

Persistence, Degradation, and Movement of Triclopyr and Its Ethylene Glycol Butyl Ether Ester in a Forest Soil

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Triclopyr, [(3,5,6-trichloro-2-pyridinyl)oxy]acetic acid, and its ethylene glycol butyl ether ester (EGBE), equivalent to 3.5 mg of the acid, were applied, separately, to the top layers of columns (40 cm × 8.9 cm i.d.) packed with a loam soil or with quartz sand. Water, equivalent to 2.5 cm of precipitation, was leached through each column every second day. After 54 days, residues were found only in the top 10-cm layers of the soils; they were triclopyr and the metabolites 3,5,6-trichloro-2-pyridinol (TP) and 2-methoxy-3,5,6-trichloropyridine, accounting for about 5, 85, and 10%, respectively, of the total of the residues which, in turn, was equivalent to 65% of the amount applied initially to the top layers. No residues were detected in the lower soil layers or the eluates. Triclopyr-treated sand: Found were 10 and 65%, respectively, in the sand and the eluates after 54 days. EGBE-treated sand: All residues were in the eluates after only 34 days. All residues from sand and eluates were in the form of triclopyr, except a few eluates from the latter treatment with traces of EGBE or TP.

INTRODUCTION

The herbicide triclopyr, [(3,5,6-trichloro-2-pyridinyl)oxy]acetic acid, was developed by The Dow Chemical Co. and is marketed under the trade name Garlon. It has been found to be more effective than 2,4,5-T against a wide range of woody plants and broad-leaved weeds (Byrd et al., 1974, 1975), with promise for use on right-of-way (Reynolds et al., 1983) and for conifer release (Newton et al., 1982) and site preparation in forestry (Gratkowski et al., 1978). Triclopyr is of low mammalian toxicity (acute oral LD₅₀, rats, 713 mg/kg) and of moderate toxicity to trout and bluegills with 96-h LC₅₀ of 117 and 148 ppm, respectively (Weed Science Society of America, 1983). According to one study, triclopyr has been found to break down in soil to 3,5,6-trichloro-2-pyridinol (TP) (McKellar et al., 1982), a compound that is relatively nontoxic to mammals (Roberts and Marshall, 1978), but its toxicity to fish is not known. Methylation of TP in soils, yielding 2-methoxy-3,5,6-trichloropyridine (MTP), a metabolite whose biological activities appear to be unknown, has been reported by Roberts and Marshall (1978).

Triclopyr is currently considered for use in forestry in the province of British Columbia, Canada, but nothing is known about its fate in this particular environment. Our objectives were to study the movement and degradation of triclopyr and its ethylene glycol butyl ether ester (EGBE) and the formation and movement of TP and MTP in a soil typical of British Columbia coastal forests under laboratory conditions.

MATERIALS AND METHODS

Reagents. Triclopyr (99.1%), EGBE (99.1%), TP (99%), MTP (99%) and Garlon 4, an emulsifiable concentrate (EPA (U.S.) Reg. No. 464554), containing 61.6% EGBE (=44.3% triclopyr equivalent = 0.48 kg/L = 4 lbs triclopyr/U.S. gal) were provided by The Dow Chemical Co., Midland, MI. Florisil PR grade (Supelco Canada Ltd.) was heated to 120° C overnight and equilibrated with 2% (w/w) water prior to use. Anhydrous granular Na₂SO₄ was kept at 120° C prior to use. All organic solvents were glass distilled. A 1% solution of paraffin oil in hexane was prepared to serve as keeper.

A solution of diazomethane in diethyl ether was prepared from *N*-methyl-*N*-nitroso-*p*-toluenesulfonamide

(Pierce Chemicals) according to the method of Maybury (1980). All ground-glass surfaces of the apparatus were covered with Teflon sleeves to prevent accidental explosions.

Safety. For purposes of health protection, all manipulations involving benzene and the preparation and use of diazomethane were carried out in fume hoods.

Soil Column Studies. The soil used in our studies (34% organic matter, 8.3% clay, 45.4% sand, 46.3% silt, pH 3.4) was collected from the top 1 m, after the duff had been removed, in a virgin cedar-hemlock forest in the wet coastal biogeoclimatical zone of British Columbia. It was from an area of predominantly Cannel type soil (Luttmerding, 1980). The soil was air-dried, passed through a No. 10 sieve (2-mm openings) to remove large particles, and mixed.

