

## DATA EVALUATION RECORD

## STUDY 49

CHEM 030053 2,4-D 2-Butoxyethyl Ester §164-2  
 CAS No. 1929-73-3  
 FORMULATION--04--GRANULAR

## STUDY ID 44525001

Norris, F. A. 1998. 2,4-D: Dissipation after application of the granular ester formulation to farm ponds. Laboratory Study No.: 96P10380. Unpublished study performed by American Agricultural Services, Inc., Lucama, NC; Agvise Laboratories, Inc., Northwood, ND; and Qualls Agricultural Laboratories, Inc., Ephrata, WA (in-life phase); and Covance Laboratories, Inc., Madison, WI (analytical phase); and submitted by Rhône-Poulenc Ag Company, Research Triangle Park, NC.

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## CONCLUSIONS

### Field Dissipation - Aquatic

1. This study is scientifically valid and provides supplemental information on the aquatic field dissipation of 2,4-D 2-butoxyethyl ester in a ponds in North Carolina, Minnesota, and Washington. This study provides useful information on the dissipation of 2,4-D 2-butoxyethyl ester from the North Carolina pond, however the data from the ponds in Minnesota and Washington are of questionable value. The study authors report that outflow water from the Minnesota and Washington ponds was not analyzed for residues, precluding the determination of whether the pesticide dissipated in the ponds or was transported out of the study area. The North Carolina pond was reported to be static and thus outflow samples were not required. Upgrading of the study will require additional information to address the concerns listed below.
2. This study does not meet Subdivision N Guidelines for the fulfillment of EPA data requirements on aquatic field dissipation for the following reasons:
  - the outflow water from the Minnesota and Washington ponds was not analyzed for residues. The continual influx of water into the system will result in the dilution of concentrations while the continual outflow of water from the system would add an additional route of dissipation to the removal of 2,4-D from the system which without sampling cannot be accounted for,
  - The esters of 2,4-D have been shown to hydrolyze to 2,4-D acid under alkaline conditions. However, the hydrolysis of the esters of 2,4-D is pH dependent with rapid hydrolysis in alkaline conditions but slower hydrolysis under neutral and acidic conditions. All three ponds represent alkaline waters and may not reflect the range of actual use conditions under which 2,4-D 2-butoxyethyl ester may be used The registrant should provide any additional information on the behavior of the esters under acidic and neutral conditions in the field,
  - the half lives for 2,4-D acid are of questionable value given that the 2,4-D 2-butoxyethyl ester granules were still present in sediment for up to 30, 186, and 14 days posttreatment at the North Carolina, Minnesota, and Washington sites, respectively. Therefore 2,4-D acid was continuing to be released from the sediment and the continual formation of 2,4-D acid precludes an accurate estimation of the rate of decline, and
  - frozen storage stability data were inadequate.

3. Residue data were reported as means of four replicates unless otherwise noted. The reviewer reported concentrations from the one replicate or the means of two or three replicates when one or more replicates were detected below the limit of quantitation. Sediment and water data were variable between replicates at all three sites.

At the North Carolina site, parent 2,4-D 2-butoxyethyl ester (AQUAKLEEN<sup>®</sup>, 27.6% a.i.), broadcast applied once at a nominal rate of 200 lb a.i./A onto a man-made pond of Norfolk loam sediment in North Carolina, dissipated with registrant-calculated half-lives of 40 days (15 to 189 day data;  $r^2 = 0.95$ ) in water and 27 days ( $r^2 = 0.78$ ) in sediment. EFED estimated the half-life in water from the North Carolina pond using linear regression of log transformed data (mean concentrations across both depths) of 39.9 days ( $r^2 = 0.99$ ) and 28.5 days ( $r^2 = 0.86$ ) in the sediment. EFED also estimated half-lives in sediment from the North Carolina pond of 9.6 days ( $r^2 = 0.87$ ) for 2,4-D BEE and 80.5 days ( $r^2 = 0.84$ ) for the degradate 2,4-DCP.

The parent compound rapidly hydrolyzes to the acid equivalent (a.e.) 2,4-D following release from the granule; therefore, half-lives were based on 2,4-D data. Parent compound was initially (day 0) present in the 0- to 5-cm sediment depth at 6.6 ppm, increased to a maximum of 7.7 ppm by 1 day posttreatment, decreased to 1.4 ppm by 7 days, and was last present at 0.87 ppm at 30 days. The parent compound was detected twice in the 5- to 10-cm depth, at 0.04 ppm at 0 and 3 days posttreatment (one replicate each). Parent was not present in the 10- to 15-cm depth and was detected twice in the 15- to 20-cm depth, at 0.03-0.05 ppm (one replicate each) from 0 to 1 day posttreatment. 2,4-D was initially (day 0) present in the 0- to 5-cm depth at 7.1 ppm, was a maximum of 8.3 ppm at 3 days posttreatment, decreased to 4.5 ppm by 15 days and 0.14 ppm by 59 days, and was 0.07 ppm at 189 days. In the 5- to 10-cm depth, 2,4-D was initially (day 0) present at 0.13 ppm, decreased to 0.05 ppm (two replicates) by 1 day posttreatment, increased to a maximum of 0.35 ppm by 30 days, and was 0.03 ppm (one replicate) at 189 days. In the 10- to 15-cm depth, 2,4-D was initially (day 0) present at a maximum of 0.31 ppm (two replicates) and was last present at 0.10 ppm (one replicate) at 90 days. In the 15- to 20-cm depth, 2,4-D was initially (day 0) present at a maximum of 0.36 ppm (one replicate), was 0.03-0.04 ppm (one or two replicates) from 3 to 30 days posttreatment, and was last present at 0.23 ppm at 90 days. The major degradate 2,4-DCP was present in the 0- to 5-cm depth at a maximum of 0.41 ppm at 15 days posttreatment and was 0.05 ppm at 189 days. In the 5- to 10-cm depth, 2,4-DCP was a maximum of 0.10 ppm (one replicate) at 0 day posttreatment and was 0.03 ppm (one replicate) at 189 days; 2,4-DCP was present in the 10- to 15-cm depth twice at 0.05 ppm (59 days) and 0.09 ppm (90 days; one replicate), and was present in the 15- to 20-cm depth once at 0.22 ppm (90 days; one replicate). The major degradate 4-CP was present in the 0- to 5-cm depth at a maximum of 0.18 ppm at 59 days posttreatment and was 0.06 ppm (three replicates) at 189 days. In the 5- to 10-cm depth, 4-CP was a maximum of 0.52 ppm (one replicate) at 30 days posttreatment and was 0.05 ppm (three replicates) at 189 days. 4-CP was present in the 10- to 15-cm depth at a maximum of 0.20 ppm (one replicate) at 59 days

posttreatment and was last present at 0.05 ppm (one replicate) at 153 days; 4-CP was present in the 15- to 20-cm depth once at 0.07 ppm (90 days; one replicate). The major degradate 4-CPA was initially (day 0) present in the 0- to 5-cm depth at a maximum of 0.05 ppm (three replicates) and was last present at 0.03 ppm (one replicate) at 15 days. In the 5- to 10-cm depth, 4-CPA was present twice at 0.05 ppm (59 days; one replicate) and 0.04 ppm (122 days; one replicate); 4-CPA was present in the 10- to 15-cm depth twice at 0.16 ppm (59 days; one replicate) and 0.13 ppm (90 days; one replicate), and was present in the 15- to 20-cm depth once at 0.10 ppm (90 days; one replicate).

