DATA EVALUATION RECORD

STUDY 4

CHEM 417300 Glyphosate acid §162-1
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CONCLUSIONS

Metabolism - Aerobic Soil

1. This study is scientifically valid and provides useful information on the aerobic soil metabolism of glyphosate acid.

2. This study meets Subdivision N Guidelines for the fulfillment of EPA data requirements on aerobic soil metabolism.

3. Radiolabeled [P-methylene-^{14}C]glyphosate acid (radiochemical purity 97.4%), at a nominal rate of 4.74 ± 0.15 ppm, degraded with a registrant-calculated half-life of 5.4 days ($r^2 = 0.86$) in sandy loam soil adjusted to 75% of 1/3 bar moisture content and incubated in darkness at 25.0 ± 1.0 °C for up to 31 days. However, the registrant-calculated half-life does not agree with the apparent first half-life; only 50.0% of the applied radioactivity was present as parent compound at 1 day posttreatment. The degradation was observed to be biphasic. Data reported as percentages of the applied radioactivity are actually percentages of the nominal application. The parent compound was initially 93.0% (4.41 ppm) of the applied radioactivity, decreased to 50.0% (2.37 ppm) by 1 day and 26.7% (1.27 ppm) by 2 days, was 21.2% (1.01 ppm) at 3 days, and was 1.3-2.0% (0.06-0.09 ppm) at 18-31 days posttreatment. The major degradate AMPA was 11.7% of the applied radioactivity at day 1, was a maximum of 24.4% at 11 days and was 19.5-22.4% (individual replicates) at 14-31 days posttreatment. Evolved $^{14}CO_2$ was 23.7% of the applied radioactivity at day 1, was 51.6% at day 4, and increased to a maximum of 65.2% by 24 days posttreatment.

METHODOLOGY

Samples (50 g) of sieved (2 mm) sandy loam soil (collected from Visalia, CA; 71.2% sand, 20.0% silt, 8.8% clay, pH 8.3, 0.60% organic matter, CEC 6.14 meq/100g; Table II, p. 33) were weighed into borosilicate biometer flasks, and adjusted to 75% of the moisture content at 1/3 bar (p. 22). Soil samples were treated with radiolabeled [P-methylene-^{14}C]glyphosate acid (N-(Phosphonomethyl)glycine; radiochemical purity 97.4%, specific activity 42.7 mCi/mmole, p. 15; Figure 4, p. 42), dissolved in water, at a nominal rate of 4.74 ± 0.15 ppm (p. 22). Flasks containing treated soil samples were sealed, shaken by hand, and incubated in darkness at 25.0 ± 1.0 °C for up to 31 days; the oxygen and soil moisture levels were maintained throughout the incubation period. Volatiles were trapped in a sidearm containing 10% KOH solution that was connected to each biometer flask by a tube containing a foam plug (p. 20; Figure 5, p. 43). Duplicate samples were removed for analysis at 0, 1, 2, 3, 4, 8, 11, 14, 18, 24, and 31 days posttreatment (p. 23).

At each sampling interval, soil samples were extracted three times with 1 M aqueous KH_2PO_4 (pH 2) by shaking; the samples were centrifuged, and the supernatants were decanted and combined (p. 23). Triplicate aliquots of each solution were analyzed for total radioactivity by LSC (p. 23). Following each extraction, aliquots of extract were analyzed by HPLC (Bio-Rad HRLC Glyphosate column) using a mobile phase gradient of 5 mM KH_2PO_4 (4% methanol) with fraction collection followed by analysis of fractions for total radioactivity by LSC. Samples were co-chromatographed with unlabeled reference standards which were detected by refractive index; the limit of detection was twice the background (pp. 24, 26). Compound identities were confirmed using the 11 day posttreatment soil extracts, which were analyzed by TLC using silica gel plates developed in methanol:ammonium hydroxide (29.9%):trichloroacetic acid:water (55:14:4.5:31, v:v:w:v; p. 25), and visualized with a radioimaging scanner. Samples were identified by comparison with reference standards which were visualized using 2% ninhydrin solution in ethanol. Following extraction, triplicate soil subsamples (0.5 g) were analyzed for total radioactivity by LSC following combustion (pp. 23, 24).

