SHOOT GRCWTH RESPONSE OF LOBLOLLY PINE, SWEETGUM, AND OAT TO SOIL-INCORPORATED DIPHENYLETHER HERBICIDES

David B. South'

Abstract. A greenhouse study was conducted to compare the relative phytotoxicity of soil incorporated bifenox [methyl 5-(2,4-dichlorophenoxy)-2-nitrobenzoate], oxyfluorfen [2-chloro-1-(3-ethoxy-4-nitrophenoxy)-4-(trifluoromethyl) benzene], and diclofop [2-[4-(2,4-dichlorophenoxy)phenoxy] propanoic acid] on shoot growth of loblolly pine (Pinus taeda L.), sweetgum (Liquidambar styraciflua L.), and oat (Avena sativa L.). Loblolly pine was tolerant of all three herbicides. Oat was susceptible to oxyfluorfen and diclofop but was tolerant to bifenox. Sweetgum showed tolerance to diclofop but was susceptible to bifenox and oxyfluorfen. Results are discussed in context with the possible residual effects on subsequent crops grown in forest nurseries. Assuming first-order half-lives of 8 weeks or less, the residue level of these herbicides after one year of normal use will likely not exceed 1.5 ppmw.

Additional index words. forest species, bioassay, oxyfluorfen, bifenox, diclofop.

Use of herbicides in forest nurseries has become an important cultural practice. However, Kozlowski (1979) suggested that a potential problem relates to the possible persistence of herbicides and accumulation of toxic residues in nurseries where beds are used repeatedly to grow a variety of tree species for different lengths of time. For example, at a forest nursery in Idaho, use of 3.4 kg ai/ha of napropamide [2-(a-naphthoxy)-N,M-diethylpropionamidel prior to June 10, 1980, resulted in severe injury to Western Larch occidentalis Nutt.) seedlings that were *sown* the following year on May 1 (Chatterton 1983). Therefore, the nurseryman should know if a herbicide can accumulate in the soil and if so, which crop species (tree or cover crop) are most sensitive and therefore likely to be the first to be affected.

Bioassay techniques have been used in northern forest nurseries to determine herbicide residues. Anderson (1970) used oats to bioassay residues of simazine [2-chloro-4,6-bis(ethylamino)-s-triazine], propazine [2-chloro-4,6-bis(isopropylamino)-s-triazine], dalapon [2,2- dichloropropionic acid], and neburon [1-butyl-3-(3,4-dichloropheny1)-1-methylurea] at the Webster Nursery in Washington. In Canada, a bioassay was used to help determine if linuron [3-(3,4-dichloropheny1)-1-methoxy-l-methylurea] residues were causing injury to Scots pine (Pinus sylvestria L.)(Canada 1980). Scots pine seedlings and radish (Raphanus sativus L.) were grown in soil containing known concentrations of linuron. By using the results of the indicator species (radish), it was determined that linuron residues in the soil were unlikely to affect pine growth.

[1] Assistant Professor and Director of the Auburn University Southern Forest Nursery Management Cooperative, Department of Forestry, Alabama Agricultural Experiment Station, Auburn University, AL. 36849-4201.

Utilization of bioassays to determine herbicide residues in southern forest nurseries has been very limited. In recent years, compounds in the diphenylether family have been the most commonly used herbicides in southern nurseries. Nitrofen [2,4-dichlorophenyl-p-nitrophenyl ether] has been used extensively in the past with sane nurserymen applying up to 20 applications each year. This would be equivalent to an annual application of 45 kg ai/ha. Nurserymen applied the herbicide at high rates without knowing the half-life and without knowing what possible effects nitrofen residues might have on future crops. Under certain conditions, nitrofen can be very persistent. Field studies in Canada have demonstrated that 38 to 46% of applied nitrofen was present 1 year later (Hayden and Smith 1980).

Presently, bifenox [methyl 5-(2,4-dichlorophenoxy)-2-nitrobenzoate] and oxyfluorfen [2-chloro-1-(3-ethoxy-4-nitrophenoxy)-4-(trifluoromethyl) benzene] are commonly used in forest nurseries in the South. Bifenox is a herbicide that is very similar in structure to nitrofen. In 1978, one nurseryman applied 10 applications of bifenox i.e. 22.4 kg ai/ha. Because pine seedlings are a high value crop, it is desirable to know what residual levels of diphenylether herbicides can cause injury to tree seedlings or to cover crops. For this reason, a bioassay study was conducted to determine the concentration of diphenylethers that would inhibit shoot growth of loblolly, sweetgum, and oats by 50 percent.

