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CONCLUSIONS

Metabolism - Aerobic Soil

1. This study is scientifically valid and provides useful information on the aerobic soil metabolism of metsulfuron methyl. However, duplicate samples were not utilized for analysis at each sampling interval. Typically, a study is considered valid only if a minimum of duplicate samples are prepared and incubated for removal at each sampling interval.

2. The study meets Subdivision N Guidelines for the fulfillment of EPA data requirements on aerobic soil metabolism.

3. Radiolabeled [^{14}C]metsulfuron methyl (both labels), at a nominal application rate of 0.1 ppm, degraded with registrant-calculated half-lives (reported as DT_{50}'s; bi-exponential equation; 0- to 4-month data only) of 10 days (r^2 = 1.0; phenyl label) and 11 days (r^2 = 0.99; triazine label) in silt loam soil adjusted to 75% of 0.33 bar moisture content and incubated in darkness at 20°C for up to 12 months. In sterile control soil samples, the parent compound degraded with a registrant-calculated half-life of 23 days (r^2 = 1.0; both labels; 0- to 4-month data only). However, sterility was maintained in the sterile control samples only through 6 months posttreatment. All data, reported as percentages of the applied, represent percentages of the nominal application. Residue concentration data (parent compound and degradates; in ppm based on parent equivalents) were reviewer-calculated based on the nominal application rate and the reported percentages of the applied radioactivity.

   In the phenyl label study, the parent was initially 98.0% (0.098 ppm) of the applied radioactivity, decreased to 60.2% (0.060 ppm) by 7 days and 43.6% (0.044 ppm) by 10 days, was 23.3% (0.023 ppm) at 1 month and 7.9% (0.0079 ppm) at 2 months, and was 1.4-1.9% (0.0014-0.0019 ppm) at 6-12 months posttreatment. The major degradate IN-NC148 was initially (day 3) 1.9% (0.0019 ppm) of the applied radioactivity, was a maximum of 15.7% (0.016 ppm) at 3 months, and was 2.1% (0.0021 ppm) at 12 months posttreatment. The major degradate IN-B5067 was initially (time 0) 3.1% (0.0031 ppm) of the applied radioactivity, was a maximum of 11.3% (0.011 ppm) at 10 days, and was 1.8% (0.0018 ppm) at 12 months posttreatment. The major degradate IN-D5803 was initially (time 0) 1.5% (0.0015 ppm) of the applied radioactivity, was a maximum of 11.2% (0.011 ppm) at 14 days, and was last detected at 3.9% (0.0039 ppm) at 1 month posttreatment. The minor degradate IN-F5438 was initially (day 1) 1.2% (0.0012 ppm) of the applied radioactivity, was a maximum of 9.2% (0.0092 ppm) at 21 days, and was last detected at 0.7% (0.0007 ppm) at 4 months posttreatment. The minor degradate IN-581 was initially (day 3) 2.8% (0.0028 ppm) of the applied radioactivity, was a maximum of 8.7% (0.0087 ppm) at 21 days, and was 2.0% (0.0020 ppm) at 12 months posttreatment. The minor degradate IN-B5685 was detected sporadically at ≤2.8%

