DATA EVALUATION RECORD

STUDY 7

CHEM 417300	Glyphosate acid	§164-1

CAS No. 1071-83-6
FORMULATION--06--WETTABLE POWDER

STUDY ID 44320648
Grant, C. L., and Y. Iwata. 1996. Glyphosate acid: residue levels in soil after applications to turf and to bare soil for trials conducted in Mississippi during 1995-1996. Laboratory Project ID: ZPMG-95-SD-01. Unpublished study performed by ZENECA Agricultural Products, Richmond, CA; and submitted by ZENECA Ag Products, Wilmington, DE.

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CONCLUSIONS

Field Dissipation - Terrestrial

1. This study is scientifically valid and provides useful information on the terrestrial field dissipation of glyphosate acid on bareground and turf plots of loam soil in Mississippi. However, sample collection was not conducted using random sampling, and data variability may have been altered relative to that which would have otherwise been observed. Also, turf sample data were incomplete, and reported data were questionable due to an inadequate analytical method used for analysis of the plant samples.

2. This study does not meet Subdivision N Guidelines for the partial fulfillment of EPA data requirements on terrestrial field dissipation for the following reasons:

   (i) the analytical method was inadequate for the analysis of the parent and degradate in turf in samples;

   (ii) storage stability data were inadequate;

   (iii) samples were not collected randomly;

   (iv) application rates were not verified; and

   (v) turf sample data were incomplete.

3. Glyphosate acid (PMG; ZPMG 50WP) broadcast applied twice (28-day interval) at a nominal application rate of 4.0 lb a.i./A/application on bareground and turf plots of loam soil, dissipated with registrant-calculated half-lives of 3.9 and 1.4 days (reported as DT_{50}'s) following the second of two applications, respectively. Following the first application to the bareground plot, the parent was initially present in the 0- to 6-inch depth at 1.0 ppm and decreased to 0.49 ppm by 25 days (three days prior to the second application). The major degradate AMPA was present in the 0- to 6-inch depth at 0.12 ppm at 25 days (three days prior to the second application). Following the second application, the parent was initially present in the 0- to 6-inch depth at 2.3 ppm, decreased to 1.3 ppm by 1 day posttreatment, was 1.01-1.20 ppm at 3-11 days, was 0.55-0.56 ppm at 28-52 days, and was last detected at 0.13 ppm at 149 days; the parent was not detected below the 0- to 6-inch depth. The major degradate AMPA was initially present in the 0- to 6-inch depth at 0.18 ppm, increased with variability to 0.44 ppm by 52 days, and was 0.22 at 319 days posttreatment. The degradate AMPA was not detected below the 0- to 6-inch depth.

   Following the first application to the turf plot, the parent was initially present in the 0- to 6-inch depth at 0.11 ppm and was 0.12 ppm at 25 days (three days prior to the second application). The major degradate AMPA was detected in the 0- to 6-inch depth at a concentration of 0.17 ppm at 25 days (three days prior to the second application). Following the second application, the parent was initially present in the 0- to 6-inch depth at 0.86 ppm, decreased to 0.50 ppm by 1 day posttreatment, and was last detected at 0.13 ppm at 11 days. The major degradate AMPA was present in the 0- to 6-inch depth at 0.22 ppm at time 0, increased to a maximum of 0.37 ppm by 4 days, decreased to 0.21 ppm by 28 days, and was last detected at 0.08 ppm at 149 days. The parent and the degradate AMPA were not detected below the 0- to 6-inch depth.

Following the first application, the parent compound was initially present in the turf at 17 ppm and was 1.1 ppm at 25 days (three days prior to the second application). The major degradate AMPA was present in the turf at 0.26 ppm at time 0, and was 0.62 ppm at 25 days (three days prior to the second application). Following the second application, the parent compound was initially present in the turf at 68 ppm and was 48 ppm at 1 day posttreatment, the last sampling interval for the turf. The major degradate AMPA was present in the turf at 3.7 ppm at time 0, and was 11 ppm at 1 day posttreatment, the last sampling interval for the turf.