MATERIALS AND METHODS

One hundred and sixty-two milk cartons (7 by 7 by 10 cm) were filled with 400 g each of a 'awry sand soil (84% sand, 6% silt, 10% clay) which had been screened and air dried. The soil was treated with stock solutions of herbicides. The stock solutions were:

a. bifenox - 8 g technical grade (97% active ingredient) in 970 ml acetone.
b. oxyfluorfen - 10 g technical grade (71% active ingredient) in 887.5 ml acetone.
c. diclofop - 20 ml commercial grade (36% active ingredient) in 900 ml acetone.

Four concentrations of each herbicide were tested on each species. Controls were replicated six times while herbicide treatments were replicated four times. Herbicide concentrations consisting of 20, 40, 80, and 160 ppmw were prepared by applying 1, 2, 4, and 8 ml to the respective containers with a mechanical pipet. For concentrations less than 20 ppmw, a diluted stock solution was prepared by adding 950 ml of acetone to 50 ml of the original stock solution. This solution *was* used to prepare treatments containing concentrations of 1/2, 1, 2, 5, and 10 ppmw. Additional acetone was added so each container would be treated with 10 ml of acetone. After the acetone evaporated, the soil in each container was placed in separate bags, thoroughly mixed, and returned to the container. In one-third of the cartons, the soil was moistened with 50 ml of water and 13 oat seed were planted at a depth of approximately 2 cm. One-third of the cartons were planted with 20 loblolly seed at a depth of approximately 1 cm. The remaining cartons were each planted with 15 sweetgum seed at a 1 cm depth. The containers were placed in a greenhouse on April 10, 1980. After 7 days, the oat plants were thinned to 10 plants per container. On May 12, the oat shoots were harvested and fresh weights recorded. The pine and sweetgum were harvested on June 13. Shoots were dried for 24 h at 70 C and dry weights were subsequently recorded.

Standard curves were developed for the oat bioassay by using the total fresh and dry weights. Standard curves for pine and sweetgum used average shoot weight. In most cases, a linear relationship appeared to exist and a linear regression was calculated. GR50 values were determined from regression equations. Analysis of variance was conducted to determine the differences among herbicides or concentrations.

RESULTS

There was a linear relationship between fresh and dry weight of oat and the concentration of oxyfluorfen and bifenox (Table 1, Figures 1 and 2). Oat shoot growth was inhibited more by diclofop than oxyfluorfen. The GR50 values for fresh and dry weight of shoots growing in diclofop treated soil were 4.9 ppmw and 3.8 ppmw respectively. The GR50 values for oxyfluorfen were greater. Fresh and dry weight GR50 values were 12.2 ppnw and 11.5 ppmw.

Bifenox showed no relationship between oat yield and herbicide concentration (Table 1). GR50 values were not determined because even at 80 ppmw, oat shoot weights were not reduced by 50%.

Standard curves for average fresh and dry weights of sweetgum are presented in Figures 3 and 4. Of the three herbicides tested on sweetgum, oxyfluorfen was most inhibitory. The fresh weight GR50 value was 1.7 ppmw while the dry weight GR50 value was 1.5 ppmw.

The sweetgum GR50 values for bifenox were greater than 10 ppmw, therefore, these values had to be extrapolated from regression equations. The GR50 value for the fresh weight was ca. 11.7 ppmw while the value for dry weight was ca. 14.6 ppmw.

Sweetgum was least affected *by* diclofop. GR50 values could not be determined or extrapolated for diclofop since regressions equations were not significant.

Standard curves for average fresh and dry weights of pine are presented in figures 5 and 6. Analysis of variance determined that at the concentrations used, the herbicides did not significantly decrease fresh or dry weights of loblolly pine shoots. Oxyfluorfen at 10 to 80 ppmw significantly increased shoot dry weight. However, seedlings growing in soil treated with oxyfluorfen did not appear to be larger than control seedlings.

