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Comparison of the Persistence of EPTC, Metribuzin, and Propanil in Saskatchewan Field Soils

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EPTC (S-ethyl dipropylthiocarbamate), metribuzin (4-amino-6-tert-butyl-3-(methylthio)-as-triazin-5(4H)-one) and propanil (3',4'-dichloropropionanilide) are used for weed control in western Canada. Although metribuzin and propanil are applied as post-emergence treatments, residues following application invariably come into contact with soil making their soil persistence a matter of concern. EPTC is a soil incorporated herbicide. The field persistence of these herbicides has not been investigated in Saskatchewan soils, though the loss of metribuzin from other Canadian field soils has been reported (WEBSTER and REIMER 1976, MARRIAGE et al. 1978).

The work to be described was undertaken to compare the field persistence of EPTC, metribuzin and propanil at three locations in Saskatchewan, using the small plot technique that the authors have used for similar studies with other herbicides (SMITH and HAYDEN 1976, HAYDEN and SMITH 1980).

MATERIALS AND METHODS

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

Persistence studies. Commercial formulations of the various herbicides were diluted with methanol, and individual solutions prepared containing EPTC (8 mg/mL), propanil (2 mg/mL) and metribuzin (2 mg/mL).

Three replicate field plots (20 X 20 cm, 400 cm^2) at Regina (heavy clay), Melfort (clay loam) and White City (sandy loam) in Saskatchewan were treated with the methanolic solutions (2 mL) of the various herbicides. Metribuzin and propanil were applied to the soil surface and were not incorporated, while EPTC was incorporated to a soil depth of 10 cm with a small fork. These application rates were equivalent to 1, 1 and 4 kg/ha for metribuzin, propanil and EPTC respectively. EPTC and propanil were applied during the second week of May in 1978, 1979 and 1980. Metribuzin was applied during the same time period in 1979, 1980 and 1981. All plots were left fallow and were hand weeded as necessary. Plots were sampled during the second week of October, 22 weeks

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following soil application, by removing the soil from the 0 to 5 cm and 5 to 10 cm (and in the case of EPTC the 10 to 15 cm) levels of the treated plots. Samples were air-dried in the laboratory at room temperature, weighed (approximately 2 kg), ground and mixed in a soil-mixer for 20 min. Subsamples were then solvent extracted and analyzed by gas chromatography. Full details for the setting up and sampling of these small treatment plots have been reported (SMITH 1971, 1972).

EPTC analysis. Soil samples (50 g) were shaken on a wrist-action shaker for 1 hour with 10% aqueous methanol (50 mL). The extracts were centrifuged at 3500 rpm for 5 min and supernatant (25 mL, equivalent to 25 g soil) was placed in a 100-mL separatory funnel with 15% aqueous sodium sulphate (25 mL) and n-hexane (10 mL). The mixture was shaken vigorously for 2 min, and after settling, the lower aqueous layer was discarded and the hexane phase collected in a glass-stoppered flask and dried over anhydrous sodium sulphate. EPTC in the hexane layer was analyzed directly using a Hewlett-Packard Model 5710A gas chromatograph, equipped with a nitrogen-phosphorus flame ionization detector operated in the nitrogen mode. The glass column (1.5 X 4 mm i.d.) was packed with 10% OV-1 on 80-100 mesh Chromosorb G, HP. Column carrier gas was helium at a flow rate of 30 mL/min. Flow rates of hydrogen and air through the detector were maintained at 3 and 50 mL/min, respectively. The detector voltage was operated at 14 volts. All samples were injected directly onto the column packing. With a column temperature of 170°C, the retention time for EPTC was 3.15 min. Chromatographic standards were prepared in hexane and the EPTC concentrations recovered from the various soil samples were calculated by comparing the sample peak heights with those from the appropriate standards. Recoveries of EPTC from all 3 soil types, fortified at the 0.1 µg/g level were in excess of 95%.

Metribuzin analysis. Soil samples (20 g) were shaken with 10% aqueous methanol (50 mL) on a wrist-action shaker for 1 hour. Following centrifugation of the soil extract at 3500 rpm for 5 min, supernatant (25 mL) was evaporated to dryness at 35°C using a rotary evaporator. The residue was dissolved in trimethylpentane (10 mL) and transferred to a glass-stoppered tube. Any metribuzin in the organic solvent was determined by direct injection into a Hewlett-Packard 5713A gas chromatograph equipped with a ^{63}Ni detector operated at 300°C. The glass column (1.5 mm X 4 mm i.d.) was packed with 100-120 mesh Ultra-Bond 20M. Carrier gas was argon containing 5% of methane at a flow rate of 40 mL/min. All samples were injected directly onto the chromatographic column. With a column temperature of 215°C, the metribuzin had a retention time of 3.35 min. All samples and standards were prepared in trimethylpentane, and the concentrations of the metribuzin recovered from the soils were calculated by comparing the sample peak heights with those of the standards. Recoveries of metribuzin from all 3 soils fortified at the 1.0 and 0.1 µg/g levels were greater than 90%.

