DATA EVALUATION RECORD

STUDY 8

CHEM 417300 Glyphosate acid §164-1
CAS No. 1071-83-6
FORMULATION--06--WETTABLE POWDER

STUDY ID 44320649
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 California during 1995-1996. Laboratory Project ID: ZPMG-95-SD-01. Unpublished study performed by ZENECA Ag Products, Richmond, CA; and submitted by ZENECA Ag Products, Wilmington, DE.

STUDY ID 44320650
Szuter, S. 1996. Glyphosate acid: independent laboratory validation of the method for determining residues of N-(phosphonomethyl)glycine and (aminomethyl)phosphonic acid in soil. Laboratory Project ID: ZPMG-96-MT-01. Unpublished study performed by McKenzie Laboratories, Inc., Phoenix, AZ; and submitted by ZENECA Ag Products, Wilmington, DE.

DIRECT REVIEW TIME = 90 Hours

REVIEWED BY:	D. R. Hunt, B.A.	Signature:
TITLE:	Scientist	Date:
EDITED BY:	C. A. Sutton, Ph.D.	Signature:
TITLE:	Sr. Scientist/Asst. Project Manager	Date:
APPROVED BY:	P. H. Howard, Ph.D.	Signature:
TITLE:	Project Manager	Date:
ORG:	Syracuse Research Corp. Arlington, VA 22202	
TEL:	703/413-9369	

APPROVED BY:	Kevin Poff
TITLE:	Chemist
ORG:	FMB/EFED/OPP
TEL:	703/305-5318

SIGNATURE:

1

CONCLUSIONS

Field Dissipation - Terrestrial

1. This study is scientifically valid and provides useful information on the terrestrial field dissipation of glyphosate acid in sandy loam soil in California. 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.

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) soil and turf samples were not collected randomly;

   (ii) the reviewer could not confirm that the parent was applied at the maximum label rate;

   (iii) the application rate was not verified; and

   (iv) storage stability data were inadequate for turf samples.

3. Glyphosate acid (ZPMG 50WP), broadcast applied twice (28-day interval) at a nominal rate of 4.0 lb a.i./A/application to bareground and turf plots of Foster sandy loam soil, dissipated after the second of two applications with registrant-calculated half-lives of 19 days ($r^2 = 0.99$) and 12 days ($r^2 = 0.79$), respectively, in Tulare County, CA. Following application to the bareground plot, the parent was initially present in the 0- to 6-inch depth at 0.95 ppm and was 0.55 ppm at 28 days (immediately prior to the second application). Immediately following the second application, the parent was present in the 0- to 6-inch depth at 1.6 ppm, was a maximum of 1.74 ppm at 1 day posttreatment, decreased to 0.81 ppm by 21 days, and was last detected at 0.17 ppm at 64 days. The parent was not detected below the 0- to 6-inch soil depth following either application. The major degradate AMPA was initially present in the 0- to 6-inch depth at 0.15 ppm immediately prior to the second application (28 days following the first application), was present at 0.19 ppm immediately following the second application, increased to a maximum of 0.33 ppm by 14 days posttreatment, and was 0.13 ppm at 315 days; AMPA was not detected below the 0- to 6-inch soil depth.

   Following application to the turf plot, the parent was present in the 0- to 6-inch depth at 0.31 ppm immediately following the first pesticide application and was detected below the limit of quantitation (LOQ) by 28 days (immediately prior to the second application). Immediately following the second application, the parent was present in the 0- to 6-inch depth at 0.38 ppm, increased to a maximum of 0.59 ppm by 5 days posttreatment, decreased to 0.22 ppm by 14 days, and was last detected at 0.14 ppm at 21 days; the

parent was not detected below the 0- to 6-inch soil depth following either application. The major degradate AMPA was initially present in the 0- to 6-inch depth at 0.11 ppm immediately prior to the second application (28 days following the first application), was 0.12 ppm immediately following the second application, increased to a maximum of 0.19 ppm by 21 days posttreatment, and was last detected at 0.14 ppm at 28 days; AMPA was not detected below the 0- to 6-inch soil depth. The parent was present in the turf at 12 ppm immediately following the first application, was 3.7 ppm at 28 days following the first application (immediately prior to the second application), was present at a maximum of 20 ppm immediately following the second application, and was last detected at 13 ppm at 1 day following the second application. The major degradate AMPA was detected in the turf at 0.1 ppm immediately following the first application, was 0.74 ppm at 28 days following the first application, was present at 0.85 ppm immediately following the second application, and was last detected at a maximum of 0.87 ppm at 1 day following the second application.