Prewashed and dried silica sand (Martin Marietta, Emmett, ID), containing no clay or organic matter, was passed through a No. 35 sieve (0.5-mm openings) and served as control.

Glass columns (73 cm × 8.9 cm i.d.) were filled with sand or soil to a height of 40 cm (Wilson and Oloffs, 1973). The contents of each column were held in place by a sheet of Teflon mesh and fastened to the outside of the base with silicone sealant and rubber bands. Each column was clamped near the top and rested on a glass funnel suspended in a round opening of a plywood plank. A 250-mL Erlenmeyer flask was placed beneath each funnel for collecting eluates. Water was applied to each column from an inverted 500-mL Erlenmeyer flask, stoppered by a cork with two glass tubes: one for pressure equalization and the other for water release. To the latter was attached rubber tubing with an adjustable clamp to facilitate restriction of the water flow from the Erlenmeyer flask to the rate of elution from the column, or less. Ten columns were set up: five columns each of soil and sand. All columns were conditioned with five applications of 250 mL of distilled water for 5 days.

For the treatments of soil and sand, triclopyr (solution A) and Garlon 4 (solution B) were dissolved in acetone at 0.35 mg/mL of either triclopyr (A) or triclopyr equivalent (B). Five 100-g samples each of soil or sand were treated: two with 10 mL of solution A, two with solution B, and one with 10 mL of acetone only. Thus, each herbicide-treated sample received 3.5 mg of triclopyr or its equivalent. Each sample, while tumbling in a round glass jar, was treated by slowly adding 2 × 5 mL of treatment solution with a syringe. Tumbling was continued for a minimum of 1 h to ensure mixing and evaporation of solvent.

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One treated sample of sand or soil was added to the top of an appropriate column, covering the nontreated sand or soil. A 9-cm Whatman #1 filter paper was placed on top of the treated layer, followed by 5 cm of untreated sand in all cases and another disk of filter paper. The filter papers and sand layers were added to prevent channelling of the applied water.

Every second day, 160 mL of water (equivalent to 2.5 cm of rain) was applied to each column at a rate not exceeding the rate of elution. The resulting eluates, collected within 48 h, were transferred to 200-mL dark glass bottles and stored at -20°C until analyzed. Twenty-seven eluates per column, ranging in volume from 156 to 161 mL, were collected during 54 days of leaching. Thereafter, each column was wrapped with aluminum foil and stored at -20°C until analyzed.

Preparation of Soil and Sand for Analysis. Each frozen soil column was taken from the freezer and clamped to a retort stand for the removal of the Teflon mesh. A sharp razor blade, rinsed with acetone, was used to remove the silicone sealant from the outside around the base of the column and hence the Teflon mesh. Paper towels were soaked in warm water and wrapped around the column to accelerate thawing of the soil adjacent to the glass surface and facilitate its separation.

A round wooden rammer (8 cm in diameter), covered with acetone-rinsed aluminum foil, was pushed gently against the top of the soil core. When there were signs of sliding, the core was pushed out onto a clean sheet of aluminum foil. A hacksaw that had been cleaned with acetone was then used to saw the core into four equally long sections labeled 1–4 from the top down. Upon complete thawing, each section was thoroughly mixed and 50-g aliquots were removed for analysis.

Extraction of Water Eluates. The eluates were thawed, and their volume was determined. Ten drops of concentrated H_2SO_4 were added to a 50-mL aliquot to acidify to about pH 2; then, it was extracted three times in a 250-mL separatory funnel with CH_2Cl_2 (dichloromethane), 50 mL for the first extraction and 25 mL for the second and third.

After each extraction, the solvent phase was passed through a bed of anhydrous granular Na_2SO_4 and collected in a 250-mL round-bottom flask. After the third extraction, a further 10 mL of CH_2Cl_2 was used to rinse the Na_2SO_4 bed. The combined extracts were concentrated to just dryness in a flash evaporator at 38°C , and the residues were redissolved in 1 mL of benzene for derivatizing with diazomethane.