At the North Carolina site, the parent compound was present (day 0) in the surface and subsurface water once at 3.9 ppb (one replicate) and 42.2 ppb (one replicate), respectively. 2,4-D was present in the surface water at a maximum of 2750 ppb at 15 days posttreatment and decreased to 134 ppb by 189 days; 2,4-D was present in the subsurface water at a maximum of 2725 ppb at 15 days posttreatment and decreased to 135 ppb by 189 days. The major degradate 2,4-DCP was present in the surface water at 2.5-4.0 ppb from 3 to 30 days posttreatment and in the subsurface water at 3.8-9.3 ppb from 1 to 30 days posttreatment. The major degradate 4-CPA was present in the surface water at a maximum of 127 ppb at 122 days posttreatment and was 58.6 ppb at 189 days; 4-CPA was present in the subsurface water at a maximum of 122 ppb at 122 days posttreatment and was 59.5 ppb at 189 days. The major degradate 4-CP was present only in the subsurface water once at 3.1 ppb (three replicates) at 1 day posttreatment.

At the Minnesota site, Parent 2,4-D 2-butoxyethyl ester (AQUAKLEEN<sup>®</sup>, 27.6% a.i.), broadcast applied once at a nominal rate of 200 lb a.i./A onto a man-made pond of clay loam sediment in Minnesota, dissipated with registrant-calculated half-lives of 11 days ( $r^2 = 0.75$ ) in water and 26 days ( $r^2 = 0.68$ ) in sediment. The parent compound rapidly hydrolyzes to the acid equivalent 2,4-D following release from the granule; therefore, half-lives were based on 2,4-D data. Parent compound was initially (day 0) present in the 0- to 5-cm sediment depth at 29.5 ppm, was 5.7-12.0 ppm from 1 to 28 days posttreatment, was 24.1 ppm (two replicates) at 60 days, and was 1.6 ppm (two replicates) at 186 days. In the 5- to 10-cm depth, parent compound was initially (day 0) present at 0.15 ppm (three replicates), was a maximum of 0.45 ppm (two replicates) at 1 day, and was 0.06 ppm (one replicate) at 186 days. Parent was present in the 10- to 15-cm depth twice at 0.40 ppm (0 day) and 0.29 ppm (1 day; one replicate), and in the 15- to 20-cm depth once at 0.03 ppm (14 days; one replicate). 2,4-D was initially (day 0) present in the 0- to 5-cm depth at 26.3 ppm, increased to a maximum of 30.7 ppm by 1 day posttreatment, decreased to 18.9 ppm by 3 days, and was 0.65 ppm at 186 days. In the 5- to 10-cm depth, 2,4-D was present at a maximum of 0.81 ppm at 1 day posttreatment, decreased to 0.48 ppm by 3 days posttreatment, and was 0.09-0.41 ppm (one to four replicates) from 7 to 189 days with the exception of 111 days (detected <limit of quantitation). In the 10- to 15-cm depth, 2,4-D was initially (day 0) present at 0.84 ppm (three replicates), decreased to 0.11 ppm by 1 day, and was last present at 0.04 ppm at 111 days. In the 15- to 20-cm depth, 2,4-D was initially (day 0) present at 0.08

ppm (two replicates), was detected below the limit of quantitation (all replicates) at 1 day posttreatment, was 0.04-0.13 ppm from 3 to 28 days, and was last present at 0.04 ppm (one replicate) at 111 days. The major degradate 2,4-DCP was present in the 0- to 5-cm depth at 0.41 ppm from 0 to 1 day posttreatment, was a maximum of 1.2 ppm (three replicates) at 60 days posttreatment, and was 0.14 ppm at 186 days. In the 5- to 10-cm depth, 2,4-DCP was present sporadically at 0.04-0.16 ppm at 1, 3, 7, 60, and 122 to 186 days (one or two replicates). 2,4-DCP was present in the 10- to 15-cm and 15- to 20-cm depths once at 0.11 ppm (150 days; one replicate) and 0.04 ppm (150 days; one replicate), respectively. The major degradate 4-CP was initially (day 0) present in the 0- to 5-cm depth at 0.15 ppm (three replicates) and was 0.11-0.49 ppm (one to four replicates) from 1 to 28 days; 4-CP was not detected below the 0- to 5-cm depth. The major degradate 4-CPA was present in the 0- to 5-cm depth at 0.21-0.23 ppm (one or two replicates) from 0 to 3 days posttreatment, was detected below the limit of quantitation from 7 to 14 days, was a maximum of 5.9 ppm at 60 days, and was 0.45 ppm (two replicates) at 186 days. In the 5- to 10-cm depth, 4-CPA was initially present at 0.06 ppm at 60 days and was 0.05-0.12 ppm (one to four replicates) from 111 to 186 days; 4-CPA was not present below the 5- to 10-cm depth.

At the Minnesota site, the parent compound was present in the surface water at 6.0 ppb (day 0; two replicates) and 40.4 ppb (1 day; two replicates) only, and in the subsurface water at 2.2 ppb (day 0; one replicate) only. 2,4-D was present in the surface water at a maximum of 237 ppb at 1 day posttreatment, decreased variably to 109 ppb by 14 days, was detected below the limit of quantitation at 28 days, and was last present at 5.6 ppb at 60 days. 2,4-D was initially (day 0) present in the subsurface water at 42.2 ppb, increased to a maximum of 164 ppb by 14 days posttreatment, and was last present at 5.7 ppb at 60 days. The major degradate 4-CPA was present only in the subsurface water twice at 3.0-4.2 ppb from 14 to 28 days posttreatment.

At the Washington site, parent 2,4-D 2-butoxyethyl ester (AQUAKLEEN<sup>®</sup>, 27.6% a.i.), broadcast applied once at a nominal rate of 200 lb a.i./A onto a man-made pond of Quincy loamy sand sediment in Washington, dissipated with registrant-calculated half-lives of 2 days ( $r^2 = 0.70$ ) in water and 5 days ( $r^2 = 0.24$ ) in sediment. The parent compound rapidly hydrolyzes to the acid equivalent 2,4-D following release from the granule; therefore, half-lives were based on 2,4-D data. Parent compound was initially (day 0) present in the 0- to 5-cm sediment depth at 8.8 ppm (three replicates), was 0.03-0.07 ppm (one to three replicates) from 1 to 7 days posttreatment, and was last present at 1.5 ppm (two replicates) at 14 days; parent was not present below the 0- to 5-cm depth. 2,4-D was initially (day 0) present in the 0- to 5-cm depth at 5.9 ppm, was 0.30-1.1 ppm from 1 to 7 days posttreatment, and was last present at 1.6 ppm (three replicates) at 14 days. In the 5- to 10-cm depth, 2,4-D was initially (day 0) present at 0.53 ppm and was last present at 0.08 ppm at 3 days. In the 10- to 15-cm depth, 2,4-D was initially (day 0) present at 0.23 ppm (three replicates) and was last present at 0.04 ppm (three replicates) at 7 days. In the 15- to 20-cm depth, 2,4-D was initially (day 0) present at 0.16 ppm

(three replicates) and was last present at 0.03 ppm (three replicates) at 7 days. The major degradate 2,4-DCP was present in the 0- to 5-cm and 5- to 10-cm depths once at 0.07 ppm (0 day; two replicates) and 0.06 ppm (1 day; one replicate), respectively; 2,4-DCP was not present below the 5- to 10-cm depth. The major degradate 4-CPA was present in the 0- to 5-cm depth once at 0.04 ppm (one replicate) at 0 day posttreatment; 4-CPA was not present below the 0- to 5-cm depth.