(≤0.0028 ppm) of the applied radioactivity throughout the incubation period. Nonextractable [^{14}C]residues were initially (time 0) 0.2% of the applied radioactivity, increased to 16.0 % by 10 days, were a maximum of 40.8% at 6 months, and were 31.1% at 12 months posttreatment; [^{14}C]residues associated with the humic acid, fulvic acid and humin fractions were 14.3%, 0.92%, and 14.0% of the applied radioactivity at 4 months posttreatment, respectively. Evolved $^{14}CO_2$ initially (day 3) accounted for 0.4% of the applied radioactivity, increased to 26.3% by 2 months, and was 48.2% at 12 months posttreatment; [^{14}C]organic volatiles were ≤1.7% of the applied radioactivity throughout the incubation period. In the sterile control samples (phenyl label), the parent was initially 107.3% (0.11 ppm) of the applied radioactivity, decreased to 44.7% (0.045 ppm) by 1 month and 17.3% (0.017 ppm) by 2 months, and was 2.5-4.3% (0.0025 ppm) from 6 to 12 months posttreatment. The major degradate IN-581 was initially (1 month) 9.5% (0.0095 ppm) of the applied radioactivity, increased to 31.7% (0.032 ppm) by 4 months, and was a maximum of 43.2% (0.043 ppm) at 12 months posttreatment. The major degradate IN-B5067 was initially (time 0) 2.5% (0.0025 ppm) of the applied radioactivity, increased to 17.0% (0.017 ppm) by 6 months, and was a maximum of 34.4% (0.034 ppm) at 12 months posttreatment. The major degradate IN-D5803 was initially (1 month) 9.5% (0.0095 ppm) of the applied radioactivity, was a maximum of 25.2% (0.025 ppm) at 6 months, and was 1.3% (0.0013 ppm) at 12 months posttreatment. The major degradate IN-MU717 was initially (1 month) 10.5% (0.011 ppm) of the applied radioactivity, was a maximum of 20.9% (0.021 ppm) at 6 months, and was last detected at 13.9% (0.014 ppm) at 9 months posttreatment. Nonextractable [^{14}C]residues were initially (1 month) 4.9% of the applied radioactivity, were a maximum of 14.4% at 9 months, and were 13.4% at 12 months posttreatment. Evolved $^{14}CO_2$ was detected once, at 0.4% of the applied radioactivity at 12 months posttreatment; [^{14}C]organic volatiles were negligible.

In the triazine label study, the parent was initially 94.4% (0.094 ppm) of the applied radioactivity, decreased to 52.8% (0.053 ppm) by 10 days and 33.2% (0.033 ppm) by 14 days, and was 1.5-2.4% (0.0015-0.0024 ppm) at 4-12 months posttreatment. The major degradate IN-A4098 was initially (time 0) 3.0% (0.003 ppm) of the applied radioactivity, was a maximum of 32.9% (0.033 ppm) at 3 months posttreatment, and was 17.4% (0.017 ppm) at 12 months. The major degradate IN-NC148 was initially (day 10) 1.4% (0.0014 ppm) of the applied radioactivity, was a maximum of 9.9% (0.0099 ppm) at 2 months, and was 8.0% (0.0080 ppm) at 12 months posttreatment. The minor degradate IN-B5528 was initially (time 0) 2.3% (0.0023 ppm) of the applied radioactivity, was a maximum of 9.0-9.2% (0.0090-0.0092 ppm) at 7-10 days, and was 7.7% (0.0077 ppm) at 12 months posttreatment. The minor degradate IN-B5067 was initially (time 0) 2.9% (0.0029 ppm) of the applied radioactivity, was a maximum of 8.9% (0.0089 ppm) at 3 days, and generally decreased to 3.6% by 12 months posttreatment. The minor degradate IN-F5438 was initially (day 7) 3.6% (0.0036 ppm) of the applied radioactivity, was a maximum of 8.2% (0.0082 ppm) at 1 month, and was last detected at 1.2% (0.0012 ppm) at 4 months posttreatment. Nonextractable [^{14}C]residues were initially (time 0) 0.9% of the applied