METHODOLOGY

Glyphosate acid (ZPMG 50WP) was broadcast applied twice (28-day interval) as a spray at a nominal rate of 4.0 lb a.i./A/application, onto one bare soil plot (66.5 x 100 ft with three subplots 13.3 x 120 ft, 0-0.25% slope; pp. 11, 14, 94) of loam soil (45% sand, 45% silt, 10% clay, 0.8% organic matter, pH 6.1, CEC 6.7 meq/100 g; p. 12) and one Bermuda turf plot (40 x 120 ft with three equivalent-size subplots, 0% slope; p. 11) of loam soil (51% sand, 36% silt, 14% clay, 1.0% organic matter, pH 7.1, CEC 10.0 meq/100 g) near Leland, Mississippi. Plot histories were not reported (see Comment #7). The bareground plot was treated six times with paraquat (Gramoxone Extra®, 2.0-3.0 pt./A or 2.0%) and once each with clethodim (Select®, 10 oz./A), propanil (Stam®, 6 lb/A), monosodium methylarsonate (6.0%) and bromoxynil (Buctril®, 1.0%) to maintain bareground conditions (p. 52). Untreated bareground (13.3 x 400 ft) and turf (13.3 x 120 ft) control plots, were located 30 and 40 feet from the treated plots, respectively (pp. 13, 14). The mean depth to the water table was 14 feet (p. 11). Precipitation was supplemented with irrigation; total water input (53 inches) was 100% of the 30-year average (p. 13). Pan evaporation data were not reported.

The application rate was not confirmed using monitoring pads or a similar method (see Comment #5). Recovery of the parent from the bareground and turf plots immediately following the first application was 48.2% and 49.9% of the nominal application rate (4.0 lb a.i./A/application), respectively (p. 24). Soils were sampled 7 days prior to the first application; 0 and 25 days (three days prior to the second application) following the first application; and 0, 1, 3, 4, 7, 11, 28, 52, 149, and 319 days following the second application (p. 14). Five soil cores were collected from transects extending diagonally across each treated subplot at both sites (15 soil cores per plot); ten soil cores were collected from the control plots at each interval (p. 100; see Comment #4). A three-phase sampling was performed using a hydraulic probe with a butyrate liner to collect a 0- to 6-inch depth core (2.0 inch i.d.), a 6- to 24-inch depth core (1.25 inch i.d.), and a 24- to 48-inch depth core (1.25 inch i.d.; pp. 96-99); soil cores collected immediately following each application were from the 0- to 6-inch depth only (pp. 14-16). Samples were shipped frozen overnight to the processing laboratory and stored frozen until processing. Soil samples were sectioned into 6-inch increments with the turf removed from the 0- to 6-inch turf plot samples and the sections composited by depth and subplot (five samples per composite; pp. 15,16).

Soil samples were analyzed for the parent compound N-(phosphonomethyl)glycine (glyphosate acid; PMG) and its degradate (aminomethyl)phosphonic acid (AMPA). Samples (20 g) of homogenized soil were extracted by shaking with 0.25 M ammonium hydroxide plus 0.10 M potassium phosphate and centrifuged (p. 231). An aliquot of the extract was shaken with activated carbon and filtered. The analytes in the purified aqueous extract were derivatized (p. 228) by the addition of trifluoroacetic anhydride and heptafluorobutanol. The derivatized extract was evaporated and the residue was reconstituted with ethyl acetate and analyzed by GC with mass-selective detection; the limit of quantitation was 0.005 ppm (p. 18).

Turf samples were analyzed for PMG and AMPA. Samples of turf were extracted by maceration with chloroform plus 0.1 *N* HCl. The extract was purified, derivatized and analyzed by GC as described above for soil extracts; the limit of quantitation was 0.1 ppm (p. 18).

In a concurrent recovery study, samples of control soil were fortified with PMG and AMPA at 0.05-3.0 ppm and extracted and analyzed as described previously. Mean recoveries of PMG and AMPA were 78 ± 10% (5 of 56 recoveries outside 70-120%) and 85 ± 8%, respectively (Tables IIA, IIB; pp. 28-35). Mean recoveries from control turf samples fortified with PMG and AMPA at 0.1-50 ppm were 34 ± 14% (6 of 6 recoveries <70%) and 43 ± 13% (6 of 6 recoveries <70%), respectively (Tables IIIA, IIIB; pp. 36, 37).