At the Washington site, the parent compound was present (day 0) in the surface and subsurface water once at 8.3 ppb (0 day; two replicates) and 4.5 ppb (three replicates), respectively. 2,4-D was initially (day 0) present in the surface water at 117 ppb, decreased to 37.8 ppb by 1 day posttreatment, and was last present at 4.0 ppb at 7 days. 2,4-D was initially (day 0) present in the subsurface water at 102 ppb, decreased to 18.6 ppb by 1 day posttreatment, and was last present at 2.8 ppb (two replicates) at 14 days.

## METHODOLOGY

Parent 2,4-D 2-butoxyethyl ester (AQUAKLEEN<sup>®</sup>, 27.6% a.i.) was broadcast applied once at a nominal rate of 200 lb a.i./A onto three man-made ponds (2.8-11.5 ft of water; Appendix B, Table 1, p. 152) of Norfolk loam (North Carolina; 0-5 cm depth: 34-45% sand, 43-57% silt, 9-14% clay, 2.2-2.9% organic matter, pH 6.0-6.4, CEC 7.9-9.3 meq/100 g; Appendix C, p. 185), clay loam (Minnesota; 0-5 cm depth: 12.9-36.6% sand, 36.7-42.3% silt, 25.2-44.8% clay, 4.0-7.5% organic matter, pH 8.1-8.3, CEC 33.5-37.9 meq/100 g; Appendix C, p. 187) and Quincy loamy sand sediment (Washington; 0-5 cm depth: 71-91% sand, 5-27% silt, 2-4% clay, 0.6-1.0% organic matter, pH 7.1-8.1, CEC 9.0-13.6 meq/100 g; Appendix C, p. 189; see Comment #7). Applications were made using a boat-mounted rotary granule spreader (p. 18). Untreated control plots were not utilized. The depth of water (pH 7.6-9.0, total dissolved solids 142-208 ppm, conductivity 0.06-0.08 mmhos, total hardness 10-13 mg/L; Appendix C, p. 184) in the North Carolina pond was maintained by the addition of well water (pp. 24, 25); the water level was raised approximately 5 inches between 7/31/96 and 8/1/96 and 10 inches between 8/20/96 and 8/22/96. The depth of water (pH 7.7-8.2, total dissolved solids 298-376 ppm, conductivity 0.55-0.59 mmhos, total hardness 252-267 mg/L; Appendix C, p. 186) in the Minnesota pond was maintained by a natural stream flowing into the pond and out over a dam. The depth of water (pH 7.4-8.1, total dissolved solids 108-188 ppm, conductivity 0.16-0.17 mmhos, total hardness 60-67 mg/L; Appendix C, p. 188) in the Washington pond was maintained by adding water from an irrigation ditch (Columbia River) at a rate of approximately 2 ft<sup>3</sup>/sec which maintained a spill rate of 0.1 ft<sup>3</sup>/sec. During the study period, the North Carolina pond was treated once with diquat at 2 lb a.i./A (Appendix B, Table III, p. 152). Parent 2,4-D 2-butoxyethyl ester or related compounds had not been applied to the ponds within the last two years (p. 17); plot history data were not reported. Environmental data were collected off-site (Appendix B, p. 182); total precipitation (reviewer-calculated) was approximately 121%, 132% and

120% of the 10-year mean annual precipitation for the North Carolina, Washington, and Minnesota sites, respectively, for the months of the study.

Sediment samples were collected at the North Carolina site at 0, 1, 3, 7, 15, 30, 59, 90, 122, 153, and 189 days posttreatment (Table V, p. 43); at the Minnesota site at 0, 1, 3, 7, 14, 28, 60, 111, 122, 150, and 186 days posttreatment (Table VI, p. 44); and at the Washington site at 0, 1, 3, 7, 14, 30, 62, 92, 124, 149, and 226 days posttreatment (Table VII, p. 45). At each sampling interval, four sediment core samples were collected from each of four subplots (p. 18). A 0- to 20-cm depth sample was collected using a custom built soil probe (unspecified i.d.) with an acetate liner. Samples were sectioned into 5-cm increments, composited by subplot and depth, and placed in frozen storage (unspecified temperature). Water samples were collected at the same sampling intervals as the sediment samples with the exception of 90 and 153 days posttreatment for the North Carolina pond (Table II, p. 40); 111, 150, and 186 days posttreatment for the Minnesota pond (Table III, p. 41); and 92, 149, and 226 days posttreatment for the Washington pond (Table IV, p. 42). At each sampling interval, four surface and subsurface water samples were collected using a glass bottle (p. 18); subsurface water samples were collected just above the sediments by attaching a capped bottle to a grab sampler. Water and sediment samples were shipped by freezer truck or overnight on dry ice to the analytical laboratory and placed in frozen storage (unspecified temperature). Samples were stored frozen for up to 11 (water) or 10 (sediment) months prior to extraction and analysis (p. 22).

Sediment and water samples were analyzed for parent 2,4-dichlorophenoxyacetic acid, butoxyethyl ester (2,4-D-BEE); 2,4-dichlorophenoxyacetic acid (2,4-D); 2,4-dichlorophenol (2,4-DCP); 2,4-dichloroanisole (2,4-DCA); 4-chlorophenoxyacetic acid (4-CPA); and 4-chlorophenol (4-CP; p. 20). Sediment subsamples were extracted in series by vortexing and sonicating with 5% acetic acid in methanol, 5% acetic acid in methanol:5% acetic acid in water (50:50, v:v), and 5% acetic acid in water (p. 21; Appendix D, pp. 508-513). Samples were centrifuged and the extracts were decanted after each extraction. The extracts were filtered, combined, and diluted with water. An aliquot of the extract was combined with hexane followed by  $\text{Na}_2\text{SO}_4$ , NaCl, and 0.5 *N* NaOH (in sequence); 2,4-D-BEE and 2,4-DCA were partitioned into hexane. The hexane fraction was separated and concentrated (method not specified). The aqueous fraction was acidified ( $\text{H}_3\text{PO}_4$ ; pH not specified) and mixed with dichloromethane; 2,4-DCP and 4-CP partitioned into the dichloromethane fraction which was removed and partitioned with 0.25 *N*  $\text{NaHCO}_3$  followed by 1.0 *N* NaOH. The remaining aqueous fraction (containing 2,4-D and 4-CPA) was acidified (pH <2,  $\text{H}_3\text{PO}_4$ ), mixed with NaCl and partitioned twice with ether. The ether fractions were removed, combined, mixed with 1%  $\text{NH}_4\text{OH}$  in methanol, and the ether was evaporated to dryness; 2,4-D and 4-CPA were derivatized to their methyl esters with 3%  $\text{H}_3\text{PO}_4$  in methanol and 12%  $\text{BF}_3$  in methanol and diluted with water. An aliquot of the hexane fraction (containing 2,4-D-BEE and 2,4-DCA) was combined with the solution containing 2,4-D ME and 4-CPA ME; the two compounds were partitioned into hexane. The aqueous and hexane fractions were

separated and the aqueous fraction was partitioned with hexane a second time; the hexane fractions were combined and concentrated. The 1.0 N NaOH fraction (containing 2,4-DCP and 4-CP) was acidified (pH <2, H<sub>3</sub>PO<sub>4</sub>), mixed with NaCl, and partitioned twice with ether. The ether fractions (containing 2,4-DCP and 4-CP) were combined, concentrated, and combined with the solution containing 2,4-D-BEE; 2,4-DCA; 2,4-D ME; and 4-CPA ME. The combined fractions were concentrated and analyzed by GC with mass selective detection (MSD); the limit of quantitation was 20 ppb (p. 20).