DISCUSSION

Compared to sweetgum and oat, loblolly pine was relatively tolerant to the three diphenylether herbicides used in this study. However, in another growth chamber study, early shoot growth of loblolly pine seedlings was reduced by oxyfluorfen at 20 ppmw (South and Mexal 1982). In addition, it should be noted that herbicides like bifenox and oxyfluorfen are much less effective when mechanically mixed into the soil that when sprayed on the soil surface. Weed control activity is greatly reduced when the chemical barrier is broken and the herbicide concentration is diluted by incorporation with additional soil. This helps explain why no stunting of pine was observed in the bioassay test (where the herbicide was soil incorporated) but hypocotyl growth of seedlings grown under nursery conditions have been reduced by surface applicatons of either bifenox at 3.4 kg ai/ha or oxyfluorfen at 1.12 kg ai/ha (South and Mexal 1982).

Oat and sweetgum responded differently to the herbicides. Bifenox was shown to have activity on sweetgum but had little effect on oat. Diclofop showed activity against oat but was relatively ineffective against sweetgum. Diclofop was the most effective in reducing shoot growth of oats. In this respect, it was three times more active than oxyfluorfen and 30 times more active than bifenox. Oxyfluorfen showed activity on both species. Based on GR50 (dry weight) values, 10 times as much bifenox and 18 times as much diclofop would be needed to achieve a sweetgum shoot-growth reduction level equivalent to that of oxyfluorfen.

Persistence of bifenox. The half-life of ^{14}C-labeled bifenox has been reported to be 7 to 14 days (Table 2). However, the biological activity resulting from an application of bifenox may be longlived. For example, data collected from one forest nursery indicates that a single application of 3.4 kg/ha of bifenox provided significant weed control 13 to 16 weeks after application (Gjerstad and South 1976). With a half-life of 14 days, 0.026 kg/ha of bifenox would remain after 14 weeks following a 3.4 kg/ha application. It seems unlikely that this level could be resulting in a 45% reduction in handweeding time. This residual herbicidal activity could possibly be explained by the activity of bifenox metabolites. Bifenox and its free-acid metabolite are illustrated in Figure 7. Figure 8 shows that as much as 40% of the applied bifenox can be in the acid form 60 days after application (Ohyama and Kuwatsuka 1978). This suggests that even though the reported half-life of a herbicide is short, the biological activity may persist due to the longevity of certain degradation products.

Normal usage of bifenox in a southern pine nursery consists of applying a preemergence application at 3.4 kg/ha followed by three postemergence applications (each at 2.2 kg/ha) at 4 to 6-week intervals. Figure 9 illustrates a possible residue pattern assuming a first-order half-life of 8 weeks for bifenox and metabolites and an application interval of 8 weeks. If the preemergence treatment was applied on May 22, then approximately 2.2 kg ai/ha would remain on January 1. Assuming no further degradation over the winter and thorough soil incorporation to a depth of 15 cm, a residue of approximately 1 ppmw would be present the following spring. Results from this study indicate that if sweetgum were sown on a soil containing 1 ppmw of bifenox, stunted seedlings would be unlikely .

Even if pine were grown continuously on the same area, the accumulated residue (assuming first-order kinetics) would be slight (Hamaker 1966). With the above assumptions, the accumulated residue for a single year would be less than 1.1 ppmw.

As previously mentioned, bifenox has been used in forest nurseries at several times the recommended rate. One nurseryman applied 2.24 kg/ha every 2 weeks for a total application during the year of 22.4 kg/ha. Figure 10 shows the possible residue pattern from this type of herbicide usage. If treatments began on May 7, then approximately 3.3 kg/ha or 1.5 ppiw of bifenox would remain on January 1 (assuming thorough soil incorporation to a depth of 15 cm). Assuming that the first-order half-life for bifenox (and its metabolites) is 8 weeks or less, it is unlikely that a sweetgum crop *sawn* the following spring would be adversely affected.

