DATA EVALUATION RECORD
STUDY 6

CHEM 128959 Fluroxypyr §164-1
CAS No. 69377-81-7
FORMULATION--00--Emulsifiable Concentrate

STUDY ID 44080347
Poletika, N. N., D. W. Roberts, A. M. Phillips, and I. W. Butler. 1996. Terrestrial field dissipation of fluroxypyr. Laboratory Study ID: ENV92023. Unpublished study performed by A & L Great Lakes Laboratories, Inc., Fort Wayne, IN; Agvise, Northwood, ND; DowElanco, Indianapolis, IN; MVTL Laboratories, Inc., New Ulm, MN; Qualls Agricultural Laboratories, Ephrata, WA; and Stewart Agricultural Research Services, Inc., Macon, MO; and submitted by DowElanco.

STUDY ID 44080355
Phillips, A. M. and B. A. Blakeslee. 1996. Frozen storage stability of fluroxypyr 1-methylheptyl ester, fluroxypyr, 4-amino-3,5-dichloro-6-fluoropyridinol, and 4-amino3,5-dichloro-6-fluoro-methoxypyridine in soil. Laboratory Study ID: RES93010/RES93051. Unpublished study performed and submitted by DowElanco, Indianapolis, IN.

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CONCLUSIONS

Field Dissipation - Terrestrial

1. This study is scientifically valid and provides information on the dissipation of fluroxypyr MHE (4-amino-3,5-dichloro-6-fluoro-2-pyridyloxyacetic acid, 1-methylheptyl ester) and fluroxypyr (acid equivalent; 4-amino-3,5-dichloro-6-fluoro-2-pyridyloxyacetic acid) under field conditions in Missouri (MO), North Dakota (ND), and Washington (WA).

2. This study does not meet Subdivision N Guidelines for the partial fulfillment of EPA data requirements on the terrestrial field dissipation of fluroxypyr MHE and fluroxypyr (acid) for the following reason:

   (i) adequate storage stability data were not presented for soils of the test sites, although a storage stability study was conducted (see Comment #1).

3. This study may be upgraded with the submission of frozen storage stability data for fluroxypyr MHE, fluroxypyr (acid), and metabolites using soils obtained from the respective test sites.

4. Total fluroxypyr residues (fluroxypyr MHE plus fluroxypyr) dissipated in Putnam silty clay loam, Gardena sandy clay loam, and Quincy loamy sand soils in MO, ND, and WA, respectively, with respective registrant-calculated half-lives of 24.8, 36.3, and 13.2 days in plots vegetated with spring wheat (see Comment #7); however, tabular values for total fluroxypyr residues were not submitted. In the 0- to 6-inch soil depth, fluroxypyr MHE was present at maximum concentrations of 29 to 140 ng/g soil by 3 to 7 DAT; fluroxypyr (acid) was observed at maximum concentrations of 180 to 220 ng/g. The metabolite pyridinol (4-amino-3,5-dichloro-6-fluoro-2-pyridinol) was observed at a maximum concentration of 12 ng/g in ND, sporadically at <10 ng/g in WA, and was not detectable in MO. The metabolite methoxypyridine (4-amino-3,5-dichloro-6-fluoro-2-methoxypyridine) was observed at maximum concentrations of 25 to 50 ng/g at all three test sites. Fluroxypyr MHE and its metabolites were not detected below the 6-inch depth, except for methoxypyridine in WA at two months after treatment (2 MAT), which was observed at <10 ng/g in the 6- to 12-inch and 12- to 18-inch soil depths.

   In samples obtained from the 0- to 1- or 2-inch depths at each site (0 and 3 DAT only), concentrations of fluroxypyr MHE and fluroxypyr (acid) were substantially higher when compared to the 0- to 6-inch depth.