Water samples were acidified (H<sub>3</sub>PO<sub>4</sub>) and concentrated by solid phase extraction (SPE; C18 column; p. 20); retained residues were eluted with acetone:hexane (unspecified volume ratio) followed by methanol:acetone (unspecified volume ratio). The methanol:acetone eluent was methylated and combined with the first eluent. The combined eluent was concentrated, redissolved in an unspecified solvent, and analyzed by GC/MSD; the limit of quantitation was 2 ppb.

In a method validation study, water and sediment samples were fortified at 2-1000 µg/L and 0.01-5 ppm, respectively, with the parent compound and degradates (see Comment #8). Mean recoveries from water samples fortified separately at 2 µg/L were 77.8 ± 7.3% (1 of 4 samples <70%) for parent; 63.8 ± 8.6% (2 of 4 samples <70%) for 2,4-D; 95.4 ± 6.5% for 2,4-DCA; 112 ± 15.1% for 2,4-DCP; 63.1 ± 23.6% (2 of 4 samples <70%) for 4-CP and 61.1 ± 4.3% (4 of 4 samples <70%) for 4-CPA (Appendix D, Table II, p. 320). Mean recoveries (reviewer-calculated) from water samples fortified separately at 10 µg/L and 1000 µg/L were 112.5% (3 of 8 samples >120%) for parent; 91.2% for 2,4-D; 106% for 2,4-DCA; 111.5% (2 of 8 samples >120%) for 2,4-DCP; 82.8% (2 of 8 samples <70%) for 4-CP and 85.1% for 4-CPA. Mean recoveries from sediment samples fortified separately at 0.01 ppm were 72.6 ± 18.9% (2 of 4 samples <70%) for parent; 129 ± 10.4% (3 of 4 samples >120%) for 2,4-D; 90.2 ± 4.1% for 2,4-DCA; 122 ± 10.8% (2 of 4 samples >120%) for 2,4-DCP; 110 ± 14.5% (1 of 4 samples >120%) for 4-CP and 98.6 ± 9.5% for 4-CPA (Appendix D, Table XV, p. 377). Mean recoveries from sediment samples fortified separately at 0.05 ppm were 90.5 ± 20.6% for parent; 86.2 ± 12.4% (1 of 4 samples <70%) for 2,4-D; 82.6 ± 6.7% for 2,4-DCA; 92.7 ± 5.2% for 2,4-DCP; 86.3 ± 4.8% for 4-CP and 75.6 ± 8.8% (1 of 4 samples <70%) for 4-CPA. Mean recoveries from sediment samples fortified separately at 5.0 ppm were 70.2 ± 6.3% (3 of 4 samples <70%) for parent; 73.6 ± 8.6% (2 of 4 samples <70%) for 2,4-D; 76.4 ± 5.8% for 2,4-DCA; 75.5 ± 8.7% (1 of 4 samples <70%) for 2,4-DCP; 66.1 ± 11.6% (2 of 4 samples <70%) for 4-CP and 70.9 ± 5.7% (1 of 4 samples <70%) for 4-CPA.

To determine concurrent recoveries, water and sediment (0- to 5-cm, 5- to 10-cm, 10- to 15-cm and 15- to 20-cm depths) samples from each test site were fortified separately with the parent compound and degradates. Mean recoveries from North Carolina pond water fortified at 2-4000 µg/L were 90.1 ± 9.4% for parent; 83.0 ± 6.7% for 2,4-D; 92.3 ± 8.7% for 2,4-DCA; 91.4 ± 12.6% (1 of 26 samples <70%) for 2,4-DCP; 80.9 ± 12.9% (7 of 26 samples <70%) for 4-CP and 76.2 ± 5.6% (2 of 24 samples <70%) for 4-CPA (Appendix



D, Table III, pp. 321-322). Mean recoveries from Minnesota pond water fortified at 5-500  $\mu\text{g/L}$  were  $99.1 \pm 14.2\%$  (4 of 24 samples  $>120\%$ ) for parent;  $87.3 \pm 8.7\%$  for 2,4-D;  $99.7 \pm 10.6\%$  (1 of 24 samples  $>120\%$ ) for 2,4-DCA;  $101 \pm 16.0\%$  (4 of 24 samples outside 70-120%) for 2,4-DCP;  $82.3 \pm 18.9\%$  (7 of 24 samples outside 70-120%) for 4-CP and  $77.0 \pm 8.1\%$  (4 of 23 samples  $<70\%$ ) for 4-CPA (Appendix D, Table IV, p. 323). Mean recoveries from Washington pond water fortified at 5-200  $\mu\text{g/L}$  were  $89.3 \pm 16.4\%$  (2 of 18 samples  $<70\%$ ) for parent;  $86.2 \pm 14.4\%$  (2 of 18 samples  $<70\%$ ) for 2,4-D;  $92.9 \pm 9.7\%$  (1 of 20 samples  $>120\%$ ) for 2,4-DCA;  $91.7 \pm 12.9\%$  (1 of 20 samples  $<70\%$ ) for 2,4-DCP;  $77.7 \pm 16.3\%$  (4 of 24 samples  $<70\%$ ) for 4-CP and  $73.9 \pm 10.7\%$  (6 of 18 samples  $<70\%$ ) for 4-CPA (Appendix D, Table V, p. 324). Recoveries (across all depths) from loam sediment (North Carolina) fortified at 0.02-20 ppm were 46.7-158% (7 of 42 samples outside 70-120%) for parent compound; 76.2-134% (3 of 42 samples  $>120\%$ ) for 2,4-D; 61.3-109% (5 of 42 samples  $<70\%$ ) for 2,4-DCA; 65.4-135% (5 of 42 samples outside 70-120%) for 2,4-DCP; 56.9-128% (4 of 41 samples outside 70-120%) for 4-CP and 61.7-115% (3 of 42 samples  $<70\%$ ) for 4-CPA (Appendix D, Tables XVI-XXI, pp. 378-383). Recoveries from clay loam sediment (Minnesota) fortified at 0.02-20 ppm were 47.1-134% (14 of 44 samples outside 70-120%) for parent compound; 59.2-128% (5 of 42 samples outside 70-120%) for 2,4-D; 64.7-109% (5 of 42 samples  $<70\%$ ) for 2,4-DCA; 55.9-128% (6 of 43 samples outside 70-120%) for 2,4-DCP; 59.9-146% (7 of 43 samples outside 70-120%) for 4-CP and 53.3-117% (7 of 43 samples  $<70\%$ ) for 4-CPA (Appendix D, Tables XXII-XXVII, pp. 384-389). Recoveries from loamy sand sediment (Washington) fortified at 0.02-15 ppm were 22.1-198% (12 of 24 samples outside 70-120%) for parent compound; 58.0-159% (5 of 26 samples outside 70-120%) for 2,4-D; 53.1-96.2% (4 of 25 samples  $<70\%$ ) for 2,4-DCA; 62.9-106% (1 of 26 samples  $<70\%$ ) for 2,4-DCP; 59.6-169% (2 of 26 samples outside 70-120%) for 4-CP and 59.7-105% (1 of 26 samples outside 70-120%) for 4-CPA (Appendix D, Tables XXVIII-XXXIII, pp. 390-395).