Persistence of oxyfluorfen. The half-life of oxyfluorfen is reported to be about 30 to 40 days (Table 2). However, as with bifenox, preemergence activity in a forest nursery can persist for several months. At one nursery, a preemergence application of oxyfluorfen at 0.56 kg/ha provided 84% weed control 2 to 3 months after treatment (Gjerstad and South 1977). Use of oxyfluorfen in a southern pine nursery has consisted of applying 0.56 to 1.12 kg/ha at time of sowing followed by up to three postemergence applications (each at 0.56 kg/ha). If we assume a first-order half-life of 8 weeks, a preemergence application on May 22 of 1.12 kg/ha, and three postemergence applications at 8 week intervals, then approximately 0.56 kg/ha would remain on January 1. Assuming no further degradation over the winter and thorough soil incorporation to a depth of 15 cm, approximately 0.25 ppmw would remain the following spring. This residue would be about one-sixth of the GR50 value for sweetgum. Even if oxyfluorfen was applied on the same area for several years (and assuming the same timing and application rates as above), the accumulated residue would only be 0.27 ppmw. Although same species may be affected Icy this level of residue, results from this study indicate that neither sweetgum nor oats would be adversely affected. However, these calculations are based only based on theory. Currently, there are no publications that demostrate decomposition of oxyfluorfen (and its metabolites) is of the first-order type.

Persistence of diclofop. The half-life of diclofop has been reported to be 6 to 38 days in soils under aerobic conditions (Martens 1978). Smith (1979) found no significant amounts of diclofop-methyl 5 months after treatment with 1.5 kg ai/ha. North and Lealy (1979) treated a sandy loam and a loam with diclofop and found less than 10% of the parent compound present 30 days after treatment. Wu and Santelmann (1976) detected no soil phytotoxicity after 8 weeks. Degradation of diclofop has been shown to be dependent on soil temperature and the moisture content (Martens 1978; Wu and Santelmann 1976).

During the degradation process, the parent compound diclofop-methyl undergos hydrolysis and converts to the acid form (Figure 7). Dichlofop acid is apparently the major degratation product (Smith 1977). It should be noted that the half-life of the acid is longer than the parent compound as Figure 8 indicates. However, Smith (1979) found no significant amounts of this degration product remaining 5 months after treatment.

Since southern forest nurseries receive irrigation throughout the growing season, it is likely that dissipation of diclofop and diclofop acid would occur more rapidly in forest nurseries than under non-irrigated agronanic conditions. The rapid degradation rate of these compounds along with the results from this study suggest that neither loblolly pine nor sweetgum would be stunted by residual levels of diclofop. However, diclofop is not currently used in southern forest nurseries due to the registration of more effective herbicides for controlling grasses.

ODNCLUSIONS

The conclusions from this study are important to forest nurserymen who frequently use diphenylether herbicides in their weed control programs. If bifenox, oxyfluorfen, diclofop, and their metabolites follow a first-order half-life of 8 weeks or less, then the residual levels of these herbicides in southern forest nurseries will likely not exceed 1.5 pprw. One must remember, however, that the half-life of a herbicide is dependent on soil conditions such as soil temperature and moisture. Degradation rates may, therefore, vary greatly among regions. Frequent use of certain diphenylether herbicides (e.g., nitrofen) in cooler climates might result in residue problems because the half-life will likely be much longer than typically found in southern nurseries.

Table 1. Regression equations used for calculation of GR50 values.

Species	Herbicide	Linear regressions of shoot weight (g) on herbicide (ppm) intercept slope	r^2	Probability of a greater F value
Oat fresh weight	oxyfluorfen	2.0059 − .0862 ppm	.88	.0180
	bifenox	1.7044 − .0066 ppm	.30	.3344
	diclofop	1.8923 − .1933 ppm	.99	.0001
Oat dry weight	oxyfluorfen	.4165 − .0178 ppm	.96	.0029
	bifenox	.3412 − .0011 ppm	.14	.5278
	diclofop	.3676 − .0416 ppm	.85	.0267
Sweetgum fresh weight	oxyfluorfen	.1018 − .0288 ppm	.98	.0946
	bifenox	.1084 − .0048 ppm	.90	.0036
	diclofop	.1231 − .0016 ppm	.33	.2297
Sweetgum dry weight	oxyfluorfen	.0150 − .0045 ppm	.81	.2906
	bifenox	.0162 − .0005 ppm	.59	.0747
	diclofop	.0209 − .0005 ppm	.55	.0936
Loblolly fresh weight	oxyfluorfen	.2183 − .00016 ppm	.14	.4604
	bifenox	.1809 + .00007 ppm	.22	.3531
	diclofop	.2199 − .00025 ppm	.16	.4382
Loblolly dry weight	oxyfluorfen	.0371 − .00002 ppm	.09	.5721
	bifenox	.0294 + .00003 ppm	.81	.0141
	diclofop	.0281 + .00002 ppm	.36	.2055

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Table 2. Reported half-life of selected herbicides.

