

An interesting study was conducted by Knyr and Sokolov²⁰⁵ on the preservation of soil samples. Storage of 2,4-D on soil samples in a solution containing 10% NaCl and 2% NaOH (both w/w) for 15 to 30 days produced recoveries of 86 to 95% compared with 7 to 67% for water alone. Air-dried samples were shown to retain only 52 to 92% after 30 days. Soil samples dried at 100 to 105°C lost 90% of their residues after 30 days. Samples stored in 3% toluene at pH 3 allowed 96.5% recovery. 2,4-Dichlorophenol degradation product was 99% retained by storage in propiolactone/pH 1 solution; 90% was recovered in 3% toluene at pH 11.

Vapor loss of 2,4-D from 2,4-D esters in soil has been also discussed by Freed and Witt.²⁰⁶ The "vaporization index" of MCPA, 2,4-D and 2,4,5-T free acids have been determined¹⁹¹ to be 1, i.e., <0.1 kg/ha/year is lost. Helling¹³⁶ also noted that methylation increased 2,4-D volatility in his soil TLC technique. The form which is transported to the surface determines the importance of volatility. If carried by water, the species is probably the anion. Thus, dust removal might be the chief atmospheric transport process for this species. If esters, free acid, or amides are the species, vaporization will be most important. Clearly, there are lacunae in our knowledge here.

D. Persistence

As remarked at the beginning of the section, the major mode of degradation of the phenoxy is biological, but detailed consideration of this topic will be deferred to Volume II. We shall now proceed to consider the overall persistence of the phenoxy in the soil system, keeping the importance of the biological contribution in mind.

Moisture conditions and the amount of organic matter radically affect persistence. On the one hand, increased moisture content enhances microbial activity which is mostly concentrated in the organic component of soils and which in turn stimulates degradation of the phenoxy. However, enhanced leaching and volatilization also occur. Plant root absorption is also stimulated. Increased organic matter also increases the adsorption of the phenoxy, enhancing contact with the microorganisms and stimulating degradation and adaptation. Clearly, a delicate balance prevails dependent on soil type, conditions, and the type of herbicide molecule involved.²⁰⁷ There is wide disagreement on just how persistent the phenoxy is in soils. Audus very early found¹¹⁷ that in general 2,4-D persisted for 2 to 4 weeks, MCPA persisted for 8 to 12 weeks, 2,4,5-T persisted for 40 to 80 weeks, and that a chlorine in the *para* ring position enhanced degradation. Thus, 4-CPA degraded in 12 days instead of 34 days, as observed for phenoxyacetic acid; 2-CPA and 3-CPA persisted 365 and 60 days, respectively; for the di- and tri-chloro substituted acids, the activating effect of the 4-substituent largely overcame the deactivating effect of other substituents. These figures were subject to the usual, notorious biological variability. For example, although the usual time for 2,4-D persistence was 2 to 4 weeks, detoxification varied from 14 to 94 days, depending on the soil. 2,4-D can also be degraded within a week if soils are suitably moist.¹²⁹ However, the MCPA sodium salt was still detectable in ground water and soil 11 months after its application to a Czech field, and in some spots, it persisted for 29 months.²⁰⁸ The propylene glycol butyl ether ester and the triethylamine salt of 2,4,5-T on application²⁰⁹ to the surface of a California Etowah silt-loam leached downwards, so that for 14 days residues were observed only at 0- to 1-cm soil depth. After 14 days, the herbicides had descended to the 1- to 15-cm zone, with none in the 0- to 1-cm band. The residues thereafter were degraded or leached over 7 months. On the other hand, 2,4-D butyl ester sprayed²¹⁰ in August in the Ukraine at 3 kg/ha on trees and shrubs caused no detectable residues in the 10- to 20-cm deep layer of soil. Also, 2,4-D isooctyl ester sprayed on 50-yd soil strips could not be detected²¹¹ after 2 weeks; 50% disappeared after 0.64 days. 2,4-D free acid was thought to be the first degrada-