In a field fortification study, untreated water and sediment samples were fortified at 40 ppb and 0.40 ppm, respectively, with the parent and degradates (p. 22). The fortified samples were transported, stored, and analyzed in the same manner as the test samples. Mean corrected recoveries from the North Carolina water samples were 83.9% for the parent compound; 84.2% for 2,4-D; 78.6% (1 of 3 samples  $<70\%$ ) for 2,4-DCA; 107% for 2,4-DCP; 79.6% for 4-CP and 73.8% (2 of 3 samples  $<70\%$ ) for 4-CPA (Appendix D, Table IX, pp. 339-340). Mean corrected recoveries from the Minnesota water samples were 106% for parent compound; 92.4% for 2,4-D; 102% for 2,4-DCA; 112% for 2,4-DCP; 106% for 4-CP and 86.5% for 4-CPA (Appendix D, Table X, pp. 341-342). Mean corrected recoveries from Washington water samples were 129% (3 of 3 samples  $>120\%$ ) for parent compound; 100% for 2,4-D; 102% for 2,4-DCA; 103% for 2,4-DCP; 89.3% for 4-CP and 100% for 4-CPA (Appendix D, Table XI, pp. 343-344). Mean corrected recoveries from loam sediment (North Carolina) were 68.4% (3 of 3 samples  $<70\%$ ) for parent compound; 66.7% (2 of 3 samples  $<70\%$ ) for 2,4-D; 77.7% (1 of 3 samples  $<70\%$ ) for 2,4-DCA; 75.0% for 2,4-DCP; 60.0% (3 of 3 samples  $<70\%$ ) for 4-CP and 82.9% for

2,4-CPA (Appendix D, Table XXXVII, pp. 414, 415). Mean corrected recoveries from the clay loam sediment (Minnesota) were 112% for parent compound; 69.0% (2 of 3 samples <70%) for 2,4-D; 67.1% (2 of 3 samples <70%) for 2,4-DCA; 93.7% for 2,4-DCP; 91.6% for 4-CP and 67.3% (1 of 3 samples <70%) for 4-CPA (Appendix D, Table XXXVIII, pp. 416, 417). Mean corrected recoveries from loamy sand sediment (Washington) were 61.3% (3 of 3 samples <70%); 80.8% for 2,4-D; 99.2% for 2,4-DCA; 18.3% (3 of 3 samples <70%) for 2,4-DCP; 43.7% (3 of 3 samples <70%) for 4-CP and 88.5% for 4-CPA (Appendix D, Table XXXIX, pp. 418-419).

In a storage stability study, water and sediment samples (from the North Carolina and Washington sites only) were fortified at 40 ppb and 0.40 ppm, respectively, with parent compound and its degradates and placed in frozen storage for up to 3 months (p. 22; see Comment #2). Mean corrected recoveries from water samples from the North Carolina field site were 117% at 0 days and 101% at 3 months posttreatment for parent; 75.1% at 0 days and 102% at 3 months posttreatment for 2,4-D; 86.0% at 0 days and 94.9% at 3 months posttreatment for 2,4-DCA; 88.9% at 0 days and 98.2% at 3 months posttreatment for 2,4-DCP; 98.6% at 0 days and 87.3% at 3 months posttreatment for 4-CP; and 94.6% at 0 days and 108% at 3 months posttreatment for 4-CPA (Appendix D, Table XII, pp. 345-347). Mean corrected recoveries from water samples from the Washington field site were 98.7% at 0 days and 91.9% at 3 months posttreatment for parent; 98.6% at 0 days and 87.8% at 3 months posttreatment for 2,4-D; 96.1-98.1% for 2,4-DCA; 98.6-101% for 2,4-DCP; 99.3-101% for 4-CP and 98.5-105% for 4-CPA (Appendix D, Table XIII, pp. 348-350). Mean corrected recoveries from silt loam sediment from the North Carolina field site were 121% at 0 days and 109% at 3 months posttreatment for parent; 87.7% at 0 days and 84.5% at 3 months posttreatment for 2,4-D; 90.8% at 0 days and 85.3% at 3 months posttreatment for 2,4-DCA; 88.7-90.7% for 2,4-DCP; 94.5-96.5% for 4-CP; and 78.7% at 0 days and 98.9% at 3 months posttreatment for 4-CPA (Appendix D, Table XL, pp. 420-422). Mean corrected recoveries from loamy sand sediment from the Washington field site were 114% at 0 days and 85.5% at 3 months posttreatment for parent; 89.3% at 0 days and 96.5% at 3 months posttreatment for 2,4-D; 94.1-98.3% for 2,4-DCA; 100% at 0 days and 72.0% at 3 months posttreatment for 2,4-DCP; 101% at 0 days and 73.6% at 3 months posttreatment for 4-CP; and 81.3-83.7% for 4-CPA (Appendix D, Table XLI, p. 423-425).

## DATA SUMMARY

Residue data were reported as means of four replicates unless otherwise noted. The reviewer reported concentrations from one replicate or the mean of two or three replicates when one or more replicates were detected below the limit of quantitation. Sediment and water data were variable between replicates at all three sites.

### North Carolina Site

Parent 2,4-D 2-butoxyethyl ester (AQUAKLEEN<sup>®</sup>, 27.6% a.i.), broadcast applied once at a nominal rate of 200 lb a.i./A onto a man-made pond of Norfolk loam sediment in North Carolina, dissipated with registrant-calculated half-lives of 40 days (15-189 day data;  $r^2 = 0.95$ ) in water and 27 days ( $r^2 = 0.78$ ) in sediment (pp. 32, 35; Figure 10, p. 56). The parent compound rapidly hydrolyzes to the acid equivalent (a.e.) 2,4-D following release from the granule; therefore, half-lives were based on 2,4-D data (see Comment #3). Parent compound was initially (day 0) present in the 0- to 5-cm sediment depth at 6.6 ppm, increased to a maximum of 7.7 ppm by 1 day posttreatment, decreased to 6.3 ppm by 3 days and 1.4 ppm by 7 days, and was last present at 0.87 ppm at 30 days (Table V, p. 43). The parent compound was detected twice in the 5- to 10-cm depth, at 0.04 ppm at 0 and 3 days posttreatment (one replicate each). Parent was not present in the 10- to 15-cm depth and was detected twice in the 15- to 20-cm depth, at 0.03-0.05 ppm (one replicate each) from 0 to 1 day posttreatment. 2,4-D was initially (day 0) present in the 0- to 5-cm depth at 7.1 ppm, decreased to 5.6 ppm by 1 day posttreatment, increased to a maximum of 8.3 ppm by 3 days posttreatment, decreased to 4.5 ppm by 15 days and 0.14 ppm by 59 days, and was 0.07 ppm at 189 days posttreatment. In the 5- to 10-cm depth, 2,4-D was initially (day 0) present at 0.13 ppm, decreased to 0.05 ppm (two replicates) by 1 day posttreatment, increased to a maximum of 0.35 ppm by 30 days posttreatment, and was 0.03 ppm (one replicate) at 189 days. In the 10- to 15-cm depth, 2,4-D was present sporadically at 0.31 ppm (0 day; two replicates), 0.05 ppm (3 days; one replicate), 0.04 ppm (15 days; one replicate), 0.09 ppm (59 days), and 0.10 ppm (90 days; one replicate). In the 15- to 20-cm depth, 2,4-D was initially (day 0) present at 0.36 ppm (one replicate), decreased to 0.13 ppm (one replicate) by 1 day posttreatment, was 0.03-0.04 ppm (one or two replicates) from 3 to 30 days posttreatment, and was last present at 0.23 ppm (one replicate) at 90 days. The major degradate