Extraction of Soil and Sand. To each 50-g soil or sand sample in a wide-mouth, 500-mL Mason jar were added 80 mL of acetone, a drop of concentrated H_2SO_4 , and 30 g of anhydrous Na_2SO_4 before the sample was blended in a Sorvall Omni mixer at 20 V (Varian speed regulator) for 5 min. The acetone extract was then filtered, under aspiration, through a Whatman #1 filter paper in a Büchner funnel, into a conical flask. Extraction was repeated with 2×70 mL of acetone. The combined extracts were made to a final volume of 250 mL in glass-stoppered measuring cylinders.

Ten milliliters of the acetone extract, equivalent to 2 g of the soil or sand, was transferred to a 250-mL separatory funnel, and 50 mL of acidified glass-distilled water (pH 2) was added. They were extracted with CH_2Cl_2 as described for water eluates for derivatizing with diazomethane.

Derivatizing with Diazomethane. To the extracts, redissolved in 1 mL of benzene, was added 1 mL of freshly

prepared diazomethane in diethyl ether. The samples were mixed and allowed to react for 30 min at room temperature. Then, 10 drops of keeper was added and unreacted diazomethane driven off with a stream of nitrogen.

Cleanup. The methylated residues were cleaned up as follows. Three grams of deactivated Florisil were evenly packed in glass columns (40 cm \times 1.4 cm i.d.) between two layers of granular anhydrous Na_2SO_4 . Each column was then washed with 10 mL of 10% diethyl ether in hexane (v/v), and the eluates were discarded.

Following the removal of unreacted diazomethane, the derivatized extracts were transferred to the Florisil columns and eluted with 25 mL of 10% diethyl ether in hexane. The eluates were concentrated to 2 mL in a flash evaporator at 38°C for analysis.

Analysis by Gas-Liquid Chromatography. We used a Microtek MT-220 gas chromatograph with a ^{63}Ni electron-capture detector and a glass column (180 cm \times 2 mm i.d.) containing 4% SE-30 and 6% SP2401 on 80/100-mesh Supelcoport, for compound separation. Nitrogen, 70 mL/min, was the carrier gas. Operating temperatures: detector, 285°C ; inlet and outlet, 220°C ; oven, 220°C for EGBE and 175°C for the methyl ester of triclopyr and for MTP.

To determine MTP and EGBE, aliquots of extracts were cleaned up directly, i.e. without being derivatized, thus avoiding the methylation of TP to produce MTP. They were determined with authentic reference standards. Triclopyr was analyzed as the methyl ester after derivatization, with reference standard similarly prepared with authentic triclopyr. TP was determined as the methylated derivative, with reference standard similarly prepared. The actual concentration of TP was determined by subtracting the MTP found in the underivatized aliquot from that of the derivatized aliquot.

Recovery and Storage Stability. Quadruplicate samples of residue-free soil, sand, and their eluates were fortified to give concentrations of 0.001, 0.1, and 1.0 ppm of triclopyr, TP, and EGBE in eluates; and 0.01, 0.1, and 5.0 ppm of triclopyr and TP or 0.1 and 5.0 ppm of EGBE in soil and sand. Fortified samples of soil and sand were thoroughly mixed, as described, and then held at 4°C for 12–18 h prior to extraction.

Periodic analyses of aliquots of experimental samples, stored at -20°C for up to 120 days, were undertaken to determine the storage stability of the compounds of interest.

RESULTS

Recovery and Storage Stability. The means of recoveries, in percent of added, for each residue from all fortified samples were as follows: TP, 90.1%; triclopyr, 96.7%; EGBE, 98.7%. Generally, the recoveries varied with the concentrations added to the substrates (Table I). When added to soil, but not when added to sand or eluates, 42–49% of EGBE was hydrolyzed to triclopyr within 12–18 h of treatment and storage at 4°C . Periodic analyses of aliquots of experimental samples, stored at -20°C for up to 120 days, were performed. Results varied within 10%, showing that the residues were stable under these conditions of storage.

