DATA EVALUATION RECORD

STUDY 6

CHEM 417300 Glyphosate acid §163-1
CAS No.1071-83-6
FORMULATION--00-ACTIVE INGREDIENT

STUDY ID 44320647
Muller, K. and M. C. G. Lane. 1996. Glyphosate acid: adsorption and desorption properties of the major metabolite, AMPA, in soil. Laboratory Project ID: 96JH001. Unpublished study performed by ZENECA Agrochemicals Ltd., Berkshire, UK; and submitted by ZENECA Ag Products, Wilmington, DE.

DIRECT REVIEW TIME = 53.5 Hours

| | | |
|---|---|---|
| REVIEWED BY: | J. M. Franti, B.S. | Signature: |
| TITLE: | Scientist | Date: |
| EDITED BY: | B. S. Perkovich, M.E.M. | Signature: |
| TITLE: | Scientist | Date: |
| APPROVED BY: | P. H. Howard, Ph.D. | Signature: |
| TITLE: | Project Manager | Date: |
| ORG: | Syracuse Research Corp.<br>Arlington, VA 22202 | |
| TEL: | 703/413-9369 | |

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

SIGNATURE:

1

## CONCLUSIONS

### Mobility - Leaching & Adsorption/Desorption

1. The study is scientifically valid and provides useful information on the soil mobility (batch equilibrium) of the glyphosate acid degradate AMPA in five soils.

2. This study does not meet Subdivision N Guidelines for the partial fulfillment of EPA data requirements on soil mobility (batch equilibrium) for the following reasons:

   (i) complete material balance data were not reported for three of the soils;

   (ii) soils were sterilized by gamma irradiation prior to use, but data for reference chemicals of known mobility were not submitted; and

   (iii) $K_{des}$ values were not reported.

3. Nonradiolabeled plus radiolabeled [$^{14}$C]AMPA, at nominal concentrations of 0.05-2.0 $\mu$g/mL, was studied in sandy loam (Visalia), British sandy loam (18 Acres), British sand, silty clay loam (Champaign), and British silty clay loam (Wisborough Green) soil:solution slurries (1:10, w:v) that were equilibrated for 21 hours at 20 ± 2°C. Freundlich $K_{ads}$ values were 10.0 for the sandy loam soil (Visalia), 74.2 for the sandy loam soil (18 Acres), 133 for the sand soil (0.5% O.M.), 237 for the silty clay loam soil (Champaign), and 509 for the silty clay loam soil (Wisborough Green, 3.9% O.M.); corresponding $K_{oc}$ values were 1720, 4130, 45900, 11100, and 22500 mL/g. Respective 1/N values were 0.78, 0.84, 0.86, 0.86, and 0.91 for adsorption. Freundlich $K_{des}$ values were not reported. The reviewer calculated coefficient of determination ($r^2$) values for the relationships $K_{ads}$ vs. organic matter, $K_{ads}$ vs. pH and $K_{ads}$ vs. clay content (%) were 0.41, 0.84, and 0.40, respectively.

## METHODOLOGY

Based on preliminary studies of the adsorption of [$^{14}$C]AMPA (aminomethylphosphonic acid; radiochemical purity >97%, specific activity 1.828 Gbq/mmol; pp. 13, 14) to sandy loam (Visalia) soil (p. 9), an equilibration period of 21 hours was chosen for all soils (p. 30). In this preliminary study, the test compound was determined to be stable (p. 30); data were not reported.

For the adsorption phase of the definitive study, aliquots (19 mL) of 0.01 M $CaCl_2$ solution were added to Teflon® centrifuge tubes containing samples (2.0 g) of air-dried sieved (2 mm) sandy loam (Visalia), British sandy loam (18 Acres), British sand (Lillyfield), silty clay loam (Champaign), and British silty clay loam (Wisborough Green)