#### 2,4-dichlorophenol (2,4-DCP)

was initially (day 0) present in the 0- to 5-cm depth at 0.10 ppm, decreased to 0.09 ppm by 1 day posttreatment, increased to a maximum of 0.41 ppm by 15 days posttreatment, and was 0.04-0.06 ppm from 59 to 189 days. In the 5- to 10-cm depth, 2,4-DCP was initially present at 0.10 ppm (one replicate) at 7 days posttreatment, was 0.04-0.07 ppm (two to four replicates) from 15 to 122 days posttreatment, and was 0.03 ppm (one replicate) at 189 days. 2,4-DCP was present in the 10- to 15-cm depth twice at 0.05 ppm (59 days) and 0.09 ppm (90 days; one replicate), and was present in the 15- to 20-cm depth once at 0.22 ppm (90 days; one replicate). The major degradate

#### 4-chlorophenol (4-CP)

was initially present in the 0- to 5-cm depth at 0.03 ppm (two replicates) at 7 days posttreatment, increased to a maximum of 0.18 ppm by 59 days posttreatment, and was 0.06 ppm (three replicates) at 189 days. In the 5- to 10-cm depth, 4-CP was initially present at 0.03 ppm (one replicate) at 7 days posttreatment, was a maximum of 0.52 ppm

(one replicate) at 30 days posttreatment, and was 0.05 ppm (three replicates) at 189 days. 4-CP was present in the 10- to 15-cm depth at 0.20 ppm (59 days; one replicate), 0.08 ppm (90 days; one replicate), and 0.05 ppm (153 days; one replicate), and was present in the 15- to 20-cm depth once at 0.07 ppm (90 days; one replicate). The major degradate

#### 4-chlorophenoxyacetic acid (4-CPA)

was initially (day 0) present in the 0- to 5-cm depth at 0.05 ppm (three replicates) and decreased to 0.03 ppm (one replicate) by 15 days. 4-CPA was present twice in the 5- to 10-cm depth, at 0.05 ppm (one replicate) at 59 days posttreatment and 0.04 ppm (one replicate) at 122 days posttreatment. 4-CPA was present in the 10- to 15-cm depth twice at 0.16 ppm (59 days; one replicate) and 0.13 ppm (90 days; one replicate), and was present in the 15- to 20-cm depth once at 0.10 ppm (90 days; one replicate). The degradate 2,4-DCA was not present at any sampling intervals or depths.

Residue data were reported from the surface and subsurface water samples. Parent compound was present (day 0) in the surface and subsurface water once at 3.9 ppb (one replicate) and 42.2 ppb (one replicate), respectively (Table II, p. 40). 2,4-D was initially (day 0) present in the surface water at 295 ppb, increased to a maximum of 2750 ppb by 15 days posttreatment, and decreased to 134 ppb by 189 days. 2,4-D was initially (day 0) present in the subsurface water at 199 ppb, increased to a maximum of 2725 ppb by 15 days posttreatment, and decreased to 135 ppb by 189 days. The major degradate

#### 2,4-DCP

was initially present in the surface water at 2.5 ppb at 3 days posttreatment and was last present at a maximum of 4.0 ppb by 30 days posttreatment. 2,4-DCP was initially present in the subsurface water at 8.0 ppb at 1 day posttreatment, decreased to 3.8 ppb by 15 days posttreatment, and was last present at a maximum of 9.3 ppb at 30 days. The major degradate

#### 4-CPA

was initially present in the surface water at 2.2 ppb at 1 day posttreatment, increased to 11.9 ppb by 15 days posttreatment, decreased to 4.2 ppb by 59 days posttreatment, was a maximum of 127 ppb at 122 days posttreatment, and was 58.6 ppb at 189 days. 4-CPA was initially present in the subsurface water at 3.4 ppb at 1 day posttreatment, increased to 12.4 ppb by 15 days posttreatment, was 4.5 ppb at 59 days posttreatment, increased to a maximum of 122 ppb by 122 days posttreatment, and was 59.5 ppb at 189 days. The major degradate

#### 4-CP

was present only in the subsurface water once at 3.1 ppb (three replicates) at 1 day posttreatment. The degradate 2,4-DCA was not present in the pond water.

### Minnesota Site

Parent 2,4-D 2-butoxyethyl ester (AQUAKLEEN®, 27.6% a.i.), broadcast applied once at a nominal rate of 200 lb a.i./A onto a man-made pond of clay loam sediment in Minnesota, dissipated with registrant-calculated half-lives of 11 days ( $r^2 = 0.75$ ) in water and 26 days ( $r^2 = 0.68$ ) in sediment (pp. 32, 35; Figure 11, p. 57). The parent compound rapidly hydrolyzes to the acid equivalent 2,4-D following release from the granule; therefore, half-lives were based on 2,4-D data (Comment #3). Parent compound was initially (day 0) present in the 0- to 5-cm sediment depth at 29.5 ppm, was 5.7-12.0 ppm from 1 to 28 days posttreatment, was 24.1 ppm (two replicates) at 60 days, and was 1.6 ppm (two replicates) at 186 days (Table VI, p. 44). In the 5- to 10-cm depth, parent compound was initially (day 0) present at 0.15 ppm (three replicates), was 0.45 ppm (two replicates) at 1 day, was 0.06-0.20 ppm (one to three replicates) from 3 to 60 days posttreatment, was not present from 111 to 150 days posttreatment, and was 0.06 ppm (one replicate) at 186 days. Parent was present in the 10- to 15-cm depth twice at 0.40 ppm (0 day) and 0.29 ppm (1 day; one replicate), and in the 15- to 20-cm depth once at 0.03 ppm (14 days; one replicate). 2,4-D was initially (day 0) present in the 0- to 5-cm depth at 26.3 ppm, increased to a maximum of 30.7 ppm by 1 day posttreatment, decreased to 18.9 ppm by 3 days posttreatment, and was 0.65 ppm at 186 days. In the 5- to 10-cm depth, 2,4-D was initially (day 0) present at 0.77 ppm, increased to a maximum of 0.81 ppm by 1 day posttreatment, decreased to 0.48 ppm by 3 days posttreatment, and was 0.09-0.41 ppm (one to four replicates) from 7 to 189 days with the exception of 111 days (detected <limit of quantitation). In the 10- to 15-cm depth, 2,4-D was initially (day 0) present at 0.84 ppm (three replicates), decreased to 0.11 ppm by 1 day, and was last present at 0.04 ppm at 111 days. In the 15- to 20-cm depth, 2,4-D was initially (day 0) present at 0.08 ppm (two replicates), was detected below the limit of quantitation (all replicates) at 1 day posttreatment, was 0.04-0.13 ppm from 3 to 28 days, and was last present at 0.04 ppm (one replicate) at 111 days. The major degradate