radioactivity, were a maximum of 34.8% at 7 months, and were 31.6% at 12 months posttreatment; [^{14}C]residues associated with the humic acid, fulvic acid and humin fractions were 12.5%, 0.43%, and 12.8% of the applied radioactivity at 4 months posttreatment, respectively. Evolved $^{14}CO_2$ initially (day 3) accounted for 0.3% of the applied radioactivity, was 10.3-18.7% from 2 to 9 months, and was 22.8% at 12 months posttreatment; [^{14}C]organic volatiles were negligible. In the sterile control samples (triazine label), the parent was initially 101.6% (0.10 ppm) of the applied radioactivity, decreased to 41.3% (0.041 ppm) by 1 month and 18.8% (0.019 ppm) by 2 months, and was 2.0% (0.0020 ppm) at 12 months posttreatment. The major degradate IN-B5067 was initially (time 0) 2.5% (0.0025 ppm) of the applied radioactivity, was a maximum of 31.3% (0.031 ppm) at 9 months, and was 25.8% (0.026 ppm) at 12 months posttreatment. The major degradate IN-A4098 was initially (1 month) 20.8% (0.021 ppm) of the applied radioactivity, was a maximum of 26.2% (0.026 ppm) at 5 months, and was 18.1% (0.018 ppm) at 12 months posttreatment. The major degradate IN-MU717 was initially (1 month) 10.1% (0.010 ppm) of the applied radioactivity, was a maximum of 22.2% (0.022 ppm) at 4 months, and was last detected at 14.9% (0.015 ppm) at 6 months posttreatment. The major degradate IN-B5528 was initially (time 0) 1.7% (0.0017 ppm) of the applied radioactivity, was a maximum of 17.8% (0.018 ppm) at 6 months, and was 10.1% (0.010 ppm) at 12 months posttreatment. Nonextractable [^{14}C]residues were initially (time 0) 0.4% of the applied radioactivity, were 13.5-17.9% at 2-5 months, and were a maximum of 27.3% at 12 months posttreatment. Evolved $^{14}CO_2$ accounted for 0.1-0.6% of the applied radioactivity at 2-9 months and was 1.8% at 12 months posttreatment; [^{14}C]organic volatiles were negligible.

METHODOLOGY

Samples (50 g) of sieved (2 mm) Matapeake silt loam soil (from the DuPont Agricultural Products Soil Bank; 27.6% sand, 55.6% silt, 16.8% clay, 1.8% organic matter, pH 5.2, CEC 7.0 meq/100 g; Table 1, p. 31) were weighed into Pyrex glass flasks and treated with uniformly phenyl ring-labeled [^{14}C]metsulfuron methyl {DPX-T6376; methyl 2-[[[[(4-methoxy-6-methyl-1,3,5,triazin-2-yl)amino]-carbonyl]amino]sulfonyl]benzoate; radiochemical purity >99%, specific activity 38.3 μCi/mg; p. 12; Figure 1, p. 42} OR triazine ring-labeled [2-^{14}C]metsulfuron methyl {radiochemical purity >99%, specific activity 49.9 μCi/mg}, dissolved in water, at a nominal application rate of 0.1 ppm (p. 14). Samples were adjusted to 75% of the soil moisture content at 0.33 bar and incubated in darkness at 20°C for up to 12 months (p. 16). The soil moisture level was monitored and adjusted as necessary throughout the incubation period. Moist, CO_2-free air was passed through the samples and into two ethylene glycol traps, a polyurethane foam plug trap, and two 1 *N* KOH traps containing phenolphthalein indicator (Figure 6, p. 59). Additional soil samples treated at an exaggerated rate of 1 ppm were prepared for metabolite isolation (p. 14). Sterile control samples were prepared using autoclaved soil and incubated as previously described (p. 16). Single samples were removed for analysis

at 0, 1, 3, 7, 10, 14, and 21 days posttreatment; and at 1, 2, 3, 4, 6, 7 (triazine label only), 9 and 12 months posttreatment. Volatile trap solutions were collected for analysis and replaced with fresh trapping solution at each sampling interval (p. 17).