soils, and the samples were pre-equilibrated overnight on a shaker (pp. 10, 14; Table 2, p. 12; Figure 2, p. 17). The centrifuge tubes containing the pre-equilibrated soil samples were treated with nonradiolabeled plus [$^{14}$C]AMPA, dissolved in 0.01 M $CaCl_2$ (1 mL), at concentrations of 0.05, 0.1, 0.2, 1.0, and 2.0 $\mu$g/mL; four tubes were prepared for each soil type/treatment rate combination. The soil:solution slurries (1:10, w:v) were equilibrated by shaking for 21 hours at 20 ± 2°C. Following the adsorption equilibration period, soil:solution slurries were centrifuged and aliquots of the adsorption supernatant were analyzed for total radioactivity by LSC (p. 16); limits of quantitation and detection were not reported. The remaining supernatant and the soil from two of the four replicate samples were placed in frozen storage (-20 ± 5°C) prior to analysis.

For the desorption phase of the definitive study, an aliquot of pesticide-free 0.01 M $CaCl_2$ solution equivalent to the volume of the solution that was decanted following the adsorption phase was added to the soil pellets of two samples from the adsorption phase of the study (p. 18). The soil:solution slurries were equilibrated by shaking for 21 hours at 20 ± 2°C. Following equilibration, the soil:solution slurries were centrifuged and aliquots of the desorption supernatant were analyzed for total radioactivity by LSC; the remaining supernatant and soil were placed in frozen storage (-20 ± 5°C) prior to analysis.

To determine the stability of the test compound in solution, aliquots of the supernatant from the adsorption and desorption phases were concentrated under a stream of compressed air, and filtered (if necessary). Aliquots of the concentrated supernatant were analyzed by one-dimensional TLC performed on cellulose plates developed with 0.1 M HCl (p. 19).

To determine the stability of the test compound in soil, soil samples from the adsorption and desorption phases were extracted twice by shaking with ammonium phosphate buffer (p. 18). Samples were centrifuged, and the supernatants were decanted. Combined supernatants were homogenized by adding HCl, optiphase safe, and pentan-1-ol and analyzed by LSC. The supernatant was sonicated, and an aliquot was filtered, concentrated to dryness, redissolved in water and analyzed by TLC as described previously (p. 18).

Following extraction, air-dried soil samples were analyzed for total radioactivity by LSC following combustion; combustion efficiency was determined by combustion of a known quantity of radioactivity spiked into a combustion cone (p. 19). Data were corrected for the combustion efficiency.

## DATA SUMMARY

Nonradiolabeled plus radiolabeled [$^{14}$C]AMPA (radiochemical purity 97%), at nominal concentrations of 0.05, 0.1, 0.2, 1.0, and 2.0 $\mu$g/mL, was studied in sandy loam (Visalia),

British sandy loam (18 Acres), British sand, silty clay loam (Champaign), and British silty clay loam (Wisborough Green) soil:solution slurries (1:10, w:v) that were equilibrated for 21 hours at 20 ± 2°C. Fruendlich $K_{ads}$ values were 10.0 for the sandy loam soil (Visalia), 74.2 for the sandy loam soil (18 Acres), 133 for the sand soil (0.5% O.M.), 237 for the silty clay loam soil (Champaign), and 509 for the silty clay loam soil (Wisborough Green, 3.9% O.M.); corresponding $K_{oc}$ values were 1720, 4130, 45900, 11100, and 22500 mL/g (Table 5, p. 22). Respective 1/N values were 0.78, 0.84, 0.86, 0.86, and 0.91 for adsorption (Table 6, p. 23). The reviewer-calculated coefficient of determination ($r^2$) values for the relationships $K_{ads}$ vs. organic matter, $K_{ads}$ vs. pH, and $K_{ads}$ vs. clay content (%) were 0.41, 0.84, and 0.40, respectively. Freundlich $K_{des}$ values were not reported.