METHODOLOGY

Fluroxypyr MHE (XRM-5316, 26% a.i.; EC formulation) was broadcast applied as a single postemergence spray at a nominal rate of 0.25 lb acid equivalents (a.e.)/A (1x the proposed maximum rate) onto Putnam silty clay loam, Gardena sandy clay loam, and Quincy loamy sand soils in three field plots (14000-18000 square feet with 6 designated subplots (for sampling) of 15 to 30 feet x 100 feet; pp. 67-69), one each located in MO, ND, and WA; all plots were vegetated with spring wheat. Untreated control plots (2500-6400 square feet) were located at least 147 feet from the treated plots at each site (p. 14). Application rates were not verified directly, but recoveries of total fluroxypyr from soil on the day of application were 76.0%, 88.7%, and 104.8% of the applied (0- to 6-inch depth) in MO, ND, and WA, respectively; respective recoveries were 134.1%, 122.1%, and 127.3% of the applied in the 0- to 2-inch depth (Table VI, p. 34; see Comment #2). Total water input via irrigation and precipitation was 137, 135, and 153% of the 30-year historical precipitation average in MO, ND, and WA, respectively. Pan evaporation data were not reported for any site.

Fifteen soil cores were randomly collected at each sampling interval in two phases. Soil cores (approximately 2.25-inch i.d.) were collected from the 0- to 6-inch depth using a two-stage hydraulic probe with an acetate sleeve (p. 15). A second core (approximately 1.75-inch i.d.) was then collected to a depth of 48 inches. The 6- to 48-inch cores were sectioned into 6-inch increments (p. 16) and all cores were composited by depth increment (into three replicates of five cores each) at each sampling interval. In the control plots, 15 soil cores were collected and combined by depth (6-inch increments) into a single composite sample. At 0 and 3 days after treatment (DAT), 15 additional samples were collected from each site (using a bulb planter) from the 0- to 1- or 2-inch depths and were used in estimating the concentration of fluroxypyr initially present in the soil. All samples were frozen shortly (time interval unspecified) after collection and stored frozen until analysis. To assess the stability of fluroxypyr and its metabolites during shipping and handling, two soil samples from each site were fortified separately with fluroxypyr, pyridinol, and methoxypyridine at 142, 14.2, and 28.4 ng/g fluroxypyr equivalents, respectively (p. 17) at 0 DAT, 1 or 2 DAT, 3 or 4 DAT, 6 months after treatment (MAT), and 12 MAT. The 0 DAT soil samples were also fortified with fluroxypyr MHE at 142 ng/g (fluroxypyr acid equivalents). Mean recoveries for fluroxypyr, pyridinol, and methoxypyridine (averaged across all sampling intervals and study sites) were
85% ± 33, 64% ± 15, and 96% ± 15, respectively (p. 19; see Comment #9). The registrant stated that the low recoveries for pyridinol reflected uncertainties in quantifying this analyte at a fortification level (approximately 11.1 ng/g moist soil) that was near its limit of quantitation (10 ng/g oven-dry soil; p. 19). Mean recovery of total fluroxypyr (fluroxypyr MHE plus fluroxypyr acid equivalent) was 118% ± 7.

Missouri: Fluroxypyr MHE (XRM-5316, 26.6% a.i.; EC formulation) was applied as a single postemergence spray at 0.25 a.e./A onto a plot (18000 square feet with six designated subplots of 30 feet x 100 feet, 0 to 1% slope; pp. 13, 67) of Putnam silty clay loam soil (7.2% sand, 64.8% silt, 28.0% clay, 1.82% organic matter, pH 7.5, CEC 13.72 meq/100 g; Table 1, p. 28) vegetated with spring wheat at the 1-leaf growth stage near Leonard, MO. A seven-year plot history indicated no prior usage of fluroxypyr or related compounds (p. 14). The depth to the seasonal high water table was estimated by the registrant to be 70 feet (p. 13). Total water input via precipitation and irrigation was 137% of the 30-year historical precipitation average (Table III, p. 31). Environmental conditions were measured at Shelbina, MO, 15 miles from the test site; pan evaporation data were not reported. Soil cores were collected randomly at 0, 3, 7, 14, and 21 DAT, and 1, 2, 3, 4, 5, 6, 10, and 12 MAT (p. 134); samples were stored 81-525 days prior to analysis.