#### 2,4-dichlorophenol (2,4-DCP)

was present in the 0- to 5-cm depth at 0.41 ppm from 0 to 1 day posttreatment, was 0.22-0.42 ppm from 3 to 28 days posttreatment, was a maximum of 1.2 ppm (three replicates) at 60 days posttreatment, and was 0.14 ppm at 186 days. In the 5- to 10-cm depth, 2,4-DCP was present sporadically at 0.04-0.05 ppm (1 to 7 days; one replicate each), 0.05 ppm (60 days; one replicate), and 0.04-0.16 ppm (122 to 186 days; one or two replicates). 2,4-DCP was present in the 10- to 15-cm and 15- to 20-cm depths once at 0.11 ppm (150 days; one replicate) and 0.04 ppm (150 days; one replicate), respectively. The major degradate

#### 4-chlorophenol (4-CP)

was initially (day 0) present in the 0- to 5-cm depth at 0.15 ppm (three replicates) and was 0.11-0.49 ppm (one to four replicates) from 1 to 28 days; 4-CP was not detected below the 0- to 5-cm depth. The major degradate

#### 4-chlorophenoxyacetic acid (4-CPA)

was present in the 0- to 5-cm depth at 0.21-0.23 ppm (one or two replicates) from 0 to 3 days posttreatment, was detected below the limit of quantitation from 7 to 14 days, was a maximum of 5.9 ppm at 60 days, and was 0.45 ppm (two replicates) at 186 days. In the 5- to 10-cm depth, 4-CPA was initially present at 0.06 ppm at 60 days and was 0.05-0.12 ppm (one to four replicates) from 111 to 186 days; 4-CPA was not present below the 5- to 10-cm depth. The degradate 2,4-DCA was not present at any sampling intervals or depths.

Residue data were reported from the surface and subsurface water samples. Parent compound was present in the surface water at 6.0 ppb (day 0; two replicates) and 40.4 ppb (1 day; two replicates) only, and in the subsurface water at 2.2 ppb (day 0; one replicate) only (Table III, p. 41). 2,4-D was initially (day 0) present in the surface water at 201 ppb, increased to a maximum of 237 ppb by 1 day posttreatment, decreased variably to 109 ppb by 14 days posttreatment, was detected below the limit of quantitation at 28 days, and was last present at 5.6 ppb at 60 days. 2,4-D was initially (day 0) present in the subsurface water at 42.2 ppb, increased to a maximum of 164 ppb by 14 days posttreatment, and was last present at 5.7 ppb at 60 days. The major degradate

#### 4-CPA

was present only in the subsurface water twice at 3.0-4.2 ppb from 14 to 28 days posttreatment. The degradates 2,4-DCA; 2,4-DCP; and 4-CP were not present in the pond water.

#### Washington Site

Parent 2,4-D 2-butoxyethyl ester (AQUAKLEEN<sup>®</sup>, 27.6% a.i.), broadcast applied once at a nominal rate of 200 lb a.i./A onto a man-made pond of Quincy loamy sand sediment in Washington, dissipated with registrant-calculated half-lives of 2 days ( $r^2 = 0.70$ ) in water and 5 days ( $r^2 = 0.24$ ) in sediment (pp. 32, 35; Figure 12, p. 58). The parent compound rapidly hydrolyzes to the acid equivalent 2,4-D following release from the granule; therefore, half-lives were based on 2,4-D data (see Comment #3). Parent compound was initially (day 0) present in the 0- to 5-cm sediment depth at 8.8 ppm (three replicates), was 0.03-0.07 ppm (one to three replicates) from 1 to 7 days posttreatment, and was last present at 1.5 ppm (two replicates) at 14 days (Table VII, p. 45); parent was not present

below the 0- to 5-cm depth. 2,4-D was initially (day 0) present in the 0- to 5-cm depth at 5.9 ppm, was 0.30-1.1 ppm from 1 to 7 days posttreatment, and was last present at 1.6 ppm (three replicates) at 14 days. In the 5- to 10-cm depth, 2,4-D was initially (day 0) present at 0.53 ppm and was last present at 0.08 ppm at 3 days. In the 10- to 15-cm depth, 2,4-D was initially (day 0) present at 0.23 ppm (three replicates) and was last present at 0.04 ppm (three replicates) at 7 days. In the 15- to 20-cm depth, 2,4-D was initially (day 0) present at 0.16 ppm (three replicates) and was last present at 0.03 ppm (three replicates) at 7 days. The major degradate

#### 2,4-dichlorophenol (2,4-DCP)

was present in the 0- to 5-cm and 5- to 10-cm depths once at 0.07 ppm (0 day; two replicates) and 0.06 ppm (1 day; one replicate), respectively; 2,4-DCP was not present below the 5- to 10-cm depth. The major degradate

#### 4-chlorophenoxyacetic acid (4-CPA)

was present in the 0- to 5-cm depth once at 0.04 ppm (one replicate) at 0 day posttreatment; 4-CPA was not present below the 0- to 5-cm depth. The degradates 2,4-DCA and 4-CP were not present at any sampling intervals or depths.

Residue data were reported from the surface and subsurface water samples. Parent compound was present (day 0) in the surface and subsurface water once at 8.3 ppb (0 day; two replicates) and 4.5 ppb (three replicates), respectively (Table IV, p. 42). 2,4-D was initially (day 0) present in the surface water at 117 ppb, decreased to 37.8 ppb by 1 day posttreatment, and was last present at 4.0 ppb at 7 days. 2,4-D was initially (day 0) present in the subsurface water at 102 ppb, decreased to 18.6 ppb by 1 day posttreatment, and was last present at 2.8 ppb (two replicates) at 14 days. The degradates 2,4-DCA; 2,4-DCP; 4-CP; and 4-CPA were not present in the pond water.