Common name	Trade name	Half-life[1] (months)
sethoxydim	Poast	1/6-1/2
fluazifop-butyl	Fusilade	<1/4
fluazifop		3/4
diclofop-methyl	Hoelon	<1/4
diclofop		1/4-1
EPTC	Eptam	1/4
bifenox	Modown	1/4-1/2
butylate	Sutan	1/3-3/4
metalochlor	Duel	1/2-1
simazine	Princep	1
perfluidone	Destun	1
oxyfluorfen	Goal	1-1.5
chlorsulfuron	Glean	1-1.5
acifluorfen	Blazer	1-2
prometryn	Caparol	1-3
hexazinone	Velpar	1-6
napropamide	Devrinol	2-3
diphenamid	Enide	3-6[2]
pronamide	Kerb	2-9[2]

1 (Weed Science Society of America 1983)

2 Persistence (not half-life)

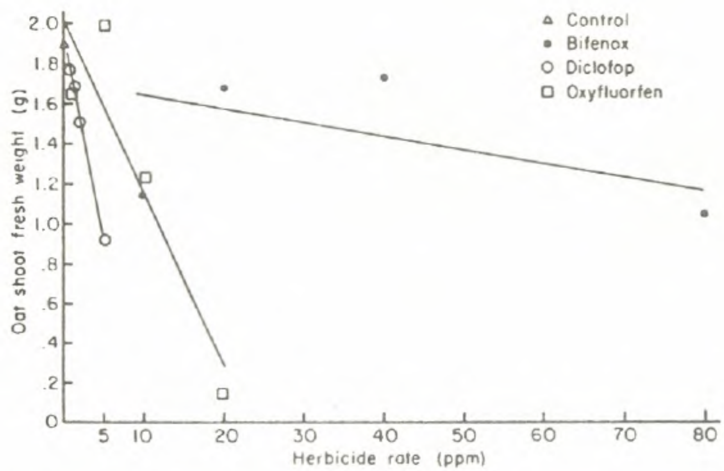

FIGURE 1. INFLUENCE OF HERBICIDE AND CONCENTRATION ON OAT SHOOT FRESH WEIGHT.

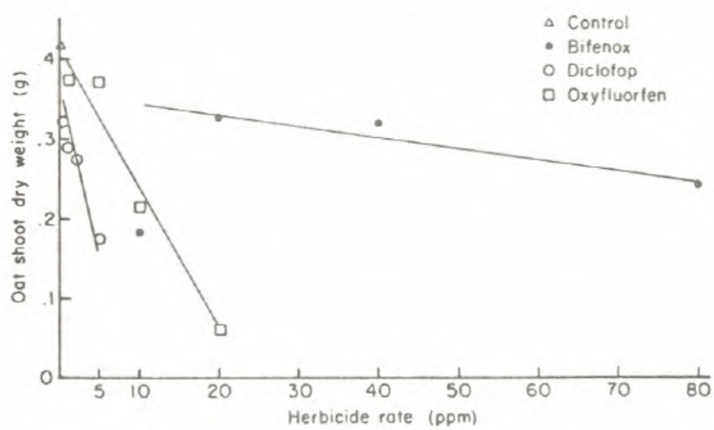

FIGURE 2. INFLUENCE OF HERBICIDE AND CONCENTRATION ON OAT SHOOT DRY WEIGHT.