North Dakota: Fluroxypyr MHE (XRM-5316, 26.6% a.i.; EC formulation) was applied as a single postemergence spray at 0.25 a.e./A onto a plot (15600 square feet with six designated subplots of 16.67 feet x 100 feet, 0 to 1% slope; pp. 13, 68) of Gardena sandy clay loam soil (59.2% sand, 16.8% silt, 24.0% clay, 2.87% organic matter, pH 6.2, CEC 15.55 meq/100 g; Table 1, p. 28) vegetated with spring wheat at the 1- to 2-leaf growth stage near Larimore, ND. A seven-year plot history indicated no prior usage of fluroxypyr or related compounds (p. 14). The depth to the seasonal high water table was estimated by the registrant to be 2.5 feet or less (p. 13). Total water input via precipitation and irrigation was 135% of the 30-year historical precipitation average (Table IV, p. 32). Environmental conditions were measured at Grand Forks, ND, 25 miles from the test site; pan evaporation data were not reported. Soil cores were collected randomly at 0, 3, 7, 14, and 21 DAT, and 1, 2, 3, 4, 5, 6, 12, and 18 MAT (p. 134); samples were stored 80-508 days prior to analysis.

Washington: Fluroxypyr MHE (XRM-5316, 26.6% a.i.; EC formulation) was applied as a single postemergence spray at 0.25 a.e./A onto a plot (14400 square feet with six designated subplots of 15 feet x 100 feet, <2% slope; pp. 13, 68) of Quincy loamy sand soil (81.2% sand, 12.8% silt, 6.0% clay, 0.88% organic matter, pH 6.8, CEC 6.80 meq/100 g; Table 1, p. 29) vegetated with spring wheat at the 1- to 2-leaf growth stage near Ephrata, WA. A seven-year plot history indicated no prior usage of fluroxypyr or related compounds (p. 14). The depth to the seasonal high water table was estimated by the registrant to be 47 feet (p. 13). Total water input via precipitation and typical irrigation was 153% of the 30-year historical precipitation average (Table V, p. 33). Environmental conditions were measured at Wenatchee, WA, 25 miles from the test site; pan evaporation data were not reported. Soil cores were collected randomly at 0, 3, 7, 13, and 21 DAT, and 1, 2, 3, 4, 5, 6, and 12 MAT (p. 134); samples were stored 92-693 days prior to analysis.

Soil samples collected from all three field sites were analyzed for fluroxypyr MHE, fluroxypyr (acid), pyridinol, and methoxypyridine. Fluroxypyr MHE was extracted (by shaking) with acetone/0.1 *N* HCl (90:10, v:v). After evaporation of the acetone, the extract was diluted with additional 0.1 *N* HCl and applied to a C18 solid phase extraction (SPE) column. The column was dried and residues were eluted with an iso-octane/ether solution (ratios unspecified). The ether was removed by evaporation and the iso-octane extract was analyzed by GC (DB-WAX column; MRID 44080355, p. 12) with electron capture detection (ECD). The method for determining fluroxypyr MHE was designated analytical method ACR 91.10 (p. 121). The limit of detection (LOD) and the limit of quantitation (LOQ) were 8 and 26 ng/g, respectively (p. 123; see Comment #4).

Methoxypyridine, pyridinol, and fluroxypyr (acid) were extracted as described above, except that the method was modified to include a robotic system of extraction (designated analytical method GRM 93.03; pp. 122, 123). For methoxypyridine determinations, the acid extract (remaining after evaporation of the acetone, as described above) was adjusted to a basic pH (unspecified) and extracted with hexane. The hexane extract was applied to a silica gel column and eluted with methyl-t-butyl ether (MTBE); methyoxypyridine residues were extracted into decane and analyzed by GC/MS (DB-5 capillary or DB-1701 columns; MRID 44080355, p. 12) with the detector operating in the selective ion monitoring (SIM) mode. The LOD and LOQ were 4 and 14 ng/g, respectively (p. 123; see Comment #4).

For pyridinol and fluroxypyr determinations (analytical method GRM 93.03; pp. 122, 123), the acid extract (from the initial extraction) was applied to a C18 column and fluroxypyr acid equivalent and pyridinol residues were eluted with ethyl ether:hexane (70:30, v:v). The eluent was diluted with MTBE and separated into two aliquots. From one aliquot, fluroxypyr was extracted with acetone (acidified with phosphoric acid to unspecified pH), derivatized with trimethylsilyl-diazomethane, extracted into toluene, and analyzed by GC/MS (DB-WAX or DB-1701 columns; MRID 44080355, p. 12) with the detector operating in the selective ion monitoring (SIM) mode. The LOD and LOQ were 4 and 12 ng/g, respectively (p. 123; see Comment #4).