During the 21-hour adsorption period, 49.7-70.2% of the applied radioactivity was adsorbed to the sandy loam (Visalia) soil (for all replicates across all application levels; Table 5, p. 22), 89.7-94.5% of the applied was adsorbed to the sandy loam soil (18 Acres), 94.6-97.2% of the applied was adsorbed to the sand soil, 96.9-98.4% of the applied was adsorbed to the silty clay loam soil (Champaign), and 98.6-99.0% of the applied was adsorbed to the silty clay loam soil (Wisborough Green). Following a 21-hour desorption equilibration period, 17-36% of the adsorbed radioactivity was desorbed from the sandy loam (Visalia) soil (for all replicates across all application levels; Table 7, p. 25), 4-6% of the adsorbed radioactivity was desorbed from the sandy loam (18 Acres) soil, 2-3% of the adsorbed radioactivity was desorbed from the sand soil, 1-2% of the adsorbed radioactivity was desorbed from the silty clay loam (Champaign) soil, and 0-1% of the adsorbed radioactivity was desorbed from the silty clay loam (Wisborough Green) soil.

TLC analysis of aqueous and soil extracts from the adsorption and desorption phases demonstrated that there had been little or no degradation of AMPA, and greater than 90% of the radioactivity in the aqueous and soil phases was present as AMPA (p. 21). Data in the form of TLC chromatographs were reported for the sandy loam (Visalia) and silty clay loam (Champaign) soils (Appendix V, Figures 14-17, pp. 45-46).

Material balances (means; based on LSC analysis) were 99-106% for the sandy loam (Visalia) soil (across all application rates; Appendix II, Table 9, p. 32). Material balances of the remaining four soils were reported only for the samples treated at 1.0 $\mu$g/mL. Material balances (means; based on LSC analysis) were 97% for the sandy loam soil (18 Acres), 105% for the sand soil, 95% for the silty clay loam soil (Champaign), and 104% for silty clay loam soil (Wisborough Green; Appendix II, Table 10, p. 33).

COMMENTS

1. Material balances were reported across all application rates only for the sandy loam (Visalia) soil (Appendix II, Table 9, p. 32). Material balances for the remaining four soils

were reported only for the samples treated at 1.0 $\mu$g/mL (Appendix II, Table 10, p. 33). Complete material balance data for all soils across all application rates must be reported to allow the reviewer to account for all radioactivity throughout the study.

2. The study authors stated that all soils were sterilized by gamma irradiation prior to use to inhibit microbial degradation of the test compound (p. 9). If the registrant chooses to use sterilized soil to study mobility, the batch equilibrium studies must include data for reference chemicals of the known mobility; such data were not submitted in the current study. The reviewer notes that gamma irradiation was also used in a batch equilibrium study (MRID 44320646) in which the parent compound, glyphosate acid, was observed to degrade.

3. Freundlich $K_{des}$ values were not reported as required by Subdivision N Guidelines. However, Freundlich $K_{oc}$ values for desorption were reported in Table 7 (p. 25).

4. Limits of quantitation and detection were not reported. It is necessary that both limits of detection and quantitation be reported to allow the reviewer to evaluate the adequacy of the method for the determination of the test compound.

5. The reviewer noted that four of the five soils were between pH 4 and 8 (Table 2, p. 12); however, the sandy loam (Visalia) soil had a pH of 8.4. Subdivision N Guidelines require the use of soils with a pH between 4 and 8.

6. The sandy loam soil from Visalia, California, was the same type of soil used in an aerobic soil metabolism study (MRID 44320645) of [P-methylene-$^{14}$C]glyphosate acid.

7. The 1/N values for the sandy loam (Visalia and 18 Acres), sand, and silty clay loam (Champaign) soils were <0.9 for the adsorption phase of the study (Table 6, p. 23); therefore, the Freundlich isotherm may not accurately depict the adsorption of the parent compound in this soil across all concentrations.

8. The study was conducted with two domestic (U.S.) and three foreign (U.K.) soils (Table 1, p. 11). The EPA prefers that domestic soils be used in mobility studies. However, the agency will accept foreign soils for two of the four soils required if the soils are characterized according to the USDA system. The soils were characterized using the USDA classification system (Table 2, p. 12).

9. The series names of the soils were not reported. Instead, the soils were referred to by their geographic locations or descriptions of their geographic locations (Table 1, p. 11).

10. The study author stated that the test compound has been shown to adsorb to glassware and, therefore, throughout the study every effort was taken to avoid the use of glass (p. 13).

Page _____ is not included in this copy.

Pages 6 through 24 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 conf
by product registrants. If you have any questions, please
the individual who prepared the response to your request.