METHODOLOGY

Glyphosate acid (ZPMG 50WP, 50% a.i.) was broadcast applied twice (28-day interval) as a spray at a nominal rate of 4.0 lb a.i./A/application (total application of 8.0 lb a.i./A) onto three bareground subplots (10 x 160 ft each, 0% slope; p. 11; Appendix A, p. 97; see Comment #12) of Foster sandy loam soil (65% sand, 23% silt, 12% clay, 1.1% organic matter, pH 8.3, CEC 6,5 meq/100 g; p. 12; see Comment #6) and onto three subplots (10 x 120 ft each, 0% slope) of Foster sandy loam soil (76% sand, 14% silt, 10% clay, 1.0% organic matter, pH 7.5, CEC 4.9 meq/100 g) planted with Blue Flyking Tall Fescue turf in Tulare County, California (Appendix A, p. 78). The three bareground subplots were located adjacent to each other (10 feet apart); an untreated control plot (10 x 160 ft) was located 20 ft from the treated bareground plots (p. 13; Appendix A, p. 96). The turf plots were a reviewer-calculated 45 feet from the bareground plots (see Comment #7); the three turf subplots were located adjacent to each other (10 feet apart) and an untreated control plot (10 x 120 ft) was located 20 feet from the treated turf plots (p. 14; Appendix A, p. 97). The height of the mature grass (turf) was 1.5-1.8 inches prior to the first application and the height of the turf (dead from the previous spraying) was 1.0 inch prior to the second application (Appendix A, p. 82). The bareground plot was treated four times with Gramoxone Extra® (paraquat; 1%), and three times with Gramoxone Extra® and Triton X-77® (surfactant, 0.125%) throughout the study period (Appendix A, p. 52). The turf plot was treated six times with Gramoxone Extra® (0.625-1.0%) and Triton X-77® (0.125%), and once with Vapam® (metam-sodium; 1 gal/60 ft^2) throughout the study period (Appendix A, p. 79). The yearly mean depth to the water table at the bareground and turf plots was 20 ft (p. 11). Precipitation was supplemented with irrigation; total water input (45.4 inches; reviewer-calculated) was 460% of the 30-year mean annual precipitation for both plots (Appendix A, pp. 63, 64, 90, 91, 109). Pan evaporation data were not reported. Soils were sampled 1 day prior to application, at 0 and 28 days

following the first application, and at 0, 1, 3, 5, 7, 14, 28, 64, 153, and 315 days after the second application (p. 14). At each sampling interval, five soil cores were collected from each of the treated subplots (15 cores total) along transects extending diagonally across the subplots and ten cores were collected from the control plot (Appendix A, pp. 101, 102; see Comment #1); a 0- to 6-inch depth sample (2-in diameter), 6- to 24-inch depth sample (1.3-in diameter), and 24- to 42-inch depth sample (1.3-in diameter) were collected using a three-stage tractor-mounted hydraulic probe with zero-contamination butyrate liners (p. 15; Appendix A, pp. 98-100). Samples were transported to the analytical lab on ice and transferred to a storage freezer (-20 ± 5 °C; Appendix C, p. 202) within 2 hours of collection (p. 16); all samples were placed on ice within 10-15 minutes of field collection (Appendix A, pp. 62, 89). At the analytical lab, soil samples were sectioned into 6-inch increments and composited by depth (five samples per composite; p. 16).

The application rate was not verified using monitoring pads or similar methods. In the 0- to 6-inch soil depth of the bareground plot, the parent concentration was 53.4% of the concentration expected following the first application based on the nominal application rate (pp. 22, 23). In the turf and 0- to 6-inch soil depth of the turf plot, the parent concentration was 41.8% (13.7% in soil and 28.1% in turf) of the concentration expected following the first application based on the nominal application rate (pp. 24, 25).

Soil samples were analyzed for the parent material (PMG, glyphosate acid) and the degradate (aminomethyl)phosphonic acid (AMPA; pp. 16, 17). Soil samples (20 g) were extracted by shaking with a solution of 0.25 M ammonium hydroxide plus 0.10 M potassium phosphate (monobasic; Appendix D, p. 225). An aliquot of the extract was shaken with activated carbon and filtered. An aliquot of the cleaned extract was derivatized using a mixture of trifluoroacetic anhydride and heptafluorobutanol, concentrated by evaporation, and redissolved in ethyl acetate prior to GC analysis (mass selective detection). The limit of quantitation for GC/MS analysis was 0.05 ppm. Turf samples were extracted by blending with a chloroform:0.1 *N* hydrochloric acid mixture (ratio not reported). The cleaned aqueous extract was derivatized and analyzed as described previously; the limit of quantitation was 0.1 ppm (p. 18).