At each sampling interval, soil samples were extracted three times by shaking with acetonitrile:2 M ammonium carbonate (90:10, v:v) and centrifuged (p. 18). The supernatants were decanted and combined, and triplicate aliquots of the extracts were analyzed for total radioactivity by LSC. Selected soil samples (those which contained >10% of the applied radioactivity as bound residues following the first extraction) were further extracted three times with CH_2Cl_2:methanol:2 M ammonium carbonate (3:4:1, v:v:v), and triplicate aliquots of the combined extracts were analyzed by LSC. The extracts from the first and second extraction procedures were combined, concentrated by rotary evaporation, redissolved in water and acetone, concentrated under nitrogen flow, and analyzed in triplicate by LSC. An aliquot of each extract was filtered and analyzed by HPLC (Zorbax Rx-C8 column) using a mobile phase gradient of water (pH 2.3, H_3PO_4):acetonitrile (100:0 to 85:15 to 60:40 to 0:100, v:v) with UV (unspecified wavelength) and radiochemical detection (p. 20); eluent fractions were collected and analyzed by LSC. Samples were co-chromatographed with nonradiolabeled reference standards of the parent and the following potential degradates: IN-581, IN-B5685, IN-D5803, IN-B5067, IN-F5438, and IN-MU717 (phenyl label study); *OR* IN-B5528, IN-A4098, IN-B5067, IN-F5438, and IN-MU717 (triazine label study; p. 15; Figure 4, pp. 49-53). To confirm compound identities, samples were analyzed by HPLC (PRP-1 column) using a mobile phase gradient of water (pH 2.3, H_3PO_4):acetonitrile (90:10 to 80:20 to 60:40 to 10:90, v:v) with UV (unspecified wavelength) and radiochemical detection. Samples were co-chromatographed with nonradiolabeled reference standards as previously described. To further confirm compound identities in the phenyl label study, samples were also analyzed by HPLC (Zorbax Rx-C8 column) using a mobile phase gradient of water (pH 2.3, H_3PO_4):acetonitrile (100:0 to 85:15 to 60:40 to 0:100, v:v) with UV (unspecified wavelength) and radiochemical detection. Samples were co-chromatographed with nonradiolabeled reference standards as previously described. To further identify metabolites, samples were analyzed by LC/MS (Hypercarb 7 μm Graphite or Zorbax SB-C18 column) using a mobile phase gradient of water:acetonitrile (both with 0.05% formic acid; 98:2, v:v; solvent system A) to acetonitrile:water (both with 0.05% formic acid; 98:2, v:v; solvent system B; A:B 100:0 to 10:90 for Hypercarb; A:B 95:5 to 10:90 for Zorbax) with electrospray ionization detection (p. 21; Appendix 3, p. 124).

Post-extracted soil samples were analyzed by LSC following combustion (p. 20). To characterize nonextractable residues, four selected (4 months; p. 29) subsamples of post-extracted soil were extracted by shaking with 1 *N* NaOH followed by centrifugation (Appendix 6, p. 145); the supernatants were decanted and combined. The soil samples were rinsed twice with 1 *N* NaOH, and the rinsate was combined with the extracts. Triplicate aliquots of the extracts were analyzed for total radioactivity by LSC. The

caustic extracts were acidified (pH 2; HCl) to precipitate humic acids. Following centrifugation, the supernatant was decanted and triplicate aliquots were analyzed by LSC to determine radioactivity associated with the fulvic acid fraction. The remaining precipitate was redissolved with 1 *N* NaOH and triplicate aliquots were analyzed by LSC to determine radioactivity associated with the humic acid fraction. The post-extracted soil samples were analyzed for total radioactivity by LSC following combustion to determine the nonextractable [^{14}C]residues associated with the humin fraction.

Triplicate aliquots of the volatile trap solutions were analyzed for total radioactivity by LSC (p. 17). The presence of $^{14}CO_2$ in the KOH traps was confirmed by $BaCl_2$ precipitation (p. 26; Appendix 5, p. 144). The polyurethane foam plugs were extracted three times with acetonitrile:2 M ammonium carbonate (90:10, v:v); triplicate aliquots of the extracts were analyzed by LSC.

To determine soil viability, soil samples were removed for microbial analysis at the initiation and termination of the study (p. 17). Microbial biomass was determined by glucose-induced respiration (p. 16). Bacteria and fungi were enumerated using plate count agar and Sabouraud agar, respectively. Results indicated that the soil was viable (Appendix 2, pp. 109-110).