In a frozen storage stability study, samples of untreated control soil from the bareground plot and turf samples were fortified with PMG and AMPA and stored frozen for up to 270 days prior to analysis (p. 19; see Comment #3). Recoveries of the parent and AMPA from soil were 65-82% and 78-97%, respectively, and did not exhibit a clear pattern of degradation; recoveries of the parent and AMPA from turf samples were 87-109% and 79-90%, respectively, and did not exhibit a clear pattern of degradation (Table VI, p. 42).

DATA SUMMARY

Glyphosate acid (PMG; ZPMG 50WP) broadcast applied twice (28-day interval) at a nominal application rate of 4.0 lb a.i./A/application on bareground and turf plots of loam soil, dissipated with registrant-calculated half-lives of 3.9 and 1.4 days (reported as DT_{50}'s) following the second of two applications, respectively (p. 21; Figures 1, 2; pp. 43, 44).

Bareground Plot

Following the first application to the bareground plot, the parent was initially present in the 0- to 6-inch depth at 1.0 ppm and decreased to 0.49 ppm by 25 days posttreatment (three days prior to the second application; Table IVA, p. 38); the parent was not detected below the 0- to 6-inch depth. Following the first application, the major degradate

(aminomethyl)phosphonic acid (AMPA)

was present in the 0- to 6-inch depth at 0.12 ppm at 25 days posttreatment and was not detected below the 0- to 6-inch depth (three days prior to the second application; Table IVB, p. 39).

Following the second application, the parent was initially present in the 0- to 6-inch depth at 2.3 ppm, decreased to 1.3 ppm by 1 day posttreatment, was 1.01-1.20 ppm at 3-11 days, was 0.55-0.56 ppm at 28-52 days, and was last detected at 0.13 ppm at 149 days posttreatment; the parent was not detected below the 0- to 6-inch depth (Table IVA, p. 38). Following the second application, the major degradate

AMPA

was initially present in the 0- to 6-inch depth at 0.18 ppm, was 0.26-0.33 ppm at 1-28 days, was a maximum of 0.44 ppm at 52 days, and decreased to 0.22 ppm by 319 days posttreatment (Table IVB, p. 39); the degradate AMPA was not detected below the 0- to 6-inch depth.

Turf Plot

Following the first application to the turf plot, the parent was initially present in the 0- to 6-inch depth at 0.11 ppm and was 0.12 ppm at 25 days posttreatment (three days prior to the second application; Table VA, p. 40). The major degradate

(aminomethyl)phosphonic acid (AMPA)

was detected in the 0- to 6-inch depth at 0.17 ppm at 25 days posttreatment (three days prior to the second application).

Following the second application, the parent was initially present in the 0- to 6-inch depth at 0.86 ppm, decreased to 0.50 ppm by 1 day posttreatment, was 0.22 at 7 days, and was last detected at 0.13 ppm at 11 days; the parent was not detected below the 0- to 6-inch depth (Table VA, p. 40; see Comment #9). The major degradate

AMPA

was present in the 0- to 6-inch depth at 0.22 ppm at time 0, increased to a maximum of 0.37 ppm by 4 days, decreased to 0.21 ppm by 28 days, was 0.16 ppm at 52 days, and was last detected at 0.08 ppm at 149 days posttreatment; the degradate AMPA was not detected below the 0- to 6-inch depth (Table VB, p. 41).

Following the first application, the parent compound was initially present in the turf samples at 17 ppm and was 1.1 ppm at 25 days posttreatment (three days prior to the second application; Table VA, p. 40). However, the analytical method was unreliable for the analysis of the parent and degradates in turf samples (see Comment #2). The major degradate

(aminomethyl)phosphonic acid (AMPA)

was initially present in the turf at 0.26 ppm, and was 0.62 ppm at 25 days posttreatment (three days prior to the second application; Table VB, p. 41). Following the second application, the parent compound was initially present in the turf at 68 ppm and was 48 ppm at 1 day posttreatment, the last sampling interval for the turf (Table VA, p. 40). The major degradate

AMPA

was initially present in the turf at 3.7 ppm, and was detected at 11 ppm at 1 day posttreatment, the last sampling interval for the turf (Table VB, p. 41).

