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CASE GS -- SYSTHANE STUDY 1 PM --

CHEM 128857 Systhane

BRANCH EAB DISC --

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CONCLUSIONS:Degradation - Photodegradation in Water

The portion of this study investigating the photodegradation of [¹⁴C]-systhane in sterile, deionized water could not be validated because the concentration of systhane was reported as percent of recovered instead of percent of applied (TLC data) and the appropriate raw data were not provided to determine the recoveries. In addition, this portion of the study would not fulfill EPA Data Requirements for Registering Pesticides because the study was conducted for an insufficient length of time (15 days), the test solutions were not buffered, the concentration of the cosolvent was not reported, the incubation temperature was not 25 ± 1°C, and the temperature of the dark control was not reported.

The portion of this study investigating the photodegradation of [¹⁴C]-systhane in the presence of a photosensitizer is scientifically invalid because of the lack of dark controls. In addition, this portion of the study would not fulfill EPA Data Requirements for Registering Pesticides because the test solutions were not buffered, the concentration

of the cosolvent was not reported, the incubation temperature was not $25 \pm 1^\circ\text{C}$, degradate characterization data (TLC data) were presented as percent of recovered rather than percent of applied, and certain quantitative data were missing.

MATERIALS AND METHODS:

Triazole or chlorophenyl ring-labeled [^{14}C]systhane (RH-3866; radiochemical purities $>96\%$; specific activities 10.98 and 10.28 mCi/g, respectively; Rohm and Haas Co.) were added at 9.5-10.0 ppm to filter-sterilized ($0.22 \mu\text{m}$) deionized water and filter-sterilized deionized water containing 2% acetone (photosensitizer). Solutions to be irradiated were incubated at 31°C in a presiliconized (with a 10% solution of Surfasil) Pyrex glass photoreactor (Figure 1) equipped with a 20-W sunlamp (FS20, Westinghouse) emitting light of 2.8 W/m^2 intensity (at the outside surface of the photoreactor) between 270 and 440 nm. The relative intensity and the spectral distribution of the artificial light source as compared to natural sunlight are presented in Figure 2. Dehumidified, CO_2 -free air was passed (10 mL/minute) through the treated solutions, then through a volatiles trap containing Chromosorb 101, and finally through methyl cellosolve:ethanolamine (70:30) to trap CO_2 . As a control, filter-sterilized deionized water was treated with chlorophenyl ring-labeled [^{14}C]systhane at 9.9 ppm and maintained in darkness. Irradiated and dark control solutions, volatiles traps, and CO_2 trapping solutions were sampled periodically up to 360 hours posttreatment.

The Chromosorb was extracted with methanol, and an aliquot of the extract was analyzed for radioactivity by LSC. Radioactivity in the CO_2 trapping solutions was quantified by LSC. The $^{14}\text{CO}_2:\text{CO}_2$ ratio was determined in selected samples. An aliquot of the CO_2 trapping solution was diluted with 1 N hydrochloric acid, then dry nitrogen was passed (60 mL/minute) through the solution and into a 1 N solution of sodium hydroxide. Total CO_2 in the sodium hydroxide solution was determined by barium chloride precipitation.

Total radioactivity in the water samples was quantified by LSC. The sample was applied to a Sep-Pak C-18 cartridge, and the water passing through the cartridge was frozen and lyophilized; the residue was dissolved in methanol. The C-18 cartridge was then eluted with methanol, the methanol eluate was evaporated to dryness, and the resulting residue was redissolved in methanol. Residues from the water and methanol eluates were analyzed for systhane and its degradates by TLC on silica gel plates developed in either ethyl acetate:isopropanol:water (65:25:10) or butanol:water:acetic acid (65:25:10). Labeled reference compounds were cochromatographed with the samples. Following development, radioactive areas were located by autoradiography. Degradates were identified by comparison to the reference compounds and quantified by scraping the silica gel from the plates and analyzing by LSC.

For further degradate analysis, triazole ring-labeled [^{14}C]systhane was added at ~ 13 ppm to filter-sterilized deionized water containing 2% acetone and irradiated for 27 hours as previously described. The entire sample was applied to C-18 cartridges and eluted as previously described, and the residue was analyzed by radio-HPLC and MS.

REPORTED RESULTS:

At 15 days (360 hours) posttreatment in the sterile, deionized water, <1% of the applied radioactivity had been evolved as $^{14}\text{CO}_2$ and 96-98% of the applied was recovered in the methanol eluate following C-18 column separation of the treated solution (Table 1). Parent systhane accounted for 91-93% of the recovered radioactivity after TLC of the methanol eluates (Table 2). After 15 days in the dark control (chlorophenyl ring-labeled [^{14}C]systhane), 100% of the applied radioactivity was recovered in the methanol eluate, and parent systhane comprised 98% of the radioactivity recovered from the TLC plate.

After 15 days in the presence of a photosensitizer, 45 and 7% of the applied radioactivity (chlorophenyl and triazole ring-labeled [^{14}C]systhane, respectively) was evolved as $^{14}\text{CO}_2$ (Table 3). Parent chlorophenyl ring-labeled [^{14}C]systhane and dechlorinated systhane (RH-5781F) accounted for 10 and 9% of the recovered radioactivity after TLC of the methanol eluate, respectively (Table 4). At 48 hours posttreatment, parent triazole ring-labeled [^{14}C]systhane accounted for 20% of the recovered after TLC of the methanol eluate.

DISCUSSION:

General

1. Results from TLC analyses of methanol eluates were presented as percent of recovered rather than percent of applied, and recoveries of radioactivity applied to the TLC plates were not reported (to demonstrate that recovery was quantitative).
2. The incubation temperature was not $25 \pm 1^\circ\text{C}$.
3. The concentration of the cosolvent (acetonitrile) was not specified.
4. The test solutions were not buffered.
5. The registrant implied, but did not specify, that radioactivity applied to the C-18 column was quantitatively recovered.

Sterile, Deionized Water

1. There were no dark controls for triazole ring-labeled [^{14}C]systhane.
2. The study was not conducted for 30 days or one half-life, and it appears that degradation was beginning to occur when the study was terminated at 15 days.
3. The incubation temperature for the dark control was not reported.

Sterile, Deionized Water with Photosensitizer

1. There were no dark controls.

2. TLC characterization of triazole ring-labeled [^{14}C]systhane degradates (Table 7 in text) was missing.
3. Quantitative data from TLC characterization of water eluates were not provided.

Natural Pond Water

Data concerning the degradation of chlorophenyl and triazole ring-labeled [^{14}C]systhane in natural water were not reviewed because the solutions were not sterile and there were no dark controls. Therefore, the data are invalid and could not be used to fulfill data requirements.