Frozen storage stability samples were prepared in an identical manner to the test samples and placed in frozen storage at time 0 (p. 16). Single samples were removed from frozen storage at 1 and 12 months posttreatment (p. 17); data were not reported.

DATA SUMMARY

Radiolabeled [^{14}C]metsulfuron methyl (radiochemical purity >99%; both labels), at a nominal application rate of 0.1 ppm, degraded with registrant-calculated half-lives (reported as DT_{50}s; biexponential equation; 0- to 4-month data only; p. 21) of 10 days (r^2 = 1.0; phenyl label) and 11 days (r^2 = 0.99; triazine label) in silt loam soil adjusted to 75% of 0.33 bar moisture content and incubated in darkness at 20°C for up to 12 months (Table 10, p. 41; Figures 10, 11; pp. 66, 67). In the sterile control soil samples, the parent compound degraded with a registrant-calculated half-life of 23 days (r^2 = 1.0; both labels; 0- to 4-month data only; Figure 12, pp. 70-71). However, sterility was maintained in the sterile control samples only through 6 months posttreatment. All data, reported as percentages of the applied, represent percentages of the nominal application. Residue concentration data (parent compound and degradates; in ppm based on parent equivalents) were reviewer-calculated based on the nominal application rate and the reported percentages of the applied radioactivity.

Uniformly phenyl ring-labeled [^{14}C]metsulfuron methyl

The parent compound was initially present at 98.0% (0.098 ppm) of the applied radioactivity, decreased to 60.2% (0.060 ppm) by 7 days and 43.6% (0.044 ppm) by 10 days, was 23.3% (0.023 ppm) at 1 month and 7.9% (0.0079 ppm) at 2 months, and was 1.4-1.9% (0.0014-0.0019 ppm) of the applied at 6-12 months posttreatment (Table 6, p. 36). The major degradate

carbamoyl guanidine (IN-NC148; chemical name not provided)

was initially (day 3) detected at 1.9% (0.0019 ppm) of the applied radioactivity, increased to 10.9% (0.011 ppm) of the applied by 14 days posttreatment, was a maximum of 15.7% (0.016 ppm) of the applied at 3 months posttreatment, decreased to 11.2% (0.011 ppm) of the applied by 9 months posttreatment, and was 2.1% (0.0021 ppm) of the applied at 12 months posttreatment. The major degradate

methyl 2-[[[[(4-hydroxy-6-methyl-1,3,5-triazine-2-yl)amino]carbonyl] amino]sulfonyl]benzoate (IN-B5067)

was initially (time 0) detected at 3.1% (0.0031 ppm) of the applied radioactivity, increased to a maximum of 11.3% (0.011 ppm) of the applied by 10 days posttreatment, was 2.4-7.6% (0.0024-0.0076 ppm) of the applied from 14 days to 9 months posttreatment, and was 1.8% (0.0018 ppm) of the applied at 12 months posttreatment. The major degradate

methyl 2-(aminosulfonyl)benzoate (IN-D5803)

was initially (time 0) detected at 1.5% (0.0015 ppm) of the applied radioactivity, increased to a maximum of 11.2% (0.011 ppm) of the applied by 14 days posttreatment, and was last detected at 3.9% (0.0039 ppm) of the applied at 1 month posttreatment. The minor degradate 2-[[[[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)amino]carbonyl]amino] sulfonyl]benzoic acid (IN-F5438) was initially (day 1) detected at 1.2% (0.0012 ppm) of the applied radioactivity, was a maximum of 9.2% (0.0092 ppm) of the applied radioactivity at 21 days posttreatment, decreased to 4.4% (0.0044 ppm) of the applied by 2 months posttreatment, and was last detected at 0.7% (0.0007 ppm) of the applied at 4 months posttreatment. The minor degradate 1,2-benzisothiazol-3(2H)-one,1,1-dioxide (IN-581) was initially (day 3) detected at 2.8% (0.0028 ppm) of the applied radioactivity, was a maximum of 8.7% (0.0087 ppm) of the applied at 21 days posttreatment, and was 2.0% (0.0020 ppm) of the applied at 12 months posttreatment. The minor degradate methyl-2-[[(aminocarbonyl)amino[sulfonyl]benzoate (IN-B5685) was detected sporadically at ≤2.8% (≤0.0028 ppm) of the applied radioactivity throughout the incubation period. Two unidentified minor degradates (designated as P1 and P2) were each detected at ≤4.2% (≤0.0042 ppm) of the applied radioactivity. Nonextractable

