Bull. Environm. Contam. Toxicol. 25, 508-511 (1980)

Comparison of the Persistence of Ethalfluralin and Trifluralin in Saskatchewan Field Soils

Brian J. Hayden and Allan E. Smith
Agriculture Canada, Research Station, Box 440, Regina, Saskatchewan, Canada S4P 3A2

Ethalfluralin (N-ethyl-N-(2-methyl-2-propenyl)-2,6-dinitro-4-(trifluoromethyl)benzenamine) is a soil applied pre-emergence herbicide used at rates of 0.5 to 1.0 kg/ha for the control of annual grasses and other weeds in a variety of crops. The chemical is volatile and must be incorporated into the soil after application to prevent evaporative losses.

Persistence studies with ethalfluralin have not been reported and the work to be described was undertaken to determine the persistence of ethalfluralin under field conditions at two Saskatchewan locations using a small plot technique (SMITH 1971, 1972, SMITH and HAYDEN 1976). Since ethalfluralin is structurally very similar to the commonly used soil incorporated dinitroaniline herbicide trifluralin (α,α,α-trifluoro-2,6-dinitro-N,N-dipropyl-p-toluidine), the persistence of the latter compound was similarly studied for comparative purposes. Following soil extraction, ethalfluralin and trifluralin residues were determined by gas chromatographic analysis.

MATERIALS AND METHODS

Soils. The physical characteristics and composition of the heavy clay (HvC) and sandy loam (SL) field soils used in this study have already been reported (SMITH 1978a).

Chemicals. Commerical formulations of emulsifiable concentrates of ethalfluralin (0.3 kg/L) and trifluralin (0.4 kg/L) were obtained from Eli Lilly & Company (Canada) Limited, Scarborough, Ontario and diluted with methanol to give individual solutions with herbicide contents of 2 mg/mL.

Field persistence studies. Separate field plots (20 x 20 cm, 400 cm^2) at Regina (heavy clay) and White City (sandy loam), Saskatchewan were treated with the methanolic solutions of ethalfluralin or trifluralin (2 mL, 4 mg herbicide) and incorporated to a depth of 5 cm, using a small fork, exactly as described (SMITH 1972). This treatment was equivalent to an application rate of 1 kg/ha. Six replicate plots were treated at each site with each chemical, on May 4, 1977; May 4, 1978; and May 31, 1979. Duplicate plots were sampled 10, 20 and approximately 52 weeks

0007-4861/80/0025-0508 $01.00

following application, by removing the soil from the 0 to 5 cm and 5 to 10 cm levels of the treated plots as described (SMITH 1971, 1972, SMITH and HAYDEN 1976). After air-drying in the laboratory at room temperature, the samples were weighed (approximately 2 kg), ground, and mixed in a soil mixer for 20 minutes. Sub-samples of 20-g were then solvent extracted and analysed gas chromatographically for ethalfluralin or trifluralin content.

Herbicide extraction and analysis. Soil samples (20 g) were placed in a 150-mL capacity glass-stoppered flask and shaken for 1 hour on a wrist-action shaker with 50 mL 30% aqueous acetonitrile containing 3% glacial acetic acid. Following centrifugation at 3000 rpm for 4 minutes, a 25-mL portion of the supernatant (equivalent to 10 g soil) was added to 100 mL 5% aqueous sodium carbonate solution in a 250-mL separatory funnel and shaken for 1 minute with 50 mL n-hexane. The hexane layer was run into a glass-stoppered flask, dried over anhydrous sodium chloride and 3 μL aliquots examined gas chromatographically.

The Hewlett-Packard 5713A gas chromatograph used for the quantitative analyses was equipped with a radioactive nickel detector operated at 300°C. The glass column (1.5 m x 4 mm i.d.) was packed with 5% Dexsil-300, on Chromosorb W, HP (80-100 mesh). Carrier gas was argon containing 5% methane, at a flow rate of 40 mL/min. All samples were injected directly onto the column packing. With a column temperature of 200°C, the retention times for ethalfluralin and trifluralin were 2.7 and 3.0 minutes respectively.

Chromatographic standards of both ethalfluralin and trifluralin were prepared in n-hexane and the concentration of the herbicides recovered from the soil samples were calculated by comparing the sample peak heights with those of the appropriate standards.