Sand. The leaching of triclopyr with water in quartz sand treated with triclopyr, or with Garlon 4 containing EGBE, is shown in Figure 1. Traces of triclopyr (0.005 ppm) were detected in the first eluates, collected after the equivalent of 2.5 cm of rain had passed through the triclopyr- and EGBE-treated sand columns. Thereafter, concentrations in the eluates increased rapidly, reaching maxima of 3.0 and 3.6 ppm, respectively, after 10 cm of

Table I. Recovery, in Percent of Added Triclopyr (T), 3,5,6-Trichloro-2-pyridinol (TP), and Ethylene Glycol Butyl Ether Ester of Triclopyr (EGBE) from Fortified Sand, Soil, and Eluates, 12–18 h after Fortification

substrate	fortificn, ppm	% recovery ($\bar{x} \pm SD$, $n = 4$)		
		T	TP	EGBE
sand	5.0	100.8 \pm 6.9	95.4 \pm 6.0	101.5 \pm 4.3
	0.1	92.0 \pm 6.8	92.2 \pm 5.6	100.3 \pm 3.1
	0.01	90.5 \pm 7.8	80.3 \pm 3.4	not done
eluates, sand	1.0	94.7 \pm 5.8	90.2 \pm 6.7	98.4 \pm 3.6
	0.1	90.3 \pm 5.8	84.4 \pm 3.6	97.0 \pm 3.8
	0.001	92.5 \pm 3.5	88.1 \pm 7.3	91.6 \pm 6.6
soil	5.0	100.0 \pm 3.9	94.7 \pm 3.3	106.9 \pm 4.7 ^a
	0.1	100.0 \pm 1.9	98.4 \pm 4.0	98.9 \pm 5.6 ^b
	0.01	102.0 \pm 4.8	80.9 \pm 3.0	not done
eluates, soil	1.0	104.2 \pm 6.6	100.6 \pm 7.2	100.8 \pm 2.9
	0.1	97.2 \pm 2.8	95.6 \pm 3.3	96.5 \pm 1.4
	0.001	95.8 \pm 5.4	80.6 \pm 6.1	95.4 \pm 3.4

^a 48.7% as T. ^b 41.7% as T.

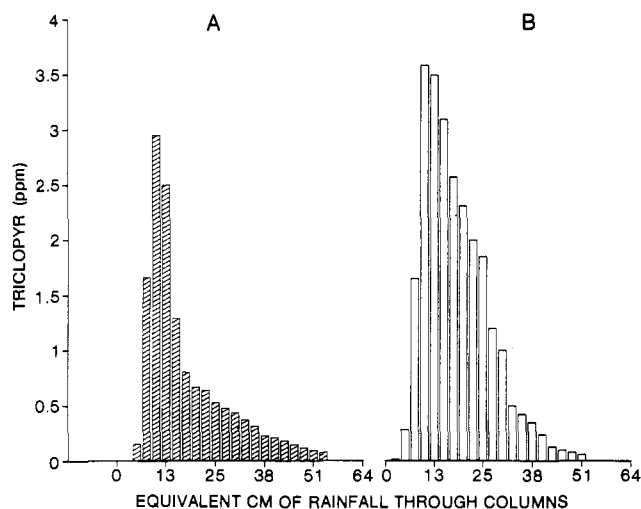


Figure 1. Concentrations of triclopyr in eluates from sand treated with triclopyr (A) and garlon 4 containing EGBE (B). Each eluate resulted from 2.5 cm of water applied to the top of the column every second day.

rainfall equivalent. A rapid decline of the residues in the eluates of the triclopyr-treated sand followed, until an equivalent of 15–18 cm of rainfall had moved through the columns (Figure 1A). Then, the concentrations tapered off more gradually.

Residue concentrations in the eluates of the EGBE-treated sand (Figure 1B) not only had a higher maximum in the eluate resulting from the fourth simulated precipitation but also remained higher in the following eight eluates (5th–12th) than the concentrations in the corresponding eluates from triclopyr-treated sand, shown in Figure 1A. After the 20th and 21st eluates, residues became nondetectable (Figure 1), except a few samples with traces of triclopyr (<0.005 ppm).

Occasionally, traces (<0.005 ppm) of TP were detected in both triclopyr and Garlon-4-affiliated eluates and traces of EGBE in a few of the latter. There was no discernible pattern for the occurrence of these traces, and they may have been artifacts.

The total quantities of triclopyr, in percent of amount added, that were found in the eluates are shown in Figure 2. Recovery from the eluates, in the form of triclopyr, was quantitative 34 days after treatment of sand with EGBE, whereas for the triclopyr-treated sand, 65% was found in the eluates after 54 days of leaching.

