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Dissipation of Foliar Dislodgeable Residues of Bendiocarb Following Application to Azaleas

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Bendiocarb, the active ingredient in TURCAM® (Nor-Am Chem. Co., Pikeville, NC), is registered for and is used for a variety of turf and ornamental pests. United States EPA regulations (40 CFR 158.140) require data on residue dissipation for each manufacturing use product likely to cause dislodgeable residues on crop surfaces and for which a safe reentry interval is required to be established. The bendiocarb registration standard calls for a foliar dislodgeable residue dissipation study to be conducted and notes that soil dissipation studies are not required. Ornamentals were chosen for this study because application rates are higher on a per acre basis than those for turf. Glasshouse ornamentals are usually grown in pots or flats on tables, thus affording more potential contact with the mid- and upper-body areas of workers, and glasshouse pesticide residues are not subject to open field weathering.

The objective of this study was to obtain sufficient data to describe the dissipation of dislodgeable residues of bendiocarb when applied to glasshouse ornamentals as TURCAM® at the highest recommended rate. These data would then be used along with the calculated safe reentry residue level for bendiocarb to determine the safe reentry time for glasshouse workers.

MATERIALS AND METHODS

This study was conducted in a glasshouse at the University of Florida Citrus Research and Education Center, Lake Alfred, Florida. The glasshouse is cooled by Arctic evaporative coolers and is heated by gas. Temperatures inside the glasshouse during this study ranged from 50°F (night) to 100°F (day). Relative humidity ranged from 45 to 100%. Thirty azalea var. Formosa plants in pots were purchased from a community nursery and placed in the glasshouse. The plants were divided into five sets of six plants each, two sets designated as controls and three sets designated as treatments. The control sets were placed in the southeast corner of the glasshouse and the treatment sets were placed 40 feet away in the southwest corner. Treatment was made

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by spraying the upper and lower sides of the leaves to runoff using a Loyalty 2001 handheld pump sprayer (Al Corp., Lowell, MI). Application was made one time at a rate of 5.9 lb ai/A. The recommended maximum application rate for azaleas is 2 lb 10 oz TURCAM® (2 lb ai) per 100 gal of water, sprayed to runoff. This equates to an application rate of 7.875 lb TURCAM® per acre, as 300 gal of water would be required. The treatment solution was prepared by mixing 11.9 g of TURCAM® per gal of double deionized water. Therefore, 2 lb 10 oz x 454 g/lb = 1191.75 g; 1191.75 g ÷ 100 gal = 11.9 g/gal. During the application, the cooler fans in the glasshouse were turned off to reduce the possibility of contamination of the control sets.

TURCAM®, lot number 8F35, with a minimum nominal bendiocarb (2,2-dimethyl-1,3-benzodioxol-4-yl methylcarbamate (IUPAC) (2,2-isopropylidenedioxyphenyl methylcarbamate (CA)) content of 76%, was used in this study. Quality control analysis of this lot number found the actual bendiocarb content to be 79.1%. NOR-AM analytical reference standard number 132-01, 99.4% purity was used to prepare both fortification standards and standards for HPLC analysis.

All sets were sampled just prior to treatment (-0 day); after the treatment spray had dried (+0 day); and 1, 2, 5, 7, 14, 21, 28 and 35 days after treatment. At all times, "control" sets were sampled first. Twenty leaves from each set were clipped with scissors into 8 oz glass jars (one per set) in a random fashion from the front and back half of each plant. The samples were then taken directly to the laboratory where the analytical process was begun immediately.

In the laboratory, the samples were immediately washed by shaking three times with 100 mL of SUR-TEN™ solution (1:12,500 dilution of a 70% solution of sodium dioctyl sulfosuccinate, SUR-TEN™ (ICN Pharmaceuticals, Plainview, NY) at 350 rpm for one minute each time (New Brunswick Model R-2 rotary shaker, New Brunswick Sci., Edison, NJ). Washes were combined in a 500 mL separatory funnel and 2 mL of 0.2M HCl was added. The combined washes were extracted with two 100 mL portions of methylene chloride and the extracts combined in a 500 mL round bottom flask. The solvent volume reduced to a few mL by rotary vacuum evaporation at 40°C. The concentrated extract was then quantitatively transferred into a 20 mL scintillation vial using methylene chloride to rinse the round bottom flask. The extract in the scintillation vial was evaporated just to dryness under a stream of nitrogen at ambient temperature. Treated samples were dissolved in 5 mL of methanol for HPLC analysis while control samples were dissolved in 1 mL. When necessary, treated samples were further diluted in methanol to obtain quantifiable peaks. To each final solution, 100 µL of 30 ng/µL β-naphthol were added per 10 mL final solution to act as a HPLC internal standard. HPLC analyses were performed on a Waters HPLC equipped with a model 721 controller, WISP autosampler and 250 mm x 4.6 mm column packed with Adsorbosphere C^{18}, 60Å, 20% C load. Mobile phase was 50% acetonitrile/50%

water, containing 1 mL acetic acid per liter, at 1.3 mL per minute. Detection was at 213 nm. Under these parameters, the retention time for bendiocarb varied during the study from 5.9 minutes to 7 minutes. A five point standard curve was constructed each analysis day using peak areas for quantification. Bendiocarb was successfully detected down to a level of 500 µg/300 mL of wash solution, the lowest fortification level in this study. Mean total leaf area for the samples taken in this study was 270.8 cm^2 (s.d. = 27.7 cm^2). Dividing this figure into 500 µg demonstrates a limit of determination of 1.8 $µg/cm^2$. Because samples were analyzed immediately after they were obtained, no storage stability study was performed.