Volatile traps were removed for analysis at each sampling interval and analyzed for total radioactivity by LSC (p. 23). The presence of $^{14}CO_2$ was confirmed by precipitation with $BaCl_2$; the supernatant was analyzed for total radioactivity by LSC (p. 24). The foam plugs were extracted with dichloromethane (20 mL) and analyzed for total radioactivity by LSC (p. 23).

To determine soil viability, microbial analyses were conducted using spread plate enumeration techniques at the initiation and conclusion of the study; serial soil dilutions were plated onto trypticase soy agar (aerobic bacteria), actinomycetes isolation agar (actinomycetes), and potato dextrose agar (fungi; p. 21). Microbial populations indicated that the soils remained viable throughout the incubation period (Table III, p. 34).

DATA SUMMARY

Radiolabeled [P-methylene-^{14}C]glyphosate acid (radiochemical purity 97.4%), at a nominal rate of 4.74 ± 0.15 ppm, degraded with a registrant-calculated half-life of 5.4 days (r^2 = 0.86) in sandy loam soil adjusted to 75% of 1/3 bar moisture content and incubated in darkness at 25.0 ± 1.0 °C for up to 31 days (Table VII, p. 38; Figure 21, p. 61). However, the registrant-calculated half-life does not agree with the apparent half-life; 50.0% of the applied radioactivity was present as parent compound at 1 day posttreatment. The degradation was observed to be biphasic. Data reported as percentages of the applied radioactivity are actually percentages of the nominal application. The parent compound was initially 93.0% (4.41 ppm) of the applied radioactivity, decreased to 50.0% (2.37 ppm) by 1 day and 26.7% (1.27 ppm) by 2 days, was 21.2% (1.01 ppm) at 3 days, and was 1.3-2.0% (0.06-0.09 ppm) at 18-31 days posttreatment (Table VI, p. 37). The major degradate

aminomethylphosphonic acid (AMPA)

was initially present (day 0) at 1.6% of the applied radioactivity, was 11.7% of the applied at day 1, increased to a maximum of 24.4% of the applied by 11 days posttreatment, and was 19.5-22.4% (individual replicates) of the applied at 14-31 days posttreatment. An unidentified minor degradate was a maximum of 1.3% of the applied radioactivity at 24 days posttreatment and was 1.2% of the applied at 31 days posttreatment. Evolved $^{14}CO_2$ was 23.7% of the applied radioactivity at day 1, was 51.6% at day 4, and increased to a maximum of 65.2% of the applied by 24 days posttreatment, and was 64.3% of the applied at 31 days posttreatment (Table V, p. 36).

Material balances, based on LSC analysis of individual replicates, were 83.4-100.7% of the applied radioactivity throughout the incubation period, with the exception of 45.3% of the applied radioactivity in one replicate at 18 days posttreatment (Table V, p. 36; see Comment #2); a pattern of decline was not observed.

COMMENTS

1. The registrant-calculated half-life of 5.4 days ($r^2 = 0.86$; Figure 21, p. 61) did not agree with the apparent first half-life; only 50.0% of the applied radioactivity was present as parent compound at 1 day posttreatment, and only 14.7% of the applied radioactivity was present as parent at 4 days posttreatment (Table VII, p. 38).

2. Material balances were less than the reasonable range of 90-110% in 4 of the 22 replicate samples (Table V, p. 36). Three replicates had material balances in the range of 83.4-89.7% of the applied radioactivity, and one replicate had a material balance of 43.3% of the applied (18 days posttreatment). The study author reported that the loss of radioactivity was possibly due to the leaking of volatile $^{14}CO_2$ (p. 28).

3. The sandy loam soil was collected in 1994 from a field site that had been treated with Gramoxone Extra in 1992, and with diazinon and malathion in 1993 (p. 21).

4. The study-author reported that the 4.74 ± 0.15 ppm treatment rate was equivalent to a maximum single application rate of 4 lb/acre (p. 16).

5. The soil series name for the sandy loam soil collected from Visalia, CA, was not provided. In future studies submitted to the EPA, it is necessary that the soil series name(s) be reported.

6. Data were reported only as percentages of the applied radioactivity and were not reported in units of concentration. In future studies submitted to the EPA, it is necessary that data also be reported in units of concentration, such as ppm.

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