Propanil analysis. The extraction and gas chromatographic

analysis of propanil were exactly the same as for metribuzin above, except that a column temperature of 200°C was used; propanil had a retention time of 2.50 min. Samples and standards were prepared in trimethylpentane. Recoveries of propanil from the different soils fortified at the 0.5 and 0.1 µg/g levels were greater than 90%.

RESULTS AND DISCUSSION

The results of the field persistence studies with propanil, EPTC and metribuzin are summarized in Table 1. At all locations, and for each of the three years, over 98% of the propanil was lost over the 22 week period following application (Table 1). No residues (less than 2% of those applied) were detected in any of the plots at the 5 to 10 cm soil depths.

Although EPTC was initially incorporated into the top 10 cm of the field plots, no detectable residues (less than 2% of those applied) were recovered after 22 weeks during any of the 3 years at the 3 locations sampled at the 5 to 10 and 10 to 15 cm soil depths. At Melfort, a residual trace of EPTC was noted (Table 1) in the top 5 cm of the clay loam plots 22 weeks after application in May of 1978 and 1980. Similarly a trace of EPTC was detected in the top 5 cm of heavy clay plots at Regina during 1979. In the sandy loam plots at White City, complete loss of EPTC occurred each of the 3 growing seasons.

These data indicate that EPTC is not particularly persistent in Saskatchewan soils and are in agreement with other field persistence studies (DANIELSON et al. 1961).

The persistence of metribuzin in Saskatchewan soils appeared to be variable (Table 1). No residues (less than 2% of those applied) were detected in any of the soils at the 0 to 5 cm and 5 to 10 cm soil depths 22 weeks after treatment in May of 1978. The following year, residues were recoverable from the top 5 cm of soils from plots sampled after 22 weeks at Melfort and Regina, but none were observed in the sandy loam plots at White City. No residues were detected in any of the soils taken from the 5 to 10 cm depths. During 1981, residues of metribuzin were recovered from the top 5 cm of soil plots at all 3 locations after 22 weeks, though persistence was less on the sandy loam plots than on the other 2 soil types. During 1981, metribuzin leached into the 5 to 10 cm soil depths at all 3 sites. Approximately 5% of the applied chemical was leached into the 5 to 10 cm soil profiles at both Melfort and White City; while at Regina about 15% of the applied metribuzin was recovered from the 5 to 10 cm soil depths.

The possibility of metribuzin leaching in the soil has been mentioned (WALKER 1978). The rainfall recorded at the Regina Research Station during the 22 week experimental periods was 174, 190 and 236 mm for each of the years 1979, 1980 and 1981. The 30 year average rainfall for this period is 255 mm. The metribuzin

TABLE 1

Residues of herbicides recovered from the 0 to 5 cm depth of soil at three locations

Herbicide	Rate kg/ha	Applied May of	% of herbicide remaining after 22 weeks*		
			Melfort Clay Loam	Regina Heavy Clay	White City Sandy Loam
EPTC	4.0	1978	7 ± 1	<2	<2
		1979	<2	5 ± 2	<2
		1980	8 ± 3	<2	<2
Metribuzin	1.0	1979	<2	<2	<2
		1980	15 ± 10	11 ± 5	<2
		1981	20 ± 1	18 ± 8	6 ± 1
Propanil	1.0	1978	<2	<2	<2
		1979	<2	<2	<2
		1980	<2	<2	<2

* Mean and standard deviation from analysis of 3 plots.

leaching at Regina during 1981 was therefore attributed to the increased rainfall that year.

Despite the seemingly variable persistence data observed for metribuzin (Table 1), the results are in general agreement with literature reports which indicate that between 5 and 20% of a spring application of the herbicide can be recovered from field top soils at the end of the growing season (HYZAK and ZIMDAHL 1974, WEBSTER and REIMER 1976, MARRIAGE et al. 1978, WALKER 1978).

The present studies indicate that the persistence of EPTC and propanil in Saskatchewan soils should not pose a problem, since carry-over to the following growing season should be minimal. However, residues of metribuzin may persist in the soil to the next growing season.

REFERENCES

DANIELSON, L.L., W.A. GENTNER and L.L. JANSEN: Weeds 9, 463 (1961).

HAYDEN, B.J. and A.E. SMITH: Can. J. Plant Sci. 60, 311 (1980).

HYZAK, D.L. and R.L. ZIMDAHL: Weed Sci. 22, 75 (1974).

MARRIAGE, P.B., A.S. HAMILL and F.G. VON STRYK: J. Environ. Sci. Health B13, 287 (1978).

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 (1978).

SMITH, A.E. and B.J. HAYDEN: Can. J. Plant Sci. 56, 769 (1976).

WALKER, A.: Weed Res. 18, 305 (1978).

WEBSTER, G.R.B. and G.J. REIMER: Weed Res. 16, 191 (1976).

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