

Persistence of Dinitramine and Trifluralin in Nova Scotia, Canada

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The two soil incorporated dinitroaniline herbicides, trifluralin (α,α,α -trifluoro-2,6-dinitro-N,N-dipropyl-p-toluidine) and dinitramine (N^4,N^4 -diethyl- α,α,α -trifluoro-3,5-dinitrotoluene-2,4-diamine) are used extensively for weed control in a wide range of field and vegetable crops. Although these are not generally acknowledged as persistent herbicides (PARKA and TEPE 1968), published accounts vary widely in their reported half-lives in soil with a possible trend toward significantly longer persistence in cooler, drier climates. For example, in selected studies the half-life of trifluralin in irrigated soils has been reported to be as short as 3 weeks in Texas (MENGENS and HUBBARD 1970) and about 5 weeks in Tennessee (DUSEJA and HOLMES 1978). In contrast, recommended rates of field applied trifluralin resulted in severe injury to susceptible indicator plants 4 and 5 months after application in Saskatchewan (RAHMAN and ASHFORD 1973). It has been shown that trifluralin persists approximately twice as long as dinitramine in the soil under comparable conditions (SAVAGE 1978, STROLLER and WAX 1977).

Trifluralin and dinitramine have been shown to be strongly adsorbed to the soil thereby limiting leaching (HARVEY 1974, SMITH *et al.* 1973). They are dissipated from the soil by such processes as volatilization, photolysis, and chemical and biological degradation (SMITH *et al.* 1973, SPENCER and CLIA TH 1974, KEARNEY *et al.* 1976, HARVEY 1974, PROBST *et al.* 1967). These processes can in turn be influenced by temperature, depth of herbicide incorporation, presence of plants, and such edaphic factors as temperature, moisture, and adsorptive capacity.

The following study was conducted to determine the persistence of trifluralin and dinitramine under the climatic and edaphic regime of Nova Scotia. Carry-over from one year to the next of phytotoxic residues resulting from herbicide treatments not normally considered "residual" (e.g. low rates of linuron and metribuzin) has commonly occurred suggesting an increased persistence of some herbicides in Nova Scotia compared with other agricultural areas of North America. In the present study, loss of herbicide from dinitroaniline treated soils was determined following two application dates:

(i) mid-May to assess residue levels at the end of the growing season when susceptible fall-sown cereals may be seeded into treated soil; and (ii) late-November to assess over-winter losses of dinitroaniline herbicides.

MATERIALS AND METHODS

Treatments and Sampling

Plots were established at two locations near Kentville, Nova Scotia, on loamy sand soils of the Berwick and Somerset series (Table 1). Herbicides were applied with a Cornell CO₂- pressurized plot sprayer in 550 l/ha water at 175 kPa and incorporated to approximately 10 cm immediately after application with a rototiller. Late-fall applications of trifluralin and dinitramine were applied at 1.0 and 0.75 kg/ha, respectively, on November 25, 1977. Spring applications of trifluralin at 0.75 kg/ha and dinitramine at 0.5 kg/ha were applied the following May 15. Following the spring application, the entire plot areas were tilled and planted to peas. Soil samples were taken to a depth of 10 cm from 15 random spots within each plot immediately after herbicide incorporation and periodically until November 23, 1978. Samples were stored frozen until analysis. Treatments were set out in quadruplicate in a randomized, complete block design.

TABLE 1

Properties of the soils at the two test sites

	% Sand	% Silt	% Clay	% O.M.	C.E.C. (Me/100g)	pH
Berwick loamy sand	71.2	10.8	18.0	4.2	12.7	5.2
Somerset loamy sand	85.0	8.8	6.0	1.3	5.0	5.3

Analysis: Soil samples were thoroughly mixed and 20 gm subsamples were extracted without drying by shaking for 1 h with 60 ml acetonitrile-water (9:1 v/v). Hereafter the extraction procedure of SMITH (1974) was followed. A Micro-Tek 220 gas chromatograph equipped with a Ni⁶³ electron-capture detector was used with a 75 x 6.4 mm glass column packed with 10% DC-200, 15% QF-1 on 80-100 mesh Gas Chrom Q. Argon/methane gas flow was 40 ml/min and column and inlet temperatures were 220° and

225°C, respectively. Under these conditions, retention times of trifluralin and dinitramine were 1.0 and 1.75 min, respectively. No clean-up of soil samples was necessary using this procedure and recoveries from spiked samples were greater than 96% for both herbicides.

RESULTS AND DISCUSSION

Overwinter losses of dinitramine and trifluralin from the November 26 application date until the first spring sampling on April 16 were substantial (Table 2). The mean overwinter losses of dinitramine and trifluralin in both soils was 55.4 and 38.1%, respectively. There were no differences ($P=0.5\%$) between the two soils with respect to these losses. It is not known whether these losses occurred uniformly throughout the winter, or in the spring when the ground had thawed. During this test the soil surface froze nightly at the time of application and snow covered the plots from mid-December until early March. Although losses attributed to microbial degradation or volatilization during this period might be expected to be minimal, rapid degradation of dinitro-aniline herbicides has been reported under anaerobic conditions (PROBST *et al.* 1967, SAVAGE 1978, WILLIS *et al.* 1974). Under anaerobic conditions, losses may be related to the redox potential of the soil and may be electrochemical in nature (WILLIS *et al.* 1974). The relatively long period of high soil moisture following the spring thaw may favour this type of herbicide degradation. The relatively large overwinter losses of these two herbicides should be an important consideration to those growers wishing to apply these herbicides in the fall.