To determine concurrent recoveries, control soil samples from the untreated plot were fortified with glyphosate acid and AMPA at 0.05-2.0 ppm, and control turf samples from the untreated turf plot were fortified with glyphosate acid and AMPA at 0.1-50 ppm (p. 18). Mean recoveries of the parent and AMPA from soil were 85% ± 9% (range of 73-116%) and 87% ± 9% (range of 73-110%), respectively (Tables IIA, IIB; pp. 31-37). Mean recoveries of the parent and AMPA from turf samples were 64% ± 9% (range of 53-78%; 6 of 8 samples < 70% recovery) and 67% ± 7% (range of 54-71%; 2 of 6 samples < 70%, respectively (Tables IIIA, IIIB; pp. 38, 39; see Comment #10).

In a storage stability study, samples of untreated soil and turf were fortified with glyphosate acid and AMPA and stored frozen for up to 273 days (conditions not reported). Recoveries of the parent and AMPA from soil samples stored frozen for up to 273 days were 76-97% and 64-82%, respectively, with no clear pattern of degradation (Table VI, p. 44; see Comment #8). Recoveries of the parent and AMPA from turf samples stored frozen for up to 269 days were 79-90% and 87-109%, respectively, with no clear pattern of degradation. Soil samples (from bareground and turf plots) and turf samples were stored frozen for 36-258 days and 393-420 days, respectively (p. 19; Appendix B, Table B.VIIA.-B.VIIIB., B.XA., B.XB.; pp. 168-181, 189, 190).

Independent Method Validation MRID 44320650

Five replicate soil samples (20 g) were each fortified with glyphosate acid or AMPA at 0.025 ppm, 0.05 ppm, and 0.5 ppm (p. 11; see Comment #9). The soil samples were extracted and analyzed as described previously for the field study. Mean recoveries of the parent compound at 0.025 ppm, 0.05 ppm, and 0.5 ppm were 80% ± 9.4%, 84% ± 14% (1 of 5 samples <70% recovery), and 80% ± 10%, respectively (Table III, p. 18). Mean recoveries were 110% ± 8.7% (1 of 5 samples >120%), 89% ± 4.0%, and 78% ± 6.8%, respectively, for soil fortified with AMPA at 0.025 ppm, 0.05 ppm, and 0.5 ppm (Table III, p. 19).

DATA SUMMARY

Bareground Plot

Glyphosate acid (ZPMG 50WP), broadcast applied twice (28-day interval) at a nominal rate of 4.0 lb a.i./A/application, dissipated after the second of two applications with a registrant-calculated half-life of 19 days ($r^2 = 0.99$) in Foster sandy loam soil in Tulare County, CA (p. 21; Figure 1, p. 45). The parent was present in the 0- to 6-inch depth at 0.95 ppm immediately following the first pesticide application and was 0.55 ppm at 28 days (immediately prior to the second application). Immediately following the second application, the parent was present in the 0- to 6-inch depth at 1.6 ppm, was a maximum of 1.74 ppm at 1 day posttreatment, decreased to 0.81 ppm by 21 days, and was last detected at 0.17 ppm at 64 days posttreatment (Table IVA, p. 40). The parent was not detected below the 0- to 6-inch soil depth following either application. The major degradate

(aminomethyl)phosphonic acid (AMPA; p. 11)

was initially present in the 0- to 6-inch depth at 0.15 ppm immediately prior to the second application (28 days following the first application), was present at 0.19 ppm immediately following the second application, increased to a maximum of 0.33 ppm by 14 days

posttreatment, and was 0.13 ppm at 315 days posttreatment; AMPA was not detected below the 0- to 6-inch soil depth (Table IVB, p. 41).

Turf Plot

Glyphosate (ZPMG 50WP), broadcast applied twice (28-day interval) at a nominal rate of 4.0 lb a.i./A/application, dissipated after the second of two applications with a registrant-calculated half-life of 12 days ($r^2 = 0.79$) in Foster sandy loam soil planted with turf in Tulare County, CA (p. 21; Figure 2, p. 46). The parent was present in the 0- to 6-inch depth at 0.31 ppm immediately following the first pesticide application and was detected below the limit of quantitation (LOQ) by 28 days (immediately prior to the second application). Immediately following the second application, the parent was present in the 0- to 6-inch depth at 0.38 ppm, increased to a maximum of 0.59 ppm by 5 days posttreatment, decreased to 0.22 ppm by 14 days, and was last detected at 0.14 ppm at 21 days posttreatment; the parent was not detected below the 0- to 6-inch soil depth following either application (Table VA, p. 42). The major degradate

(aminomethyl)phosphonic acid (AMPA; p. 11)

was initially present in the 0- to 6-inch depth at 0.11 ppm immediately prior to the second application (28 days following the first application), was 0.12 ppm immediately following the second application, increased to a maximum of 0.19 ppm by 21 days posttreatment, and was last detected at 0.14 ppm at 28 days posttreatment; AMPA was not detected below the 0- to 6-inch soil depth (Table VB, p. 43).