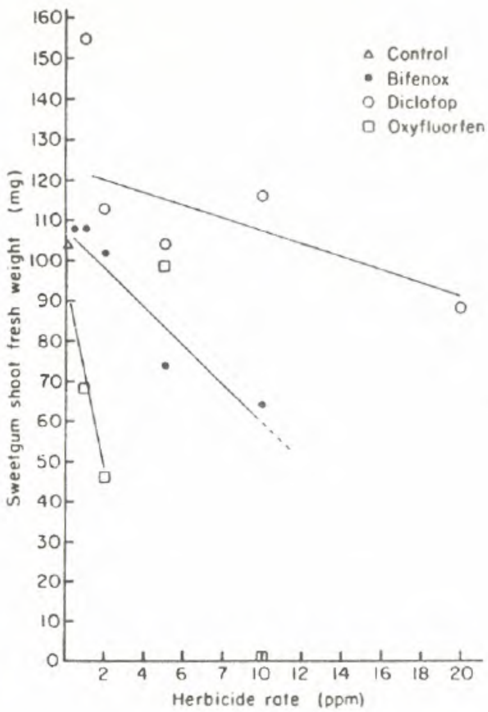

FIGURE 3. INFLUENCE OF HERBICIDE AND CONCENTRATION ON SWEETGUM SHOOT FRESH WEIGHT.

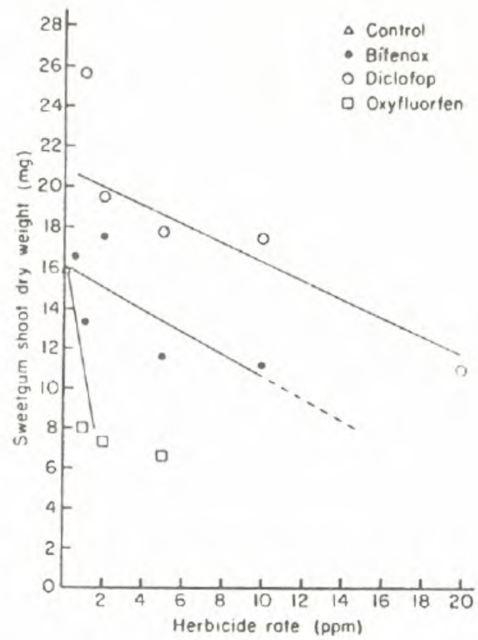

FIGURE 4. INFLUENCE OF HERBICIDE AND CONCENTRATION ON SWEETGUM SHOOT DRY WEIGHT.

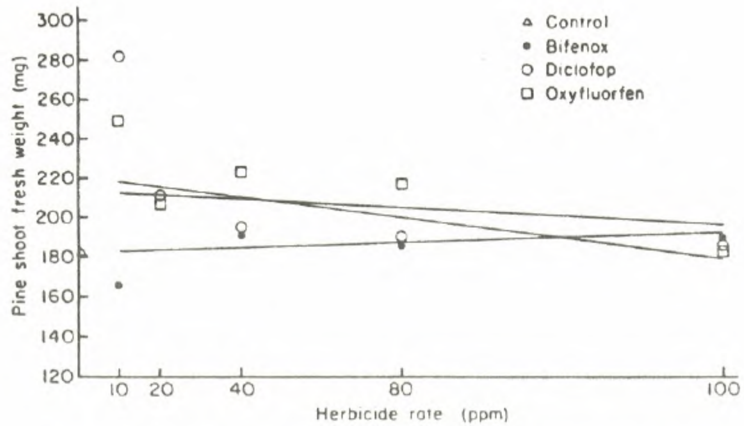

FIGURE 5. INFLUENCE OF HERBICIDE AND CONCENTRATION ON PINE SHOOT FRESH WEIGHT.

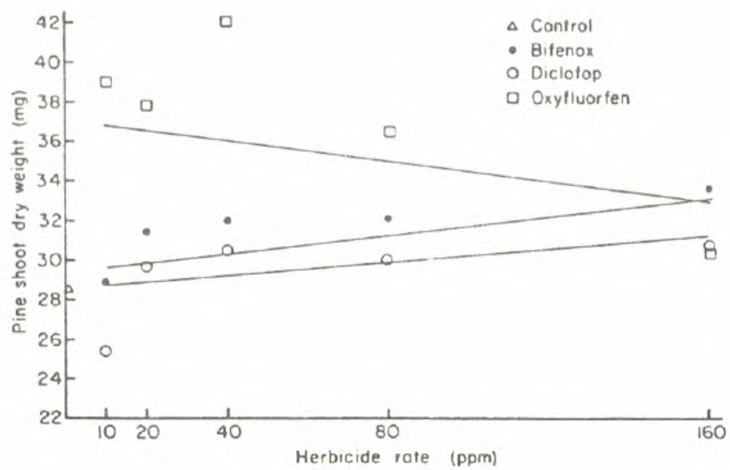

FIGURE 6. INFLUENCE OF HERBICIDE AND CONCENTRATION ON PINE SHOOT DRY WEIGHT.