TABLE 2

Levels (ppm) of dinitramine and trifluralin remaining in two sandy loam soils following application on Nov 26

Herbicide	Soil	Date of Sampling					
		Nov 26	Apr 16	May 16	Jun 20	Aug 29	Nov 23
Dini- tramine	Somer- set	0.36	0.17	0.15	0.10	0.05	0.05
Dini- tramine	Ber- wick	0.44	0.20	0.19	0.14	0.10	0.08
Tri- fluralin	Somer- set	0.36	0.22	0.23	0.17	0.17	0.17
Tri- fluralin	Ber- wick	0.42	0.26	0.18	0.16	0.14	0.12
Standard error of mean		0.0088	0.0072	0.0060	0.0045	0.0051	0.0036

Following the generally accepted spring application (Table 3), there had been significant losses of dinitramine from both soils and trifluralin from the Berwick soil by the time of the June 20 sampling. For unknown reasons, trifluralin levels were erratic in the Somerset soil following both fall and spring applications. The asymptotic regression equation, $Y=A+Brx^1$, accounted for 82.9 to 98.9% of the variability in herbicide levels over time for dinitramine in both soils and for trifluralin in the Berwick soil at either application time. However, less than 68% of the variability of trifluralin in the Somerset soil was explained by this equation.

The relative rate of disappearance of dinitramine was faster than trifluralin in both soils (Table 3). The half-lives, calculated from the asymptotic regression curves, were 51 and 72 days for dinitramine in the Somerset and Berwick soil, respectively, and 126 days for trifluralin in the Berwick soil following the spring application. Trifluralin had not dissipated to the 50% level in the Somerset soil over the 190 day test period. Others have also reported that the relative persistence of trifluralin is greater than dinitramine (HELLING 1976, STROLLER and WAX 1977, SAVAGE 1978). Under greenhouse conditions, SAVAGE (1978) determined the half-lives of dinitramine and trifluralin in a sandy loam soil to be 33 and 50 days, respectively. This agrees with our results in that trifluralin persists at least twice as long as dinitramine in sandy soils. However, under field conditions in Nova Scotia the persistence of phytotoxic levels of dinitroaniline herbicides, particularly trifluralin, may extend beyond the 4 month growing season. By August 29, about 60 and 85% of the spring applied trifluralin remained in the Berwick and Somerset soil, respectively (Table 3); and about 40% of the dinitramine remained in the Berwick soil. These levels of herbicide would be expected to damage sensitive fall-sown crops such as winter cereals. An indicator plant, Setaria viridis (L.) Beauv. showed severe stunting when grown in soils sampled in late-September from the trifluralin treated plots.

¹ Where A = asymptote; B = constant; r = relative rate of decrease/100 days; x = time/100.

TABLE 3

Levels (ppm) of dinitramine and trifluralin remaining in two sandy loam soils following application on May 16

Herbicide	Soil	Date of Sampling				
		May 16	Jun 20	Jul 20	Aug 29	Nov 23
Dini- tramine	Somer- set	0.23	0.15	0.10	0.04	0.03
Dini- tramine	Ber- wick	0.31	0.21	0.16	0.13	0.08
Tri- fluralin	Somer- set	0.38	0.40	0.30	0.32	0.28
Tri- fluralin	Ber- wick	0.34	0.24	0.19	0.20	0.15
Standard error of mean		0.0074	0.0087	0.0057	0.0071	0.0036

There were significant differences in the loss of herbicide from the two soils, but these differences were not consistent for both herbicides. The lower clay and organic matter content, and hence adsorptive capacity, of the Somerset soil (Table 1) would favor faster loss of herbicide from this soil. However, trifluralin was more persistent in the Somerset soil. Again the reason for this is not clear.

Regression curves indicated that the rate of herbicide dissipation was greatest immediately following herbicide application and this was followed by a slower rate of loss. Following the overwinter loss of herbicide after the fall applications, the remaining herbicide dissipated much slower than that applied in mid-May. It appears that with time the rate of loss decreases, possibly due to the binding of an increasing proportion of the remaining herbicide with soil constituents (KEARNEY et al. 1976).

PARKA and TEPE (1969) analyzed samples from 107 locations in the U. S. that had been commercially treated with trifluralin. Their results indicated that 5 to 7 months after application, trifluralin levels at the majority of sites were less than 15% of that originally applied, and that there was no evidence of trifluralin accumulation in soils with consecutive annual applications. Our results indicate that under Nova Scotia conditions, trifluralin is more persistent than in the majority of U. S. locations. Prolonged persistence of dinitroaniline herbicides has also been

reported in heavier prairie soils of Saskatchewan (RAHMAN and ASHFORD 1973). It appears that these herbicides are more persistent than previously assumed in the lighter soils of eastern Canada.

ACKNOWLEDGEMENTS

The authors thank Edmund Read for his assistance in setting out and sampling the test plots. Commercial products and analytical standards of dinitramine and trifluralin were supplied by Chipman Chemicals Ltd. and Eli Lilly and Company (Canada) Ltd.

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