Analysis of the individual sections of the sand columns showed that some triclopyr had remained in the sand

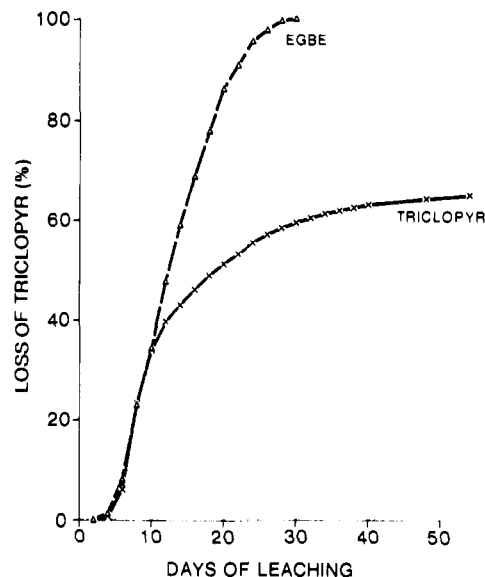


Figure 2. Disappearance of triclopyr from sand columns into eluates, in percent of applied.

Table II. Concentrations of Triclopyr (T), 3,5,6-Trichloro-2-pyridinol (TP), and 2-Methoxy-3,5,6-trichloro-2-pyridinol (MTP) in the Sections of the Soil or Sand Columns, Found by Analysis after the Leaching Had Been Terminated

column	treatment ^a	core section	concn, ppm		
			T	TP	MTP
sand	T	1	0.296	0 ^b	0
		2	0.012	0	0
		3	0.005	0	0
		4	0.032	0	0
	EGBE	1	0	0	0
		2	0	0	0
		3	0	0	0
		4	0	0	0
soil	T	1	0.158	1.935	0.402
		2	0	0	0
		3	0	0	0
		4	0	0	0
	EGBE	1	0.214	2.667	0.206
		2	0	0	0
		3	0	0	0
		4	0	0	0

^a EGBE = ethylene glycol butyl ether ester. ^b Not detectable.

Table III. Amounts (μg) of Triclopyr (T), 3,5,6-Trichloro-2-pyridinol (TP), and 2-Methoxy-3,5,6-trichloro-2-pyridinol (MTP) Recovered from Soil or Sand after Completion of Leaching (Ethylene Glycol Butyl Ether Ester (EGBE) Not Detected)

treatment	T	TP ^a	MTP ^a	total	% of appl
Soil					
T	81.5	1666	304.8	2052	58.6
EGBE	123.2	1998	144.2	2266	64.7
Sand					
T	341.3	0 ^b	0	341.3	9.75
EGBE	0	0	0	0	

^a As triclopyr equivalent. ^b Not detectable.

treated with triclopyr, but not in sand treated with EGBE; the concentrations were low and variable (Table II). Neither TP, EGBE, nor MTP was detected. The amount of triclopyr recovered from these columns was 9.75% of that applied originally (Table III). Thus, total amounts of triclopyr recovered in the eluates and sand from these columns were approximately 75% of that applied (Figure 2; Table III).

Soil. No residues could be detected in eluates or in Sections 2-4 of the soil columns, regardless of treatment (Table II). Triclopyr, TP, and MTP were present only in Section 1 of each soil column; the concentrations were TP > MTP ≥ triclopyr (Table II). The actual quantities of TP were more than 1 order of magnitude above that of triclopyr (Table III) and accounted for 81% and 88% of the totals of the residues (T + TP + MTP) found by analysis. Somewhat more residues were found in EGBE than in T-treated soil, namely 65% and 59%, respectively (Table III).

DISCUSSION

Each treatment of the soil and sand columns with triclopyr or EGBE was equivalent to 5.6 kg/ha, thus exceeding $2^{1/2}$ - to 5-fold the rates of 1.1-2.2 kg/ha considered for conifer release and for the control of broad-leaved weeds (Newton et al., 1982; Byrd et al., 1975). Therefore, any interpretation of our results will tend to be conservative, i.e. on the side of caution.