[^{14}C]residues were initially (time 0) 0.2% of the applied radioactivity, increased to 16.0 % of the applied by 10 days posttreatment, were 20.2-29.9% of the applied from 14 days to 4 months posttreatment, increased to a maximum of 40.8% of the applied by 6 months posttreatment, and were 31.1% of the applied at 12 months posttreatment (Table 2, p. 32); [^{14}C]residues associated with the humic acid, fulvic acid and humin fractions were 14.3%, 0.92%, and 14.0% of the applied radioactivity at 4 months posttreatment, respectively (p. 29). Evolved $^{14}CO_2$ initially (day 3) accounted for 0.4% of the applied radioactivity, was 7.4-7.9% of the applied at 21 days to 1 month posttreatment, increased to 26.3% of the applied by 2 months posttreatment, and was 48.2% of the applied at 12 months posttreatment; [^{14}C]organic volatiles were $\leq$ 1.7% of the applied radioactivity throughout the incubation period.

In the sterile control samples, the parent compound was initially present at 107.3% (0.11 ppm) of the applied radioactivity, decreased to 44.7% (0.045 ppm) by 1 month and 17.3% (0.017 ppm) by 2 months, and was 2.5-4.3% (0.0025 ppm) of the applied from 6 to 12 months posttreatment (Table 8, p. 39; see Comment #5). The major degradate

IN-581

was initially (1 month) detected at 9.5% (0.0095 ppm) of the applied radioactivity, increased to 31.7% (0.032 ppm) of the applied by 4 months posttreatment, was 16.0-27.8% (0.016-0.028 ppm) of the applied at 6-9 months posttreatment, and was a maximum of 43.2% (0.043 ppm) of the applied at 12 months posttreatment. The major degradate

IN-B5067

was initially (time 0) detected at 2.5% (0.0025 ppm) of the applied radioactivity, increased to 17.0% (0.017 ppm) of the applied by 6 months posttreatment, and was a maximum of 34.4% (0.034 ppm) of the applied at 12 months posttreatment. The major degradate

IN-D5803

was initially (1 month) detected at 9.5% (0.0095 ppm) of the applied radioactivity, increased to a maximum of 25.2% (0.025 ppm) of the applied by 6 months posttreatment, and was 1.3% (0.0013 ppm) of the applied at 12 months posttreatment. The major degradate

acetyl triuret (IN-MU717; chemical name not provided)

was initially (1 month) detected at 10.5% (0.011 ppm) of the applied radioactivity, increased to a maximum of 20.9% (0.021 ppm) of the applied by 6 months posttreatment,

and was last detected at 13.9% (0.014 ppm) of the applied at 9 months posttreatment. Two unidentified minor degradates (designated as SP2 and SP3) were each detected at ≤3.9% (≤0.0039 ppm) of the applied radioactivity throughout the incubation period. Nonextractable [^{14}C]residues were initially (1 month) 4.9% of the applied radioactivity, increased to a maximum of 14.4% of the applied by 9 months posttreatment, and were 13.4% of the applied at 12 months posttreatment (Table 4, p. 34). Evolved $^{14}CO_2$ was detected once, at 0.4% of the applied radioactivity at 12 months posttreatment; [^{14}C]organic volatiles were negligible.

Material balances (based on LSC analysis) for the viable soils were 91.6-105.0% of the applied radioactivity (Table 2, p. 32), with no observed pattern of decline. Material balances (based on LSC analysis) for the sterile control samples were 91.4-109.8% of the applied radioactivity (Table 4, p. 34), with no observed pattern of decline.

