loam soil were treated with uniformly pyridine ring-labeled [^{14}C]pymetrozine and maintained in a temperature-controlled chamber. Air was passed through the system and volatiles were collected in a series of traps (trapping solutions unspecified; p. 44). Evolved $^{14}CO_2$ accounted for 5-13% of the applied radioactivity by 15 days posttreatment. The study authors stated that the preliminary results indicated that the pyridinyl carbon undergoes rapid mineralization to $^{14}CO_2$ in the test soil and that rapid decline in radioactivity recovered in the field study was believed to be due to emission of $^{14}CO_2$ following treatment (p. 44).