The registrant stated that data reported for treated samples were expressed on a fluroxypyr acid equivalent basis and summed across all depths to account for rapid hydrolysis of the parent to fluroxypyr acid and for losses due to leaching (p. 20).

Mean analytical method recovery from 66 soil samples fortified with the parent at 10-500 ng/g was 104% ± 19 (Table II, pp. 135, 136). Mean recovery of fluroxypyr (acid) from 117 samples fortified at 10-1000 ng/g was 82 ± 11% (Table III, pp. 137-140). Mean recovery of pyridinol from 112 samples fortified at 10-200 ng/g was 101% ± 14 (Table IV, pp. 141-144). Mean recovery of methoxypyridine from 117 samples fortified at 10-200 ng/g was 95% ± 14% (Table V, pp. 145-148). Sample data were corrected for soil

moisture and method recoveries. Occasional analytical method recovery sample sets were low (see Comment #5).

Storage Stability Study (MRID 44080355)

In a separate storage stability (MRID 44080355), subsamples (150-200 g) of Catlin silty clay loam soil obtained from Henry County, IL (soil characterization data were not submitted; see Comment #1), were fortified separately with fluroxypyr MHE, fluroxypyr acid equivalent, and pyridinol at 0.10 μg/g for each compound (p. 10) and stored frozen (temperature not specified); 20 g soil subsamples were fortified with 0.10 μg/g methoxypyridine (see Comment #6). Samples fortified with the parent were analyzed (analytical method ACR 91.10) after 0, 3, 8, 11, 32, 92, 119, 200, and 422 days of storage. Samples fortified with fluroxypyr acid equivalent and pyridinol were analyzed (method GRM 93.03) after 0, 7, 31, 92, 199, 429, and 938 days of storage (pp. 10, 11). Samples fortified with methoxypyridine were analyzed (analytical method GRM 93.03) after 0, 3, 7, 30, 92, 184, 414, and 923 days for storage (pp. 10, 11). Recoveries of parent wee 94-107% after 0-938 days of storage (p. 13); recoveries of pyridinol wee 90-103% after 0-938 days of storage (p. 14); and recoveries of methoxypyridine were 92-108% after 0-923 days of storage. Samples were analyzed in duplicate or triplicate and recovery data were corrected for concurrent procedural recoveries for each compound (pp. 18-29). Samples collected from all three sites were stored frozen (temperature not specified) 80-693 days prior to analysis (pp. 10-14).

DATA SUMMARY

Fluroxypyr MHE (4-amino-3,5-dichloro-6-fluoro-2-pyridyloxyacetic, 1 methylheptyl ester) broadcast applied as a single postemergence spray at a nominal rate of 0.25 a.e./A (1x the proposed maximum rate) dissipated (as the sum of the parent plus fluroxypyr acid equivalent) in Putnam silty clay loam, Gardena sandy clay loam, and Quincy loamy sand soils in MO, ND, and WA, respectively, with respective registrant-calculated half-lives of 24.8, 36.3, and 13.2 days in plots vegetated with spring wheat; half-lives were calculated from 0 DAT (see Comment #7). The parent and fluroxypyr (acid) were confined primarily to the 0- to 6-inch soil depth; no data were reported for analyses of the parent below the 6-inch depth at MO and WA. The fluroxypyr metabolites

4-amino-3,5-dichloro-6-fluoro-2-pyridinol (pyridinol) and

4-amino-3,5-dichloro-6-fluoro-2-methoxypyridine (methoxypyridine)

were detected at all three test locations primarily in the 0- to 6-inch soil depth. Total fluroxypyr residues were not reported in tabular form and residue concentration means were not reported for any sampling interval.

Missouri: Total fluroxypyr (fluroxypyr MHE plus fluroxypyr acid) dissipated with a registrant-calculated half-life of 24.8 days in Putnam silty clay loam soil vegetated with spring wheat (Figure 14, p. 48; see Comment #7). In the 0- to 6-inch depth, fluroxypyr MHE was initially observed at soil concentrations of 53 and 75 ng/g (in two of three composite samples), was <26-88 ng/g by 3 DAT, and was <26 and 29 ng/g (in two of three composite samples) at 7 DAT (Table X, pp. 157, 158). Samples were not analyzed for fluroxypyr MHE after 7 DAT.