Triazine ring-labeled [2-^{14}C]metsulfuron methyl

The parent compound was initially present at 94.4% (0.094 ppm) of the applied radioactivity, decreased to 52.8% (0.053 ppm) by 10 days and 33.2% (0.033 ppm) by 14 days, and was 1.5-2.4% (0.0015-0.0024 ppm) of the applied at 4-12 months posttreatment (Table 7, pp. 37-38). The major degradate

4-methoxy-6-methyl-1,3,5-triazin-2-amine (IN-A4098)

was initially (time 0) detected at 3.0% (0.003 ppm) of the applied radioactivity, was not detected from 1 to 10 days posttreatment, was 16.9% (0.017 ppm) of the applied at 14 days posttreatment, increased to a maximum of 32.9% (0.033 ppm) of the applied by 3 months posttreatment, and was 17.4% (0.017 ppm) of the applied at 12 months posttreatment (see Comment #4). The major degradate

IN-NC148

was initially (day 10) detected at 1.4% (0.0014 ppm) of the applied radioactivity, increased to a maximum of 9.9% (0.0099 ppm) of the applied by 2 months posttreatment, and was 8.0% (0.0080 ppm) of the applied at 12 months posttreatment. The minor degradate 4-amino-6-methyl-1,3,5-triazine-2-ol (IN-B5528) was initially (time 0) detected at 2.3% (0.0023 ppm) of the applied radioactivity, was a maximum of 9.0-9.2% (0.0090-0.0092 ppm) of the applied at 7-10 days posttreatment, was not detected from 14 days to 2 months posttreatment, was 1.4-2.0% (0.0014-0.0020 ppm) of the applied from 3 to 9 months posttreatment (with the exception of 39.4% at 6 months; see Comment #4), and was 7.7% (0.0077 ppm) of the applied at 12 months posttreatment. The minor degradate IN-B5067 was initially (time 0) detected at 2.9% (0.0029 ppm) of the applied radioactivity, increased to a maximum of 8.9% (0.0089 ppm) of the applied by 3 days posttreatment, and generally decreased to 3.6% of the applied by 12 months

posttreatment. The minor degradate IN-F5438 was initially (day 7) detected at 3.6% (0.0036 ppm) of the applied radioactivity, increased to a maximum of 8.2% (0.0082 ppm) of the applied by 1 month posttreatment, and was last detected at 1.2% (0.0012 ppm) of the applied at 4 months posttreatment. One unidentified minor degradate (designated as T12) was ≤9.8% of the applied radioactivity and eight unidentified minor degradates were each detected at ≤4.5% of the applied radioactivity throughout the incubation period. Nonextractable [^{14}C]residues were initially (time 0) 0.9% of the applied radioactivity, increased to 14.1% of the applied radioactivity by 10 days posttreatment, were 17.6-27.5% of the applied from 21 days to 6 months posttreatment, increased to a maximum of 34.8% of the applied by 7 months posttreatment, and were 31.6% of the applied at 12 months posttreatment (Table 3, p. 33); [^{14}C]residues associated with the humic acid, fulvic acid and humin fractions were 12.5%, 0.43%, and 12.8% of the applied radioactivity at 4 months posttreatment, respectively (p. 29). Evolved $^{14}CO_2$ initially (day 3) accounted for 0.3% of the applied radioactivity, was 10.3-18.7% of the applied from 2 to 9 months posttreatment, and was 22.8% of the applied by 12 months posttreatment; [^{14}C]organic volatiles were negligible.