The parent was present in the turf at 12 ppm immediately following the first application, was 3.7 ppm at 28 days following the first application (immediately prior to the second application), was present at a maximum of 20 ppm immediately following the second application, and was last detected at 13 ppm at 1 day following the second application (Table VA, p. 42). The major degradate AMPA was detected in the turf at 0.1 ppm immediately following the first application, was 0.74 ppm at 28 days following the first application, was present at 0.85 ppm immediately following the second application, and was last detected at a maximum of 0.87 ppm at 1 day following the second application (Table VB, p. 43).

COMMENTS

1. Soil and turf samples were not randomly collected. The study authors reported that soil cores were collected in a particular pattern to minimize variability between subplots (p. 14; Appendix A, p. 102). Subdivision N Guidelines require, and sound scientific practice dictates, that samples be collected randomly.

2. The reviewer could not confirm that the parent compound was applied at the highest recommended label rate. Subdivision N Guidelines require that terrestrial field dissipation studies be conducted at the maximum label rate in order to determine the dissipation half-life of the parent at that level. Clarification by the registrant is necessary. The reviewer notes, however, that the target application rates were not achieved (see Comment #3).

3. Confirmation of the application rate was not performed. Typically, application monitoring pads or similar devices of a known surface area are utilized to confirm the application rate. The study authors reported that the concentration of the parent in the soil of the bareground plot and in the soil and turf of the turf plot was 53.4% and 41.8% of the expected concentrations, respectively, following the first application (pp. 23-25).

4. Plot history data were not reported which demonstrated that the parent or related compounds were not used in the three years prior to the study. Subdivision N Guidelines require that a three-year plot history, at a minimum, be reported; however, the reviewer noted that the parent and degradate AMPA were not present in pretreatment soil samples (pp. 8, 9; Tables IVA-VB; pp. 40-43).

5. Frozen storage stability data were inadequate for the turf samples. Storage periods for the turf samples were 393-420 days (p. 19). Frozen storage stability data (taken from an on-going 3-year study) were only reported up to 269 days (Table VI, p. 44). The study authors stated that there was no indication that additional storage would result in significant degradation of the parent or degradate AMPA. Storage stability samples should be conducted using samples collected from the test site, fortified separately with the parent and degradates, and stored for a length of time equal to the longest storage interval utilized for the test samples.

6. Soil characterization data for the bareground and turf plots (p. 12) were the mean of three samples taken adjacent to each of the plots (Appendix A, pp. 103-106). The reviewer noted that only one sample was collected within the bareground or turf subplots.

7. The reviewer-calculated distance of 45 feet between the bareground and turf control plots was determined using plot maps located in Appendix A (pp. 96, 97, 103, 104). The reviewer noted that the plot representation in Appendix A (p. 97) is misleading because the bareground plot (designated as 02-CA-95-161) is not accurately depicted with respect to specified landmarks (irrigation valves).

8. The study authors stated that the residue data for the major degradate AMPA in soil at 18 months following the second application had not yet been collected, and that based on available data, the expected residues would have been below the LOQ (<0.05 ppm; p. 10). The reviewer noted that AMPA was last detected at 0.13 ppm (315 days following the second application) in the bareground plot (Table IVB, p. 41). The study authors

7

stated that the present report (MRID 44320649) was an interim report and that a final report would be submitted in the future. The reviewer did not have access to the final report at the time of this review.

9. The soils used in the independent method validation (MRID 44320650) were collected from the untreated control soil subplot of the bareground plot of the present study (MRID 44320649).

10. The study authors stated that low mean recoveries of the parent and the degradate AMPA (64% and 67%, respectively) from the fortified turf samples may have been due to the soil associated with the root zone of the turf samples (p. 19).

11. The study authors inadvertently reported that the mean percentage sand for the Site No. LOF09a-LOF09d was 13% (Appendix A, p. 106); the reviewer-calculated mean was 73%.

12. The reviewer noted that it was reported in Appendix A (p. 51) that the bareground subplots were 120 feet in length; dimensions for the bare soil subplots reported in the "Test Method" section (p. 13) and a plot map (Appendix A, p. 96) were 10 x 160 ft.

13. The reviewer noted that the treated subplots were true replicate subplots separated by 10-ft buffer zones (Appendix A, pp. 96, 97).

Page _____ is not included in this copy.

Pages 9 through 61 are not included in this copy.

The material not included contains the following type of information:

_____ Identity of product inert ingredients.

_____ Identity of product impurities.

_____ Description of the product manufacturing process.

_____ Description of quality control procedures.

_____ Identity of the source of product ingredients.

_____ Sales or other commercial/financial information.

_____ A draft product label.

_____ The product confidential statement of formula.

_____ Information about a pending registration action.

X FIFRA registration data.

_____ The document is a duplicate of page(s) _____.

_____ The document is not responsive to the request.

The information not included is generally considered confi by product registrants. If you have any questions, please the individual who prepared the response to your request.