COMMENTS

1. The reviewer could not confirm the registrant-calculated half-lives because complete raw data (i.e., replicate data) were not reported as required by Subdivision N Guidelines. The registrant calculated half-lives using both a first-order multi-compartment (FOMC) model and a simple exponential model based on mean data (p. 21; pp. 43-44). The half-lives based on the FOMC model were 3.9 and 1.4 days, respectively, for the bareground and turf plots; respective half-lives were determined to be 32.2 and 4.6 days in the bareground

and turf plots using the exponential model. The FOMC model most closely fit the observed half-lives of 3-7 days and 1-3 days in the bareground and turf plots, respectively.

2. The analytical method was unreliable for the analysis of the parent and degradates in turf samples. Mean recoveries from control turf samples fortified with PMG and AMPA at 0.1-50 ppm were 34 ± 14% (6 of 6 samples <70% recovery) and 43 ± 13% (6 of 6 samples <70%), respectively (Tables IIIA, IIIB; pp. 36, 37).

3. Storage stability data were inadequate. Soil samples were stored frozen for 38-287 days and turf samples were stored frozen for 403-436 days prior to analysis (p. 19). Storage stability data were only reported through 270 days of storage. The study authors stated that the data reported were part of an on-going 3-year study. It is necessary that the registrant submit the remainder of the data so that a complete analysis of the storage stability of the parent and degradate can be conducted.

4. Samples were not collected randomly as required by Subdivision N Guidelines. Soil cores were collected from transects extending diagonally across the subplots at both trial sites in a systematic fashion (p. 100). The study authors stated this sampling plan was designed to minimize variability between subplots; however, it was not random sampling (p. 14). Subdivision N Guidelines require that soil cores be collected randomly in order to accurately represent variability in the test plots and to minimize variability among data points.

5. The application rate of the parent to the bareground and turf plots was not verified by monitoring pads or other acceptable means. Recovery of the parent from the bareground and turf plots immediately following the first application was 48.2% and 49.9% of the expected, respectively, based on the nominal application rate (p. 24). A similar comparison following the second application was not made since residues of the parent were detected prior to the second application.

6. The reviewer was unable to confirm that the application rate utilized (4 lb a.i./A/application) was the highest recommended label rate for each application or for multiple applications. Clarification by the registrant may be necessary.

7. A plot history was not reported, so the reviewer could not determine if glyphosate acid or a related compound was applied to the test sites in the three years prior to the study. However, pre-treatment analysis of soil cores from the test sites detected no residues of the parent compound or degradate AMPA from 0- to 42-inches in depth (Tables IVA-B, VA-B; pp. 38-42).

8. Limits of detection were not reported. It is necessary that both limits of detection and limits of quantitation be reported so the reviewer can assess the adequacy of the method for the determination of the parent and degradates.

9. Analysis of the turf samples was discontinued after 1 day following the second application. The study authors stated that turf sample analysis was used only to estimate zero-time recovery (p. 19). Sampling was not conducted until patterns of formation and/or decline were determined. The study (turf) was terminated before a pattern of decline was observed (Tables VA, VB, pp. 40, 41). Turf samples should have been collected until the rate of dissipation of the parent material in turf was adequately assessed.

10. The study author stated that residue data for the degradate AMPA for the 18-month sampling interval would be included in a final report (p. 10); data were included in the interim report available to the reviewer. In addition, storage stability data reported in the present study were taken from an on-going 3-year storage stability study (p. 19). The study authors stated that the full report would be submitted at a later (unspecified) date.

11. The study was poorly written and poorly organized. The reviewer had to search for details and had to repeatedly skip back and forth between sections in an attempt to determine the details and sequence of the methodology.

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