In the sterile control samples, the parent compound was initially present at 101.6% (0.10 ppm) of the applied radioactivity, decreased to 41.3% (0.041 ppm) by 1 month and 18.8% (0.019 ppm) by 2 months, and was 2.0% (0.0020 ppm) of the applied at 12 months posttreatment (Table 9, p. 40; see Comment #5). The major degradate

IN-B5067

was initially (time 0) detected at 2.5% (0.0025 ppm) of the applied radioactivity, was 12.0-18.9% (0.012-0.019 ppm) of the applied from 1 to 6 months posttreatment, increased to a maximum of 31.3% (0.031 ppm) of the applied by 9 months posttreatment, and was 25.8% (0.026 ppm) of the applied at 12 months posttreatment. The major degradate

IN-A4098

was initially (1 month) detected at 20.8% (0.021 ppm) of the applied radioactivity, increased to a maximum of 26.2% (0.026 ppm) of the applied by 5 months posttreatment, and was 18.1% (0.018 ppm) of the applied at 12 months posttreatment (see Comment #4). The major degradate

acetyl triuret (IN-MU717; chemical name not provided)

was initially (1 month) detected at 10.1% (0.010 ppm) of the applied radioactivity, increased to a maximum of 22.2% (0.022 ppm) of the applied by 4 months posttreatment, and was last detected at 14.9% (0.015 ppm) of the applied at 6 months posttreatment. The major degradate

was initially (time 0) detected at 1.7% (0.0017 ppm) of the applied radioactivity, increased to a maximum of 17.8% (0.018 ppm) of the applied by 6 months posttreatment, and was 10.1% (0.010 ppm) of the applied at 12 months posttreatment (see Comment #4). Nonextractable [^{14}C]residues were initially (time 0) 0.4% of the applied radioactivity, were 13.5-17.9% of the applied at 2-5 months posttreatment, and were a maximum of 27.3% of the applied by 12 months posttreatment (Table 5, p. 35). Evolved $^{14}CO_2$ accounted for 0.1-0.6% of the applied radioactivity at 2-9 months posttreatment and was 1.8% of the applied at 12 months posttreatment; [^{14}C]organic volatiles were negligible.

Material balances (based on LSC analysis) for the viable samples were 93.2-103.0% of the applied radioactivity (Table 3, p. 33), with no observed pattern of decline. Material balances (based on LSC analysis) for the sterile control samples were 90.7-117.9% (Table 5, p. 35), with no observed pattern of decline.

COMMENTS

1. Duplicate samples were not utilized in this study. The use of single samples is generally not considered to be a sound scientific practice; at a minimum, duplicate samples should be utilized for each sampling interval (for each label and treatment).

2. The study authors stated that the temperature was held constant at approximately 20°C throughout the incubation with the exception of one 12-hour and one 3-hour period (p. 16). The temperature during each of these time periods did not exceed 28°C.

3. Residue data for the parent and degradates were reported only as percentages of the nominal application rate; concentration data were not reported. Concentration data for the parent and degradates (in ppm based on parent equivalents) were reviewer-calculated from the nominal application rate and the reported percentages fo the applied radioactivity. In future studies submitted to the EPA, it is necessary that residue data for the parent and degradates be reported as both percentages of the applied radioactivity and in units of concentration, such as ppm.

4. The data reported for the 6-month samples (triazine label only) and the 4-month sterile control samples (triazine label only) were outliers for the degradates IN-B5528 and IN-A4098 (Tables 7, 9, pp. 37, 40). The study authors stated that IN-A4098 can convert to IN-B5528 if the presence of an organic solvent (Footnote of Tables 7, 9, pp. 37, 40).

5. The study authors stated that the sterile control samples did not remain sterile after 6 months of incubation (p. 22).

6. The study authors stated that the nominal treatment rate (0.1 ppm) was equivalent to the seasonal application rate of 210 g a.i./ha. (p. 15).

7. Method detection limits were not reported. Both limits of detection and quantitation should be reported to allow the reviewer to evaluate the adequacy of the methods for determination of parent and degradates in the test system.

8. Full chemical names were not reported for the degradates IN-MU717 and IN-NC148. The trivial names of these compounds were reported as acetyl triuret and carbamoyl guanidine, respectively, and their structures were provided in Figure 4 (p. 50). In future studies submitted to the EPA, it is necessary that the chemical names and structures for the parent and degradates be reported.

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