In the 0- to 6-inch depth, fluroxypyr (acid) was observed at soil concentrations of 94-120 ng/g at 0 DAT, was 100-150 ng/g by 3 DAT, and increased to a maximum of 160-190 ng/g by 7 DAT (Tables XII-XIV, pp. 163, 164). Pyridinol was not detectable in any 6-inch depth increment down to 18 inches. Methoxypyridine was first observed in the 0- to 6-inch depth at <10 ng/g (in two of three composite samples) at 14 DAT, was variable (10-26 ng/g) between 21 DAT and 10 MAT, and was <10 ng/g (in two of three composite samples) at 12 MAT (Tables XIV-XIX, pp. 162-167). Methoxypyridine was not detected below the 6-inch depth.

In samples obtained from the 0- to 1- or 2-inch depths at 0 and 3 DAT, concentrations of the parent and metabolites were substantially higher. In the 0- to 2-inch depth (0 DAT, 15 samples) concentrations of the parent and fluroxypyr acid equivalent were 200-870 ng/g and 350-850 ng/g, respectively (Table X, pp. 157-158; Table XII, p. 160). In the 0- to 1-inch depth (3 DAT, 15 subsamples)concentrations of the parent and fluroxypyr (acid) were 73-390 ng/g and 490-1100 ng/g, respectively; pyridinol was observed at concentrations of <10-17 ng/g (Table X, pp. 157, 158; Table XIII, p. 161). Methoxypyridine was not detectable in the 0- to 1-inch or 2-inch depths at 0 and 3 DAT. The 0- to 1- or 2-inch depths were not analyzed after 3 DAT.

North Dakota: Total fluroxypyr (fluroxypyr MHE plus fluroxypyr acid) dissipated with a registrant-calculated half-life of 36.3 days in Gardena sandy clay loam soil vegetated with spring wheat (Figure 15, p. 49; see Comment #7). In the 0- to 6-inch depth, fluroxypyr MHE was initially observed at soil concentrations of 87-100 ng/g at 0 DAT, was 91-140 ng/g by 3 DAT, and was 51-70 ng/g at 7 DAT (Table XX, pp. 169, 170). Samples were not analyzed for fluroxypyr MHE after 7 DAT.

In the 0- to 6-inch depth, fluroxypyr (acid) was observed at soil concentrations of 70-77 ng/g at 0 DAT, increased to 140-220 ng/g by 7 DAT, and plateaued at 92-160 ng/g between 14 DAT and 1 MAT (Tables XXII-XXV, pp. 172-175). Fluroxypyr acid them decreased to 19-48 ng/g by 2 MAT, <10-14 ng/g by 3 MAT, and was not detectable by 6 MAT (Tables XXVI-XXX, pp. 176-180). Fluroxypyr (acid) was not detected below the 6-inch depth, except at 7 DAT where it was detected at 11-30 ng/g in the 6- to 12-inch depth. Pyridinol was first observed in the 0- to 6-inch depth at <10 ng/g (in one of three samples) at 14 DAT, was <10-12 ng/g at 2 MAT and was not detectable at 3 MAT; pyridinol was not detected below the 6-inch depth at any sampling interval.

Methoxypyridine was first observed in the 0- to 6-inch depth at <10 ng/g at 21 DAT, was variable (24-50 ng/g) between 2 and 14 MAT, and was 21-30 ng/g at 18 MAT. Methoxypyridine was not detected below the 6-inch depth.

In samples obtained from the 0- to 2-inch depth at 0 and 3 DAT, concentrations of the parent and metabolites were substantially higher. In the 0- to 2-inch depth (0 DAT, 15 samples) concentrations of the parent and fluroxypyr (acid) were 210-440 ng/g and 220-420 ng/g, respectively (Table XX, pp. 169, 170; Table XXII, p. 172); pyridinol was observed at concentrations of <10 ng/g (in four of 15 samples). At 3 DAT (15 samples), concentrations of the parent and fluroxypyr acid equivalent were 140-530 ng/g and 240-1600 ng/g, respectively (Table XX, pp. 169, 170; Table XXIII, p. 173). Pyridinol (at 3 DAT) and methoxypyridine (at 0 and 3 DAT) were not detectable. The 0- to 2-inch depths were not analyzed after 3 DAT.

