(TDR03B) DATA EVALUATION RECORD (5-3-85)

CASE GS0178 GLYPHOSATE STUDY 2 PM 25 06/16/83

CHEM 103601 Isopropylamine glyphosate

BRANCH EFB DISC 30 TOPIC

FORMULATION 00 - ACTIVE INGREDIENT

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Rueppel, M., B. Brightwell, and J. Marvel, et al. 1972. Final Report on MON-0573, residue and metabolism: Part 3. The degradation and metabolism of MON-0573 in soil: Agricultural Research Report No. 269. Unpublished study received Jan. 30, 1973 under unknown admin. no.; submitted by Monsanto Co., Washington, DC; CDL:120303-D.

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CONCLUSIONS:

Metabolism - Aerobic Aquatic

This portion of the study is scientifically invalid because the data were too variable to assess the decline of glyphosate and patterns of formation and decline of degradates in water. In addition, this portion of the study would not fulfill EPA Data Requirements for Registering Pesticides because the soil was only sampled at one interval and the water was not characterized.

Metabolism - Anaerobic Aquatic

This portion of the study is scientifically invalid because the data were too variable to assess the decline of glyphosate and patterns of formation and decline of degradates in water. In addition, this portion of the study would not fulfill EPA Data Requirements for Registering Pesticides because

the soil was only sampled at one interval, the flooded soil was not aged for 30 days before treatment, and the water was not characterized.

MATERIALS AND METHODS:

Metabolism - Aerobic Aquatic

Samples (5.0 g) of four soils (Table 1) were placed in flasks with 100 ml of distilled water and treated with methyl-labeled, 1-glycine-labeled, or 2-glycine-labeled [^{14}C]glyphosate (specific activity 8.06-10.02 mCi/mM, >96% pure, Monsanto Corporation) at ~10.0 ppm. The flasks were incubated on a shaker, at 30 C for 28, 35, 84, and 112 days, for the Ray, Lintonia, Drummer and Norfolk soils, respectively.

Radioactivity evolved as $^{14}CO_2$ was collected in ascarite traps by purging the flasks with air. Radioactivity was removed from the ascarite by acidification and quantified by LSC. Samples of the supernatant were taken at 14 and 28 days (Ray silt loam); 14, 28, 56, and 112 days (Norfolk sandy loam); 14, 28, 56, and 84 days (Drummer silty clay loam); and 14 and 35 days (Lintonia sandy loam). The supernatant was spotted on TLC plates and developed two dimensionally with eluant I (84 ml aqueous phenol:16 ml water:37.2 g disodium EDTA:1.0 ml acetic acid) followed by eluant II (1.2 g disodium EDTA:100 ml 17N ammonium hydroxide:475 ml water:350 ml 1-propanol:75 ml 2-propanol:75 ml 1-butanol:2.5 l isobutyric acid). Radioactivity was quantified by beta analysis. Soil samples, taken at day 28, 35, 84, and 112 days for the Ray, Lintonia, Drummer and Norfolk soils respectively, were extracted with water, centrifuged, and lyophilized. Total radioactivity in subsamples was determined by combustion and LSC. The remaining sample was extracted 3 times with 0.5 N ammonium hydroxide, and the extracts (water and ammonium hydroxide) were analyzed by TLC/beta camera analysis. Nonextractable radioactivity in the extracted soil samples was determined by combustion and LSC. Recovery values were 76.3, 95.9 and 100% from the Drummer, Ray and Norfolk soils, respectively.

Metabolism - Anaerobic Aquatic

Sample flasks with identical soil samples as in Metabolism- Aerobic Aquatic (treated with [^{14}C]glyphosate and incubated as described previously) were purged with nitrogen gas and the radioactivity evolved as $^{14}CO_2$ was collected and quantified. Supernatant and soil samples were collected and quantified as previously described.

REPORTED RESULTS:

Metabolism - Aerobic Aquatic

The radioactivity detected in aerobically incubated flooded soil is shown in Table 2. Parent glyphosate and degradates detected in the soil extracts and the water are shown in Table 3 and 4. The data presented for all soils and water were variable.