Duplicate SUR-TEN^{TM} solutions were fortified with known amounts of bendiocarb standard and then taken through the analytical procedure as described below with each set of samples.

Leaf surface areas were measured with a LiCor Leaf Area meter (Lambda Instrument Corp., Lincoln, NB) and multiplied by two to obtain total leaf surface area.

The plants were kept well-watered throughout the study by adding water to the soil surface, avoiding any leaf wetting. Each plant received 26 liters total. They were not fertilized during the study.

The safe reentry level for bendiocarb was calculated as 13.0 $µg/cm^2$ by the method of Thougsinthusak and Krieger (1989).

RESULTS AND DISCUSSION

Mean recovery for 18 fortifications was 89%, with a standard deviation of 14% (Table 1).

Table 1. Recovery efficiencies during the experiment.

Time (days)	Fortification (µg bendiocarb)	Bendiocarb found (µg)	% Recovery
+0	3 mg	2.94, 3.64	98, 121
1	3 mg	2.66, 2.63	89, 88
2	3 mg	2.56, 2.43	85, 81
5	2 mg	1.82, 2.14	91, 107
7	2 mg	1.86, 1.82	93, 91
14	1 mg	0.87, 0.74	87, 74
21	1 mg	0.68, 0.57	68, 57
28	1 mg	0.81, 0.92	81, 90
35	0.5 mg	0.48, 0.52	96, 104

Number = 18; Mean ± SD = 89 ± 14%

Results of analyses of treated leaf samples are summarized in Table 2. Results are corrected for mean recovery from fortified wash solutions but are not corrected for apparent residues in control samples (Table 3).

Table 2. Bendiocarb disappearance from glasshouse azaleas.

Sampling interval (days)	µg Bendiocarb per cm^2 mean ± SD n = 3
-0	--
+0	10.9 ± 0.6
1	11.5 ± 0.6
2	12.3 ± 1.7
5	8.6 ± 0.3
7	9.0 ± 1.7
14	8.4 ± 1.1
21	3.9 ± 1.0
28	2.7 ± 1.4
35	4.5 ± 0.7

Apparent dislodgeable residues of bendiocarb in control samples ranged from non-detectable (i.e., no peak seen at the retention time of bendiocarb) to 0.027 µg/cm^2 (Table 3). We speculate that residues appearing after day 0 may have been redeposited by the glasshouse coolers. Based on the amount of residue found on treatment plants, control residues do not indicate any effect of air movement on these results.

Table 3. Apparent dislodgeable bendiocarb residues in control samples.

Time (days)	Total leaf area (cm^2)	Bendiocarb found (µg)	µg bendiocarb per cm^2
-0	290.6, 233.8	N.D., N.D.	N.D., N.D.
+0	248.7, 352.4	N.D., N.D.	N.D., N.D.
1	256.3, 294.3	1.58, 2.52	0.006, 0.009
2	277.4, 237.6	1.17, 2.35	0.004, 0.010
5	230.9, 264.9	6.13, 3.25	0.027, 0.012
7	262.1, 289.3	1.85, 2.56	0.007, 0.009
14	302.0, 272.9	1.67, N.D.	0.006, --
21	230.0, 274.9	N.D., N.D.	--
28	262.0, 240.9	3.04, 2.50	0.012, 0.010
35	273.2, 297.7	3.66, 2.14	0.013, 0.007

N.D. = no peak detected at t_R of bendiocarb

Application of TURCAM® to azalea plants at a rate of 7.9 lb TURCAM® (6 lb ai)/A in 300 gal of water per acre resulted in dislodgeable bendiocarb residues on the leaves ranging from 10.9 μg/cm^2 at day 0 to 4.5 μg/cm^2 at day 35. The half-life of the bendiocarb on azalea leaves was 18.3 days (1st order, R = 0.914). The day 0 dislodgeable residue level is less than the calculated safe reentry level of 13.0 μg/cm^2. The highest level found was 12.3 μg/cm^2 on day 2.

Conducting this study in a glasshouse and watering the plants only at the base is probably a worst-case situation in that any dislodgeable residues present on the leaves were not subjected to the weathering effects of open field wind, irrigation, and rain, or to normal watering/irrigation in a glasshouse.

Bendiocarb dislodgeable residues resulting from application of TURCAM® at maximum label rates appear to present minimal reentry hazard to workers. Under present standards, workers would be able to enter a treated area after the spray had dried or the dust had settled.

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