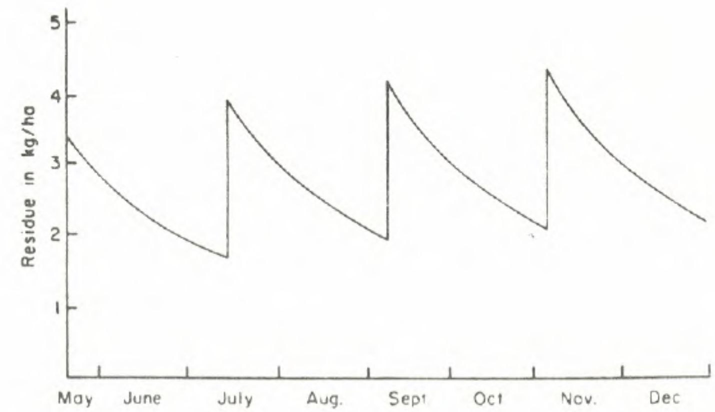

FIGURE 7. RESIDUE PATTERN FOR FOUR APPLICATIONS OF A HERBICIDE WITH A FIRST-ORDER HALF-LIFE OF 8 WEEKS.

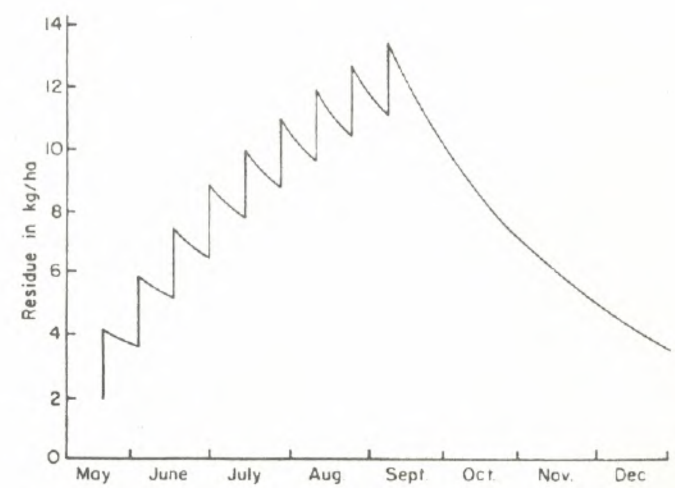

FIGURE 8. RESIDUE PATTERN FOR TEN APPLICATIONS OF A HERBICIDE WITH A FIRST-ORDER HALF-LIFE OF 8 WEEKS.