Metabolism - Anaerobic Aquatic

The radioactivity detected in anaerobically incubated flooded soil is shown in Table 5. Parent glyphosate and degradates detected in the soil and supernatant are shown in Tables 6 and 7, respectively. The data presented for all soils and water were variable.

DISCUSSION:

General - Both Experiments

1. Soil samples were analyzed only at the end of the study. Consequently, the pattern of decline of glyphosate and patterns of formation and decline of degradates in the flooded soils could not be determined.

2. The soil textural classes could not be verified for all soils because the sum of the sand, silt, and clay fractions did not total 100%. Based on the percentages reported, the Drummer silty clay loam and the Norfolk sandy loam would be a silty clay and a loamy sand, respectively according to the USDA Soil Textural Classification System.

3. Detection limits were not reported.

4. It was not clear whether the test substance was added before or after flooding.

5. The water was not characterized.

Metabolism - Anaerobic Aquatic

The soil was not flooded for 30 days prior to treatment with [^{14}C]glyphosate.

Table 1. Soil characteristics.

Soil type	Sand	Silt	Clay	Organic matter	pH
		%			
Ray silt loam	6.0	82.3	0.6	1.0	6.5
Drummer silty clay	2.0	55.4	36.8	6.0	7.0
Lintonia sandy loam	70.0	21.0	9.0	1.0	6.0
Norfolk loamy sand	86.0	11.0	2.3	1.0	5.7

Table 2. Radioactivity (% of applied) detected in four flooded soils[a] treated with [^{14}C]glyphosate at 10 ppm and aerobically incubated, with shaking, at 30 C.

Test substance	$^{14}CO_2$	Water soluble		Soil		Total recovered
		Supernatant	Soil wash	Extractable	Nonextractable	
			Ray silt loam			
1-methyl-labeled [^{14}C]glyphosate	46.8	5.4	1.6	22.9	8.5	85.2
1-glycine-labeled [^{14}C]glyphosate	55.3	0.3	0.1	2.7	9.7	68.1
2-glycine-labeled [^{14}C]glyphosate	55.3	1.4	0.1	6.4	40.3	103.5
			Drummer silty clay			
1-methyl-labeled [^{14}C]glyphosate	34.7	18.1	6.9	19.6	16.7	96.0
1-glycine-labeled [^{14}C]glyphosate	41.4	15.9	8.7	13.0	18.0	97.0
2-glycine-labeled [^{14}C]glyphosate	38.3	8.4	5.3	12.0	33.9	97.9
			Norfolk loamy sand			
1-methyl-labeled [^{14}C]glyphosate	5.8	0.8	0.6	81.8	10.5	99.5
1-glycine-labeled [^{14}C]glyphosate	9.3	16.3	6.0	65.4	4.6	101.6
2-glycine-labeled [^{14}C]glyphosate	8.5	2.4	4.1	81.3	13.5	109.8
			Lintonia sandy loam			
1-methyl-labeled [^{14}C]glyphosate	14.2	66.1	2.0	18.3	2.6	103.5

[a] Ray silt loam samples were analyzed after 28 days; Drummer silty clay samples were analyzed after 84 days; Norfolk loamy sand samples were analyzed after 112 days; Lintonia sandy loam samples were analyzed after 35 days.

Table 3. [^{14}C]Glyphosate and AMPA[a] (% of applied) detected in four flooded soils[b] treated at 10 ppm and aerobically incubated with shaking at 30 C.

Test substance	Glyphosate	AMPA	Other
	Ray silt loam		
1-methyl-labeled [^{14}C]glyphosate	1.5	21.2	0.2
1-glycine-labeled [^{14}C]glyphosate	1.7	ND	1.0
2-glycine-labeled [^{14}C]glyphosate	1.1	ND	5.3
	Drummer silty clay		
1-methyl-labeled [^{14}C]glyphosate	12.0	7.1	0.5
1-glycine-labeled [^{14}C]glyphosate	12.7	ND	0.3
2-glycine-labeled [^{14}C]glyphosate	9.9	ND	2.2
	Norfolk loamy sand		
1-methyl-labeled [^{14}C]glyphosate	71.4	6.6	4.7
1-glycine-labeled [^{14}C]glyphosate	63.5	ND	2.0
2-glycine-labeled [^{14}C]glyphosate	79.2	ND	2.2
	Lintonia sandy loam		
1-methyl-labeled [^{14}C]glyphosate	13.6	4.9	ND

[a] Aminomethylphosphonic acid.