That residues were recovered quantitatively, in the form of triclopyr, from the eluates of EGBE-treated sand, while only about 65% was recovered from the eluates of triclopyr-treated sand, may be attributable to adjuvants in Garlon 4 such as surfactants facilitating the affinity to water of nonpolar substances of low water solubility. Comparison of results suggests further that adjuvants present in the formulation, although not blocking hydrolysis of EGBE to triclopyr, the herbicidal principle, may have stabilized triclopyr, preventing its breakdown to TP and to innocuous substances. Such an effect, exerted by adjuvants, could explain why about 25% of the residues could not be found by analysis in the samples from sand, treated with triclopyr, and why about 10% had not been eluted, although 1.6 times more water had been leached through this than through the EGBE-treated sand. A paucity of microbiological activity or the lack of soil colloids with catalytically active sites in the quartz sand does not explain these results. It seems that the disappearance of 25% triclopyr—presumably through reactions yielding innocuous products—in the experiments with triclopyr-treated sand but not in those with EGBE-treated sand, can only be ascribed to the formulation.

Pure quartz sand, used here as a control, is not a soil. It does not support growth of plants under field conditions, and our results obtained with the pure quartz sand must not be seen to evince any perils to the environment. Nevertheless, it is of interest to note that only triclopyr was in the eluates, with the highest concentration of about 3.5 ppm in one or two of them (Figure 2), and that the LC_{50} values of triclopyr to aquatic animals, notably trout, bluegill, daphnia, are from 1 to 3 orders of magnitude higher (Weed Science Society of America, 1983; Gersich et al., 1984) than this peak concentration. Even if bare, pure quartz sand near a fish-bearing water was treated with an unrealistically high dose of EGBE, it would be difficult to predict disaster for fish and invertebrates, owing to leaching from the sand into an adjacent water body.

By contrast, no movement of the described residues with water through or within the soil profile occurred. We found residues only in each soil's top layer (Section 1) to which EGBE or triclopyr had been applied; but we could detect residues in neither Sections 2-4 (Table II) nor any of the 27 eluates from either column. This indicates very strongly that the residues of the applied EGBE and the applied triclopyr were held in the soil by sorptive forces that did not permit desorption by water and thus downward movement through the profile with water. Moreover, the soil's sorptive capacity was high enough for all residues

to be held at, or near, the point of application—an important finding, especially in view of the lower rates forecast for the use of Garlon in forest management.

Both clay mineral particles and organic matter, present in the soil at 8.3% and 34%, respectively, possess large specific surfaces that are responsible for sorption of herbicides (Bailey and White, 1970). Sorbed pesticides tend to resist displacement by water from organic soil matter, but not desorption from clay mineral (Harris, 1973; Oloffs, 1975). Factors preventing desorption from organic soil fractions are mainly the shielding from water of sorbed molecules, held by hydrophobic interactions inside nonpolar, hydrophobic areas and attractive forces that are stronger than those between the same sites in the soil and water, e.g. ionic bonds, or cooperative hydrogen bonding (Lehninger, 1982).

Another factor probably related to triclopyr's limited leaching in soil is the low soil pH (3.4). Triclopyr has a pK_a of 2.68 (Spencer, 1982), so that a significant proportion will not be ionized, especially since pH at the surfaces of soil particles is usually considered to be about 2 pH units below the pH of the soil in bulk solution.

The composition of the residues, in percent of the total found by analysis in triclopyr-treated soil, was 4% triclopyr, 81% TP, and 15% MTP; in EGBE-treated soil it was 5.5% triclopyr, 88% TP, and 6.5% MTP. The formation of large amounts of TP and MTP in this soil, but not in sand, suggests microbial degradation of triclopyr. Microorganisms are known to be involved in the degradation of pesticides near the colloidal surfaces of soil particles (Burns, 1978). The methylation of TP to MTP in soils by microorganisms was suggested by Roberts and Marshall (1978).

The average rainfall from August to December in the area from which the soil was obtained is 71 cm, for the period recorded for 52 and 7 years at two meteorological stations of the Government of Canada, one located approximately 24 km west, the other approximately 20 km east-southeast of the sampling site (Kendrew and Kerr, 1955). The water used for the experiments was equivalent, therefore, to the precipitation to be expected for the 5-month period following application in the field. In view of this and of our results, it may be concluded that Garlon used properly in forest management under conditions as described, is unlikely to be leached into water from a site of application.

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Registry No. EGBE, 64700-56-7; TP, 6515-38-4; MTP, 31557-34-3; triclopyr, 55335-06-3.

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