Washington: Total fluroxypyr (fluroxypyr MHE plus fluroxypyr acid) dissipated with a registrant-calculated half-life of 13.2 days in Quincy loamy sand soil vegetated with spring wheat (Figure 16, p. 50; see Comment #7). In the 0- to 6-inch depth, fluroxypyr MHE was initially observed at soil concentrations of 54-72 ng/g at 0 DAT, decreased to <26-47 ng/g by 3 DAT, and was <26 ng/g (composite of 3 samples) at 7 DAT (Table XXXI, pp. 182, 183). Samples were not analyzed for fluroxypyr MHE after 7 DAT. Residue concentration means were not reported.

In the 0- to 6-inch depth, fluroxypyr (acid) was observed at soil concentrations of 120-150 ng/g at 0 DAT, increased to a maximum of 180 ng/g (composite of three samples) by 7 DAT, and decreased to 94-100 ng/g by 14 DAT, 35-44 ng/g by 21 DAT, and <10-16 ng/g (in two of three samples) by 1 MAT, and was not detectable by 2 MAT (Tables XXXIII-XXXVII, pp. 185-189). Fluroxypyr (acid) was not detected below the 6-inch depth, except at 21 DAT where it was detected at <10-14 ng/g in the 6- to 12-inch depth. Pyridinol was first observed in the 0- to 6-inch depth at <10 ng/g between 3 and 21 DAT, and was not detectable at 1 MAT; pyridinol was not detected below the 6-inch depth at any sampling interval. Methoxypyridine was first observed in the 0- to 6-inch depth at <10 ng/g at 14 DAT, was variable (17-25 ng/g) between 21 DAT and 1 MAT, decreased to <10 ng/g by 3 MAT, and was sporadically detected at <10 ng/g between 4 and 12 MAT (Tables XXXVIII-XL, pp. 190-192). Methoxypyridine was not detected below the 6-inch depth, except at 2 MAT where it was observed at <10 ng/g in the 6-12-inch and 12- to 18-inch soil depths.

COMMENTS

1. The soils used in the field dissipation study (MRID 44080347; 0- to 6-inch depth, Table I, pp. 28, 29) were Putnam silty clay loam (MO), Gardena sandy clay loam (ND), and Quincy loamy sand (WA). A Catlin silty clay loam soil from a site in Henry County, IL,

was used to assess the stability of fluroxypyr MHE and its metabolites in frozen storage (MRID 44080355, p.10). The soil used in the storage stability study was not representative of the soils found at the test sites, particularly in ND and WA. Differences in texture, bulk density, moisture content, CEC, pH, and/or microbial populations/activity could differentially affect the stability of the analytes in frozen storage. It is recommended that storage stability studies be conducted using soils obtained from each test site.

2. Application rates were not verified by use of monitoring pads or other valid means. Expected soil concentrations of total fluroxypyr residues were not calculated by the registrant, nor were the soil concentration data expressed as total fluroxypyr residues in tabular form. However, recoveries of total fluroxypyr from soil on the day of application were reported as 76.0%, 88.7%, and 104.8% of the applied (0- to 6-inch depth) in MO, ND, and WA, respectively; respective recoveries were 134.1%, 122.1%, and 127.3% of the applied in the 0- to 2-inch depth (Table VI, p. 34).

   In the absence of soil monitoring pad data, application rates may be verified by comparison of the expected 0 DAT soil concentrations of total fluroxypyr residues (fluroxypyr MHE plus fluroxypyr acid) at each site with the measured 0 DAT soil concentrations. The registrant should provide total (summed) residue concentration data for total fluroxypyr in tabular form and calculate mean concentrations for these compounds (and the metabolites) for each sampling interval. The registrant should also calculate the expected concentrations of total fluroxypyr at 0 DAT. Separate calculations of the expected total fluroxypyr concentrations in soil should be made for each site due to differences in soil bulk density.

3. Detailed descriptions of the instrumentation used with residue analytical methods ACR 91.10 and GRM 93.03 were not included in the terrestrial dissipation study (MRID 44080347), but were described in the accompanying storage stability study (MRID 44080355).

