

US EPA ARCHIVE DOCUMENT



## MATERIALS AND METHODS:

A slurry of air-dried, sieved (1 mm) Keyport silty clay loam soil (5% sand, 67% silt, 28% clay, 1.4% organic matter, pH 6.8, CEC 6.25 meq/100 g) and distilled water was spread over microscope slides and air-dried to form a soil layer approximately 1 mm thick with a surface area of 16.5 cm<sup>2</sup> per slide. A solution of carbonyl-labeled [<sup>14</sup>C]fosamine ammonium (radiochemical purity >98%, specific activity 6.8 uCi/mg, du Pont) and unlabeled fosamine ammonium (purity not specified), dissolved in pH 7.4 phosphate buffer and absolute ethanol, was applied to each slide at 0.269 ug/cm<sup>2</sup> (equivalent to 24 lb ai/A). The slides were dried, then placed on individual stainless steel heat exchangers (5 x 5 inches) contained inside watertight Lucite boxes (7 x 7 x 2 inches) (Figure 2). The samples were maintained at 24-28 C by pumping water through the system (rate of 1 L/minute) using a refrigerated circulating water bath. In order to trap volatiles, air (10 mL/minute) was drawn over the slides in each box and then through a gas-washing bottle containing 1 N sodium hydroxide. The soil photolysis boxes were covered with quartz windows and irradiated with sunlight outdoors on a roof in Wilmington, Delaware (39° 40' N latitude; 75° 36' W longitude) from August 13 to September 15, 1986. The cumulative spectral energy distribution was 142,329 watt-hrs/m<sup>2</sup> (Table III). The lids of similar boxes containing soil samples were covered with aluminum foil, and the boxes were placed outside along with the irradiated samples to serve as dark controls. Irradiated and dark control soil samples, and sodium hydroxide trapping solutions were removed for analysis at 1, 3, 5, 7, 9, 20, and 30 days posttreatment.

Soil was scraped from each slide and then extracted three times by sonication at 45-50 C for 30 minutes with methanol:0.5 M aqueous ammonium carbonate (1:1); the scraped slides were rinsed with the same solution. Aliquots of each soil extract and rinses from the slides were analyzed for total radioactivity by LSC. The combined soil extracts were concentrated by rotary evaporation and analyzed by TLC on silica gel plates developed in methanol:0.5 M aqueous ammonium carbonate (4:1). The samples were cochromatographed with reference standards of radiolabeled fosamine ammonium and carbamoylphosphonic acid (CPA). Radioactive areas were located and quantified with a radiochromatogram scanner, and identified by comparison to the reference standards. Rinses from the slides containing >10% of the applied radioactivity were also analyzed by TLC as described. The extracted soils were analyzed for unextractable [<sup>14</sup>C]residues by LSC following combustion.

In an attempt to characterize an unidentified polar degradate, the remainder of the concentrated extracts from the day 30 irradiated sample and the day 7 dark control sample, and the [<sup>14</sup>C]carbamoyl phosphonic acid standard, were streaked onto silica gel TLC plates that were subsequently developed as previously described. Radioactive areas were located and quantified with a radiochromatogram scanner. The region of the plate containing the unidentified degradate was scraped and the silica gel was extracted three times by sonication at 50 C for one hour with ethanol:0.1 M aqueous ammonium carbonate (1:1). The combined extracts were concentrated using rotary evaporation followed by a stream of nitrogen. The concen-

trated residues were dried in a vacuum oven maintained at room temperature for 7 days, and then frozen and lyophilized to remove remaining ammonium carbonate. The residues were analyzed by MS.

Aliquots (100 uL) of the sodium hydroxide trapping solutions were analyzed for total [ $^{14}\text{C}$ ]residues by LSC. In order to confirm that radioactivity in the sodium hydroxide trapping solutions was  $^{14}\text{C}$   $\text{CO}_2$ , the solutions were treated with saturated barium chloride solution and 2 M potassium hydroxide, and then were analyzed for radioactivity which had not precipitated as barium carbonate.

#### REPORTED RESULTS:

Carbonyl-labeled [ $^{14}\text{C}$ ]fosamine ammonium (radiochemical purity >98%), at 0.269 ug/cm<sup>2</sup> (equivalent to 24 lb/A), degraded with a registrant-calculated half-life of 82 days in Keyport silty clay loam soil irradiated with natural sunlight at 24-28 C in Wilmington, Delaware (cumulative solar energy 142,329 watt-hours/m<sup>2</sup>), during August-September 1986. In the dark controls, the calculated half-life was 77 days. At 30 days posttreatment, fosamine ammonium comprised 73.3 and 72.7% of the applied radioactivity in the irradiated and dark control samples, respectively (Table III). In the irradiated and dark control samples, the degradate carbamoyl phosphonic acid (CPA) comprised 18.9%-24.2% of the applied, unidentified polar compounds comprised 2.3-4.3%,  $^{14}\text{C}$   $\text{CO}_2$  totaled  $\leq 1\%$ , and unextractable [ $^{14}\text{C}$ ]residues were 1.4% of the applied at 30 days posttreatment (Table III). The material balances for the irradiated and dark control samples ranged from 94.8 to 119.5% of the applied during the study (Table II).

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