

3/3/99

DATA EVALUATION RECORD
DER 3

SHAUGHNESSY No. 417300
COMMON NAME: Glyphosate
CHEMICAL NAME: N-(phosphono-methyl) glycine
FORMULATION: Active Ingredient
DATA REQUIREMENT: (162-3)

MRID No:44125718 (Interim Report)
John Mao, September 17, 1996, Glyphosate Acid - Determination of Aquatic Metabolism Under Anaerobic Conditions. Sponsored by Industria Prodotti Chimici S.P.A. Via F. Beltrami 11-20026 Novate Milan, Italy. Performed by Springborn Laboratories, Inc. Health and Environmental Sciences, 790 Main Street, Wareham, Massachusetts 02571-1075. SLI Study #13582.0795.6101.755; #96-6-6536.

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CONCLUSIONS:

1. Study MRID #44125718 is acceptable and completely satisfies the anaerobic aquatic metabolism (162-3) data requirement for glyphosate.
2. [C^{14}]glyphosate [N-phosphonomethyl glycine, radiochemical purity 95.9%] at 2.78 ppm, degraded with a first order half-life of 203 days ($r^2 = 0.8580$) in anaerobic (flooded plus nitrogen atmosphere) Ohio clay loam sediment that was incubated in the dark at 25 ± 1 C for 181 days. In general, parent glyphosate and its major degradate aminomethyl phosphonic acid (AMPA) was associated with the sediment phase and was mineralized. Glyphosate was dosed in the aqueous phase but approximately 85% of the applied dose was found in the sediment a few hours after dosing (time 0). The only degradate identified in the sediment and water fraction was AMPA. AMPA was at almost 10% of applied at time 0 and increased to a total maximum average of 21.2% of applied by day 181. [C^{14}]CO₂ accounted for approximately 22.4% of the applied by day 181. Material balances ranged from 88.3% to 103% of the applied and averaged $98 \pm 6\%$ (n=20) for all samples (Table 6).

MATERIALS AND METHODS:

Samples (50 g dry weight) of sieved (2 mm) clay loam sediment (28% sand, 42% silt, 30% clay, 3.4% organic matter, pH 7.7) were placed in 250 ml flasks and flooded with 100 ml of pond water (total alkalinity 88, total hardness 320, both as mg/l CaCO₃, dissolved O₂ 8.7 mg/l at 21 C, specific conductivity 500 umhos/cm). Anaerobic conditions were established before the dosing by continuously flushing of the flasks with nitrogen for 24 days. [¹⁴C]Glyphosate (radiochemical purity 95.5%, specific activity 54 mCi/mmol), at 5.55 ppm, was added to the flasks then incubated in an environmental chamber at a target temperature of 25 C (conditions not described). Duplicate samples were taken on days 0, 1, 3, 7, 14, 30, 60, 90, 120 and 181. The water was decanted and submitted for LSC, triplicate samples of sediment were removed for pre-extraction combustion. A single sample was removed for extraction and the remaining contents stored in a freezer (-4C). The ethylene glycol and potassium hydroxide trapping solutions were sampled at each interval except day 0. At each sampling interval two sets of quality control samples were extracted and analyzed for the water and the sediment phases. Blank pond sediment and water samples were also extracted and analyzed at each interval.

Sediment was extracted sequentially twice with 0.5 N sodium hydroxide then the supernatant was transferred to a membrane filter, centrifuged, then adjusted to pH 7 with phosphoric acid. The two extracts were analyzed separately by LSC and HPLC. Quantification of radioactivity associated with bound residues were accomplished by oven drying the samples, combustion (Packard model 306 Tri-carb Oxidizer) and analysis by LSC.

RESULTS:

[¹⁴C]glyphosate [N-phosphonomethyl glycine, radiochemical purity 95.9%] at 2.78 ppm, degraded with a first order half-life of 209 days ($r^2 = 0.8580$) in anaerobic (flooded plus nitrogen atmosphere) Ohio clay loam sediment that was incubated in the dark at 25 ± 1 C for 181 days. In general, parent glyphosate and its major degradate aminomethyl phosphonic acid (AMPA) was associated with the sediment phase and was mineralized. Glyphosate was dosed in the aqueous phase but approximately 85% of the applied dose was found in the sediment a few hours after dosing (time 0). In sediment glyphosate decreased from 85% at time 0 to 65% at 14 days, then decreased slowly to approximately 45% by day 180 (figure 14). The only degradate identified in the sediment and water fraction was AMPA. AMPA was at almost 10% of applied at time 0 and increased to a total maximum average of 21.2% of applied by day 181. AMPA was associated with the sediment phase (Table 6, Figure 13,14). One unidentified compound noted as an impurity was present at an average of 4 to 5% throughout the study. [¹⁴C]CO₂ accounted for approximately 22.4% of the applied by day 181. Sediment bound residues were <10% for day 0 through 30 samples and exceeded 10% for day 60 through 181. These

residues were further characterized in humic, fulvic acid and humin fractions (Table 10). The redox potential of the treated systems increased from -135.8 (day 2) to -16.8 mV by day 181. The pH values ranged from 7.41 to 7.93 (Table 4). Temperatures ranged between 23.2 to 26.7 C. Material balances ranged from 88.3% to 103% of the applied and averaged $98 \pm 6\%$ (n=20) for all samples (Table 6).

DISCUSSION:

1. This study is ongoing and will include two more samplings at day 270 and 360. The current data set is from the first 181 days of the study.
2. Anaerobic conditions were established before study initiation by flushing the water/sediment systems for 24 days with nitrogen. Oxidation-reduction measurements were confirmed by an Orion model 96-78-00 redox electrode and an Orion Model EA 940 ion analyzer. Redox measurements were taken at multiple intervals by lowering the redox probe directly into the incubation flasks. Monitoring of pH was performed concurrently.
3. One day before test initiation and monthly thereafter, microbial plate counts using the pour plate method were performed to estimate the size of the anaerobic microbial community associated with the sediment.
4. Since bound residues were present at >10% of applied, a fulvic acid/humin fraction was performed at day 60 through day 181. The extracted soil was placed in a centrifuged tube with 0.1 N NaOH/CaCl₂, then shaken overnight, centrifuged and decanted. The remaining soil was dried and weighed and adjusted to pH 1 with con HCL. The contents were then centrifuged and the precipitate representing the humic acid fraction was filtered out. The supernatant representing the fulvic acid portion was also collected. LSC analysis was performed on the fulvic acid, humic acid and the remaining soil, humin portions.
5. The method detection limit was 0.0017 ppm in water samples and sediment extracts; in sediment it was 0.017 ppm. The quantitation limit was defined as 2X the detection limit.
6. Total [¹⁴C]residues (Table 6, Figure 2) partitioned into the various phases were given rather than individual parent glyphosate and AMPA. The fractionation of glyphosate into the aqueous and sediment phases was presented in Figure 14, but the major degradate AMPA was not addressed.
7. It was not clear whether there was a control sediment/water system completed concurrently with the test study. There was no mention of a control system when discussing redox potentials or pH. Dissolved oxygen content was also not reported.
8. The half-life was recalculated using a linear regression by

method of least squares. The y intercept was -0.376 the rate constant was 0.00331 (half-life 209 days) and the correlation coefficient was 0.8580 . There were two references in the study to a half-life of 199 days.

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