4. The limits of detection (LOD) and limits of quantitation (LOQ) for each analyte were calculated by the registrant from the recovery data obtained from soil samples fortified with each analyte at 10 ng/g (p. 123). The registrant stated that the target LOD and LOQ values (5 and 10 ng/g, respectively) for the fluroxypyr (acid), pyridinol, and methoxypyridine were used in this study because they were the lowest levels at which the samples were fortified with these analytes (pp. 123, 124). For fluroxypyr MHE (the parent), the registrant-calculated statistical values for LOD and LOQ (8 and 26 ng/g, respectively) were used in this study because they were higher than the target values.

5. Concurrent analytical method recoveries of the parent and its metabolites from soil samples fortified separately with each analyte at 10-500 ng/g soil were variable. Field sample data were corrected for method recoveries on their respective days of analysis.

Analytical method recoveries of fluroxypyr MHE from 66 soil samples fortified at 10-300 ng/g were 46-142% of the applied (Table II, pp. 135, 136), including one set of six samples that were 46-96% (p. 136); recoveries from 53 of 66 samples were ≥70% and ≤120%.

Analytical method recoveries of fluroxypyr (acid) from 117 samples fortified at 10-500 ng/g were 58.6-118.2% of the applied (Table III, pp. 137-140); recoveries from 100 of 117 samples were ≥70% and ≤120%.

Analytical method recoveries of pyridinol from 112 samples fortified at 10-200 ng/g were 60.9-128.6% of the applied (Table IV, pp. 141-144); recoveries from 102 of 112 samples were ≥70% and ≤120%.

Analytical method recoveries of methoxypyridine from 117 samples fortified at 10-200 ng/g were 53.8-128.0% of the applied (Table V, pp. 145-148); recoveries from 112 of 117 samples were ≥70% and ≤120%.

6. Soil subsamples to be fortified with fluroxypyr MHE, fluroxypyr (acid), and pyridinol (150-200 g) were initially weighed into plastic bags, separately fortified with each compound (dissolved in acetone) at 0.10 μg/g soil, and transferred to metal paint cans prior to frozen storage. The registrant stated that due to the volatility of methoxypyridine, 20-g subsamples of soil were weighed directly into the metal paint cans, then fortified with 0.10 μg methoxypyridine/g soil.

7. The registrant assumed first-order dissipation kinetics for total fluroxypyr residues (fluroxypyr MHE plus fluroxypyr) at each site and plotted best-fit curves. Half-lives were calculated from 0 DAT as well as from the sampling interval where peak concentrations (3 or 7 DAT) of total fluroxypyr were observed. Half-lives calculated from the 0 DAT sampling interval were reported above. Half-lives calculated using the peak concentration sampling intervals as time = 0 were 9.0 or 16 days, 14.3 or 21.3 days, and 9.6 or 12.6 days in MO, ND, and WA, respectively. The two values reported for each site reflect discrepancies between the half-lives reported in Figures 14-16 (first value; pp. 48-50) and the text (second value; p. 22). No explanation was provided for the discrepancies in the reported half-lives between the Figures 14-16 and the text (p. 22). Additionally, dissipation curves were presented as the disappearance of the "percent of applied, on an acid equivalent (a.e.) basis." It is not clear whether the half lives of total fluroxypyr were calculated using the "percent of applied" values or using the measured total fluroxypyr soil concentrations. Clarification of this issue by the registrant is necessary.

Additionally, because half-life calculations were determined based on the dissipation of fluroxypyr MHE plus fluroxypyr (acid), the registrant should provide total (summed)

residue concentration data for total fluroxypyr in tabular form and report mean concentrations for these compounds (and the metabolites) for each sampling interval.

8. It is preferred that soil residue concentration data be presented in tabular form as the mean concentrations of all the replicates for a given sampling interval, in addition to the values presented for the individual replicates.

9. Field spike recoveries for all analytes across all sites and sampling intervals were extremely variable. Recoveries of fluroxypyr (acid), pyridinol, and methoxypyridine ranged 35% to 241%, 34% to 85%, and 65% to 116%, respectively (Tables XLI-LVI, pp. 194-209). The impact on the study of unusually low or high recoveries of analytes from field spike samples at particular sampling intervals was not discussed by the registrant.