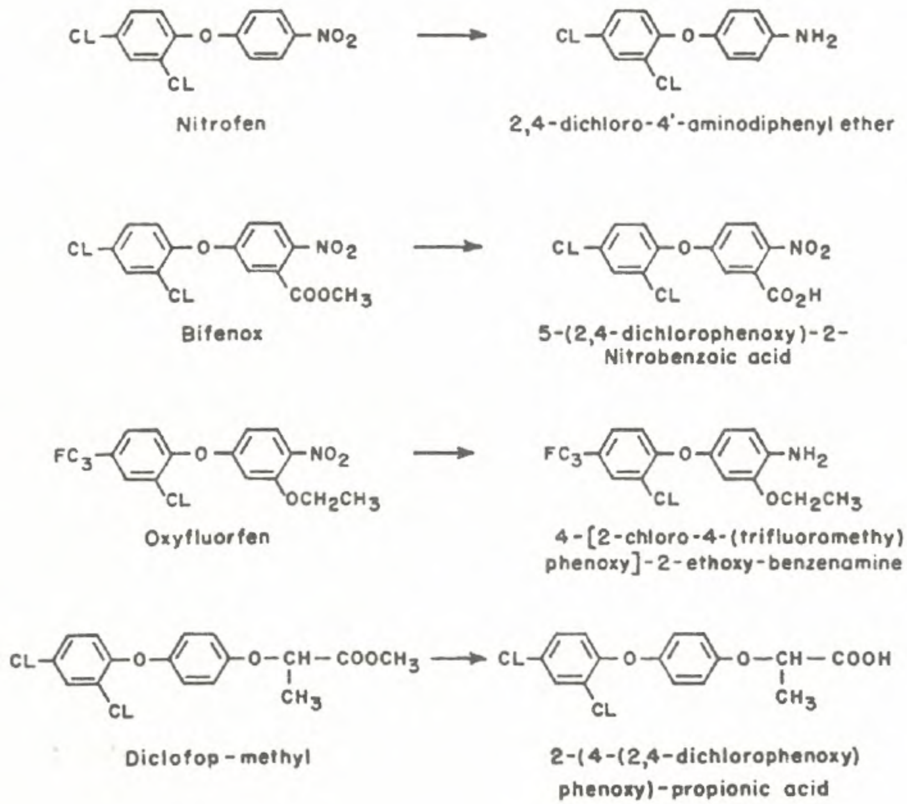

FIGURE 9. PRIMARY DEGRADATION PRODUCTS OF SELECTED DIPHENYLETHER HERBICIDES.

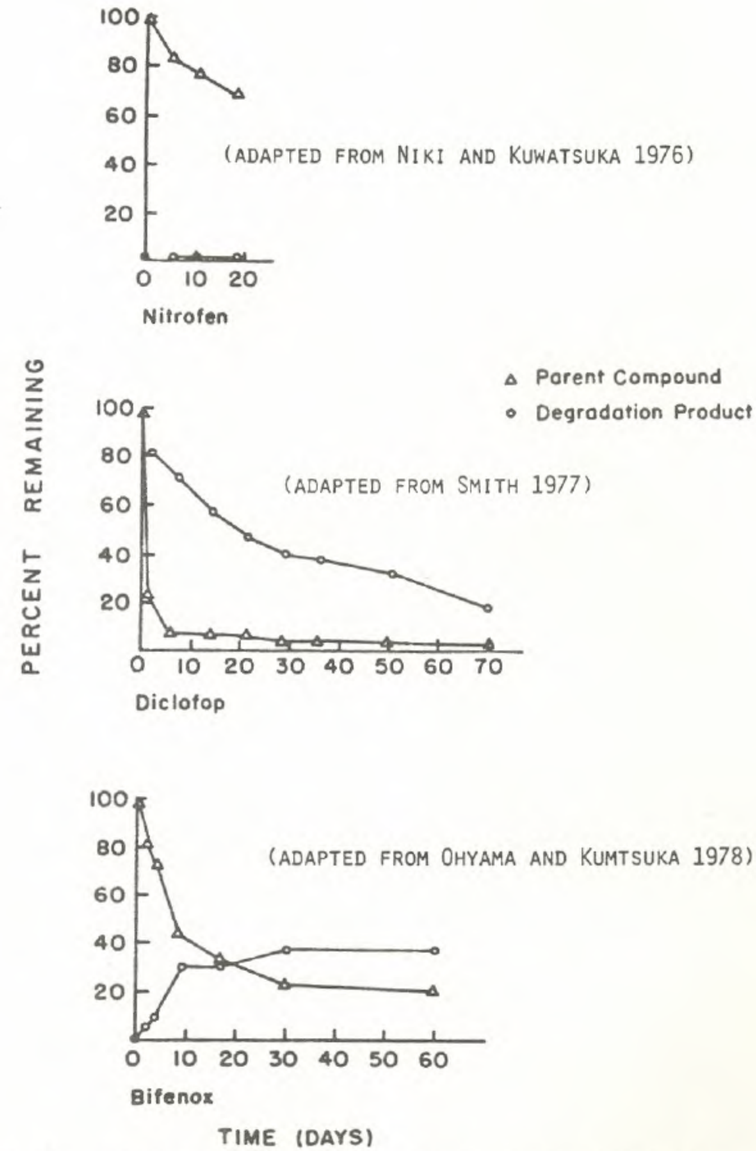

FIGURE 10. DEGRADATION OF SELECTED DIPHENYLETHER HERBICIDES.