[b] Ray silt loam samples were analyzed after 28 days; Drummer silty clay samples were analyzed after 84 days; Norfolk loamy sand samples were analyzed after 112 days; Lintonia sandy loam samples were analyzed after 35 days.

Table 4. [^{14}C]Glyphosate and AMPA[a] detected (% of applied) in supernatant from shake flasks treated with [^{14}C]glyphosate at 10 ppm and incubated aerobically at 30 C.

Test substance	Sampling interval (days)	Glyphosate	AMPA	Other
	Ray silt loam			
1-methyl-labeled [^{14}C]glyphosate	14	0.2	8.5	0.6
	28	ND[b]	4.4	1.0
1-glycine-labeled [^{14}C]glyphosate	14	0.7	ND	ND
	28	ND	ND	ND
2-glycine-labeled [^{14}C]glyphosate	14	7.4	ND	2.7
	28	ND	ND	ND
	Drummer silty clay			
1-methyl-labeled [^{14}C]glyphosate	14	12.5	1.8	0.3
	28	13.7	5.6	1.1
	56	13.4	8.4	2.1
	84	7.6	8.3	2.1
1-glycine-labeled [^{14}C]glyphosate	14	17.5	ND	ND
	28	20.9	ND	0.8
	56	20.8	ND	1.0
	84	15.7	ND	0.2
2-glycine-labeled [^{14}C]glyphosate	14	25.5	ND	ND
	28	14.0	ND	0.4
	56	16.1	ND	0.6
	84	8.3	ND	0.1
	Norfolk loamy sand			
1-methyl-labeled [^{14}C]glyphosate	14	45.6	0.5	ND
	28	65.2	1.7	ND
	56	28.1	ND	ND
	112	0.8	ND	ND
1-glycine-labeled [^{14}C]glyphosate	14	48.3	ND	ND
	28	76.3	ND	ND
	56	72.6	ND	ND
	112	16.3	ND	ND
2-glycine-labeled [^{14}C]glyphosate	14	80.1	ND	ND
	28	77.6	ND	ND
	56	57.6	ND	ND
	112	2.2	ND	0.2
	Lintonia sandy loam			
1-methyl-labeled [^{14}C]glyphosate	14	69.5	6.9	ND
	35	59.5	6.6	ND

[a] Aminomethylphosphonic acid.

[b] Not detected; detection limit not reported.

Table 5. Radioactivity (% of applied) detected in four flooded soils[a] treated with [^{14}C]glyphosate at 10 ppm and anaerobically incubated, with shaking at 30 C.

Test substance	$^{14}CO_2$	Water soluble		Soil		Total recovered
		Supernatant	Soil wash	Extractable	Nonextractable	
			Ray silt loam			
1-methyl-labeled [^{14}C]glyphosate	37.1	2.6	0.3	33.5	12.8	86.3
1-glycine-labeled [^{14}C]glyphosate	51.4	0.2	0.1	4.1	14.1	69.9
2-glycine-labeled [^{14}C]glyphosate	33.5	4.8	0.2	7.5	29.7	75.7
			Drummer silty clay			
1-methyl-labeled [^{14}C]glyphosate	25.1	18.8	4.2	15.3	15.1	78.5
1-glycine-labeled [^{14}C]glyphosate	38.6	8.1	0.8	3.3	15.6	66.4
2-glycine-labeled [^{14}C]glyphosate	24.2	16.1	2.9	15.5	31.6	90.3
			Norfolk loamy sand			
1-methyl-labeled [^{14}C]glyphosate	2.4	1.7	0.8	92.2	12.0	109.1
1-glycine-labeled [^{14}C]glyphosate	5.0	64.2	7.9	26.7	2.4	101.2
2-glycine-labeled [^{14}C]glyphosate	1.4	89.9	2.3	0.5	0.4	94.5
			Lintonia sandy loam			
1-methyl-labeled [^{14}C]glyphosate	6.0	60.9	3.9	28.8	6.5	106.7

[a] Ray silt loam samples were analyzed after 28 days; Drummer silty clay samples were analyzed after 84 days; Norfolk loamy sand samples were analyzed after 112 days; Lintonia sandy loam samples were analyzed after 35 days.