To check recoveries of the herbicides from treated soils, 20-g samples of untreated air-dried soils (4 replicates for each soil type) were fortified at the 0.5 and 0.05 μg/g levels with ethalfluralin and trifluralin. After thorough mixing, the treated soils were equilibrated in the dark at laboratory temperature for 72 hours. Following extraction and analysis as described above, recoveries of both herbicides from both soils were in excess of 95%. Similar analysis of untreated soils confirmed the absence of interfering substances.

RESULTS AND DISCUSSION

Aqueous acetonitrile containing a small volume of glacial acetic acid was selected as extraction solvent for ethalfluralin and trifluralin, since this solvent has been used to recover the latter herbicide from Saskatchewan soils (SMITH 1979). This solvent system has also proved satisfactory for the extraction of a variety of herbicides, including a dinitroaniline, from weathered field soils (SMITH 1978b). As mentioned, recoveries of both dinitroanilines from fortified soils were in excess of 95%. This recovery for ethalfluralin is greater than that of 85% reported by DAY (1978) who used 1% aqueous acetonitrile for its extraction from soils.

For each set of duplicate soil plots sampled at the various dates, there was less than ±5% variation in the amounts of ethalfluralin or trifluralin recovered. At all sampling dates and at both sites, all the residues were located in the soil from the top 5 cm of the treated plots and less than 2% of the applied ethalfluralin or trifluralin was detected in the 5 to 10 cm soil levels. This confirmed that neither herbicide was leached under field conditions.

The results of the persistence studies are summarized in Table 1. Initial losses of both chemicals were rapid from the treated plots at both locations (Table 1), with over 64% of the ethalfluralin and trifluralin being lost within 10 weeks. Dissipation during the 10 to 20 week period following application was slower and accounted for less than 15% of the initial treatments. Overwinter losses from the plots tended to be minimal and amounts observed in the May of the year following treatment were similar to those noted the previous October (Table 1). A similar finding for losses of trifluralin from Saskatchewan soils has already been reported (SMITH and HAYDEN 1976).

TABLE 1

Ethalfluralin and trifluralin recovered from the top 5 cm of field plots at two locations after various time intervals, following applications of 1 kg/ha during May of 1977, 1978 and 1979.

Date applied	Weeks after application	% Remaining *			
		Ethalfluralin		Trifluralin	
		HvC	SL	HvC	SL
May 4, 1977	10	15	19	16	20
	20	14	4	9	7
	52	5	3	6	7
May 4, 1978	10	17	20	27	34
	20	9	5	16	19
	55	6	6	10	13
May 31, 1979	10	30	29	36	30
	20	21	17	29	25
	48	17	16	20	15

* Average of 2 samples, less than 2% of applied chemicals in the 5 to 10 cm soil levels.

Dissipation rates for both ethalfluralin and trifluralin from the heavy clay and sandy loam plots (Table 1) appeared to be slower in 1979 than during the other years, and this was attributed to the later application date and to the fact that 1979 was considerably drier than either 1977 and 1978. At the Regina site where the rainfall could be monitored, precipitation recorded for the period following treatment until freeze-up for 1977, 1978 and 1979 was 25.4 cm, 27.8 cm, and 10.8 cm respectively. Since volatility losses are perhaps the most important means of dissipation of dinitroaniline herbicides from treated soils and since such losses are greater from moist than dry soils (cf HELLING 1976) a greater persistence would be expected during 1979.

In general, the carry-over of ethalfluralin residues to the next crop year was very similar to that of trifluralin for any particular year and site.

REFERENCES

DAY, E.W.: Analytical Methods for Pesticides and Plant Growth Regulators, Vol. X. G. Zweig and J. Sherma eds. pp. 341-352. New York: Academic Press 1978.
HELLING, C.S: J. Environ. Qual. 5, 1 (1976).
SMITH, A.E.: Weed Sci. 19, 536 (1971).
SMITH, A.E.: J. Agric. Fd. Chem. 20, 829 (1972).
SMITH, A.E.: Weed Res. 18, 275 (1978a).
SMITH, A.E.: Pestic. Sci. 9, 7 (1978b).
SMITH, A.E.: Weed Res. 19, 165 (1979).
SMITH, A.E. and B.J. HAYDEN: Can. J. Plant Sci. 56, 769 (1976).