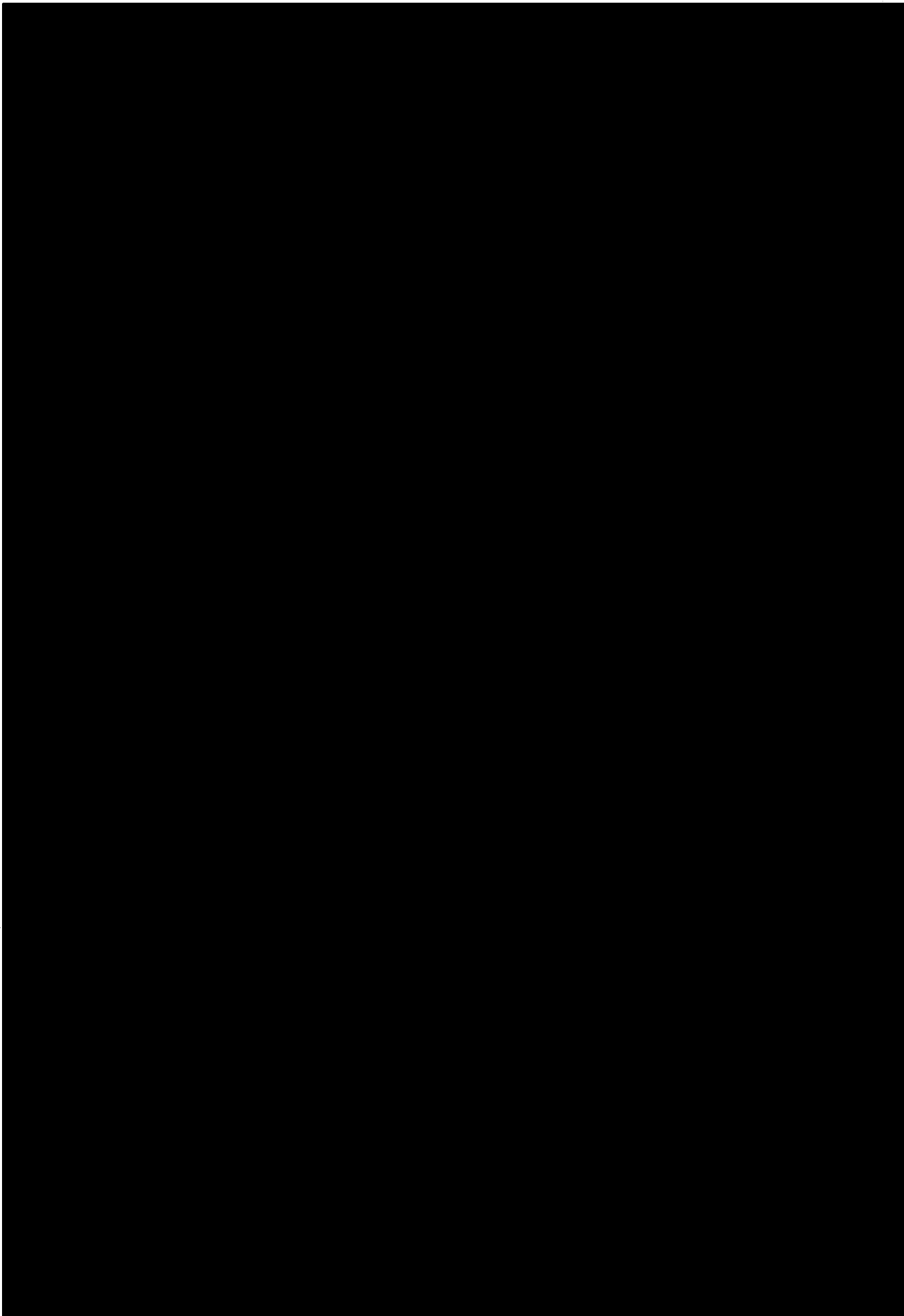
### COMMENTS

1. The outflow water from the Minnesota and Washington sites was not sampled for analysis of residues; therefore, it could not be determined whether the pesticide dissipated in the pond or was transported out of the study area. The study author stated that the depth of water in the Minnesota pond was maintained by a natural stream flowing into the pond and out over a dam and that the depth of water in the Washington pond was maintained by adding water from an irrigation ditch (Columbia River) at a rate of approximately 2 ft<sup>3</sup>/sec which maintained a spill rate of 0.1 ft<sup>3</sup>/sec (p. 25). At the North Carolina site, a static system was utilized and the study author stated that the North Carolina pond was stagnant with opaque water (p. 32).

2. Frozen storage stability data were inadequate. Water and sediment samples were stored frozen for up to 329 days and 302 days, respectively, prior to analysis (p. 33); however, frozen storage stability data were submitted for up to 3 months only (Appendix D; Tables XII, XIII, XL, XLI; pp. 345-350, 420-425) and a storage stability study was not conducted with water and sediment samples from the Minnesota site. Frozen storage stability data are necessary to determine the stability of the parent compound and its degradates in the test soil under typical storage conditions. Storage stability studies should be conducted using sediment and water samples collected from each test site that are fortified separately with the parent compound and its degradates and stored for a duration equal to the longest storage interval for the test samples. The study author stated that storage stability data for up to 11 months (water) and 10 months (sediment) will be reported as supplemental data (p. 33).
3. Registrant-calculated half-lives (based on 2,4-D data) were questionable. The parent compound rapidly hydrolyzes to 2,4-D acid following release from the granule; however, 2,4-D was not completely released from the granule immediately following application (p. 32). Parent was present in the sediments of the North Carolina, Minnesota, and Washington sites for up to 30, 186, and 14 days posttreatment, respectively (Tables V-VII, pp. 43-45). The study author stated that these detections may reflect granules collected as part of the sediment which had not fully released the herbicide.
4. Greater than 50% of 2,4-D degraded in the sediment (5.9 ppm to 0.76 ppm) and water (117 ppb to 37.8 ppb) at the Washington site between consecutive sampling intervals (0 to 1 day; Table IV, p. 42; Table VII, p. 45). Because the half-lives were short, an additional study would not provide new information.
5. Plot history data were not reported. However, the study author stated that 2,4-D 2-butoxyethyl ester or related compounds had not been applied to the ponds within the last two years (p. 17). Subdivision N Guidelines require a three-year plot history.
6. The study author stated that trace residues present in the lower sediment depths in less than four replicates (especially at day 0), was an indication of contamination during sampling (p. 33). The study author stated that contamination problems are common when sampling under at least a meter of water.
7. The reviewer noted that the sediments classified as Norfolk silt loam (North Carolina) and Quincy loamy sand (Washington) on p. 17 were reported as Norfolk sandy loam and Timmerman Coarse sandy loam in Appendix C (pp. 185, 189); the soil series name of the clay loam sediment from the Minnesota site was not reported. Based on the mean percentages of sand, silt, and clay in the 0- to 5-cm depth, the reviewer classified the North Carolina, Washington, and Minnesota sediments as loam, loamy sand, and clay loam, respectively; soil classifications determined by the reviewer were reported.



8. The reviewer could not determine the origin of the sediment used in the method validation study.
9. The reviewer reported water characteristics as ranges of four replicates of the surface and subsurface waters (Appendix C, pp. 184, 186, 188). Characteristics of the source and effluent (Minnesota and Washington sites only) waters were also reported by the study author; these characteristics were not reported by the reviewer.
10. The study author stated that test compound was applied at the expected maximum label rate of 200 lb/A (38 lb 2,4-D equivalents/A; p. 13).
11. The reviewer noted a discrepancy with the reported half-life of 2,4-D in the Washington pond water. A registrant-calculated half-life of 2 days was reported on page 32 and a registrant-calculated half-life of 3 days was reported on page 35. Clarification by the registrant is necessary.
12. The study author stated that reported half-lives of 2,4-D in the water and sediment were "upper bound" half-lives (p. 32). Clarification by the registrant is necessary.



DATA EVALUATION RECORD

STUDY 32

CHEM 030053                                                  2,4-D 2-Butoxyethyl Ester                                                  §164-2  
 CAS No. 1929-73-3  
 FORMULATION--04--GRANULAR

STUDY ID 44525001

Norris, F. A. 1998. 2,4-D: Dissipation after application of the granular ester formulation to farm ponds. Laboratory Study No.: 96P10380. Unpublished study performed by American Agricultural Services, Inc., Lucama, NC; Agvise Laboratories, Inc., Northwood, ND; and Qualls Agricultural Laboratories, Inc., Ephrata, WA (in-life phase); and Covance Laboratories, Inc., Madison, WI (analytical phase); and submitted by Rhône-Poulenc Ag Company, Research Triangle Park, NC.

DIRECT REVIEW TIME = 145 hours

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