Table 6. [^{14}C]Glyphosate and AMPA[a] (% of applied) detected in four flooded soils[b] treated at 10 ppm and anaerobically incubated with shaking at 30 C.

Test substance	Glyphosate	AMPA	Other
	Ray silt loam		
1-methyl-labeled [^{14}C]glyphosate	3.0	29.7	0.9
1-glycine-labeled [^{14}C]glyphosate	2.4	ND	1.7
2-glycine-labeled [^{14}C]glyphosate	1.7	ND	5.9
	Drummer silty clay		
1-methyl-labeled [^{14}C]glyphosate	3.3	12.0	4.9
1-glycine-labeled [^{14}C]glyphosate	2.8	ND	0.5
2-glycine-labeled [^{14}C]glyphosate	11.8	ND	3.5
	Norfolk loamy sand		
1-methyl-labeled [^{14}C]glyphosate	81.2	6.1	4.9
1-glycine-labeled [^{14}C]glyphosate	21.7	ND	ND
2-glycine-labeled [^{14}C]glyphosate	ND	ND	ND
	Lintonia sandy loam		
1-methyl-labeled [^{14}C]glyphosate	27.2	2.2	ND

[a] Aminomethylphosphonic acid

[a] Ray silt loam samples were analyzed after 28 days; Drummer silty clay samples were analyzed after 84 days; Norfolk loamy sand samples were analyzed after 112 days; Lintonia sandy loam samples were analyzed after 35 days.

Table 7. [^{14}C]Glyphosate and AMPA[a] detected (% of applied radioactivity) in supernatant from shake flasks treated with [^{14}C]glyphosate at 10 ppm and incubated anaerobically at 30 C.

Test substance	Sampling interval (days)	Glyphosate	AMPA[a]	Other
		Ray silt loam		
1-methyl-labeled [^{14}C]glyphosate	14	0.5	12.9	0.6
	28	ND[b]	1.9	0.6
1-glycine-labeled [^{14}C]glyphosate	14	0.9	ND	ND
	28	ND	ND	ND
2-glycine-labeled [^{14}C]glyphosate	14	2.2	ND	0.7
	28	ND	ND	ND
		Drummer silty clay		
1-methyl-labeled [^{14}C]glyphosate	14	16.0	7.2	0.3
	28	2.3	14.8	1.1
	56	1.3	13.5	2.1
	84	1.0	15.0	2.1
1-glycine-labeled [^{14}C]glyphosate	14	18.0	ND	0.1
	28	14.0	ND	0.2
	56	6.8	ND	0.5
	84	8.1	ND	ND
2-glycine-labeled [^{14}C]glyphosate	14	30.8	ND	ND
	28	20.3	ND	0.4
	56	16.5	ND	0.2
	84	15.2	ND	1.0
		Norfolk loamy sand		
1-methyl-labeled [^{14}C]glyphosate	14	77.3	ND	ND
	28	66.1	1.1	ND
	56	71.5	1.1	ND
	112	1.7	ND	ND
1-glycine-labeled [^{14}C]glyphosate	14	82.5	ND	ND
	28	73.9	ND	0.2
	56	82.1	ND	0.7
	112	63.9	ND	0.1
1-glycine-labeled [^{14}C]glyphosate	14	82.5	ND	ND
	28	73.9	ND	0.2
	56	82.1	ND	0.7
	112	63.9	ND	0.1
2-glycine-labeled [^{14}C]glyphosate	14	90.7	ND	ND
	28	99.5	ND	0.3
	56	103.3	ND	0.5
	112	82.9	ND	0.7
		Lintonia sandy loam		
1-methyl-labeled [^{14}C]glyphosate	14	82.9	3.9	ND
	35	58.2	2.7	ND

[a] Aminomethylphosphonic acid.

[b] Not detected; detection limit not reported.