tion product. 2,4,5-T esters (i-octyl) applied to Texas grasslands at 0.5 to 116 per acre produced²¹² initial upper soil residues of 943 to 5390 ppb which decreased to 0 to 6 ppb at 12 weeks. Degradation appears to be fast²¹³ on warm, irrigated soils under subtropical conditions. The half-lives of 2,4-D, 2,4,5-T, and MCPA on Prairie Oxbow chernozemic soils in laboratory experiments were²¹⁴ 20.5, 59.0, and 22.0 days (50 $\mu\text{g}/20\text{ g}$ soil under field moisture capacity). These half-lives decreased with increasing soil moisture content. In other soils, $t_{1/2}$ of 2,4-D ranged from 10.8 to 31.4 days, and in the same given soil for MCPA and 2,4,5-T, the values were twice those obtained on Oxbow soils. The initial cleavage of 2,4-D appeared to be at the ether link, and no decarboxylation was noted. However, similar experiments using South Vietnamese soils incubated in the laboratory showed²¹⁵ that 74.5 to 95.4% of applied ^{14}C -carboxyl-2,4,5-T (15 ppm) produced $^{14}\text{CO}_2$ with 13.3 to 33.9% remaining in the soil over 168 days. Only 2 to 14% of the soil residues could be recovered on hydrolysis. In applications for oak-brush control, it was found²¹⁶ that the soil $t_{1/2}$ of 2,4-D, dichlorprop, Silvex, and 2,4,5-T were 4, 10, 17, and 20 days, respectively, using three soil types. Degradation apparently was not affected by soil type or vegetative cover. Persistence values of 50 to 60 days were obtained²¹⁷ in a Polish experiment for 2,4-D and dichlorprop. No phytotoxic residues were noted after harvest except for mecoprop (0.1 to 0.2 ppm). 2,4,5-T (2.24 kg/ha) was mostly found²¹⁸ in the 0- to 7.5-cm layer of soil, with very little downslope movement on application to some U.S. soils; 90% disappeared in 15 days. Wilson¹⁴⁰ noted that 1% of the applied i-octyl ester and dimethyl amine salt of 2,4-D persisted in a Washington soil and 0.25% in runoff. At application rates of 0.8 to 1.1 kg/ha for the amine and 8.0 to 11.2 kg/ha for the ester, both were degraded at similar speeds or the first 45 days. After 6 months, soils contained ~ 0.03 ppm 2,4-D. The phytotoxicity of applied 2,4-D was lost²¹⁹ after 5 weeks in another study. In another, 2,4-D was shown²²⁰ to leach as deeply as 90 cm and to degrade quickly under aerobic, but not anaerobic conditions. Type of salt formulation can also influence persistence. An application of 2,4-D sodium salt + B_2O_3 was found²²¹ to be more persistent than 2,4-D dimethylamine salt + B_2O_3 . It is apparent that persistence in soils cannot be predicted with any great certainty. The major reason for this is almost certainly the fact that since breakdown is mainly microbiological in soils, the degradation is a function of the microbiota which in turn is a function of the organic matter content. The situation is further complicated by the finding that this breakdown is accelerated by repeated applications of the phenoxys.^{117, 229}

The microbial population adapts to the more accessible provided carbon source. Under such conditions, it is almost impossible to provide a predictable persistence time without detailed knowledge of the type and history of the soil and microbiota involved. Thus, Kearney et al. in their 1969 review adjudged 2,4,5-T, 2,4-D, and MCPA to be persistent in general only for 5, 1, and 3 months, respectively. However, Edwards in his 1975 review²²² asserted that 1.25 years was the "safe" time to expect 95% disappearance of most phenoxys, remembering that 2,4,5-T derivatives are five to ten times more persistent under the same conditions than are the corresponding 2,4-D compounds. Clearly, more interdisciplinary-type studies are required in this area.

Many reviews are available on the general topic of persistence of pesticides in soils.^{163, 168, 169, 174, 175, 198, 222-231} The reader is directed especially to the biography list of Reference 225 for the phenoxy residue information. Decontamination of soils is treated in Reference 230.

IV. RESIDUES IN AQUEOUS SYSTEMS

A. Introduction

The loss of the phenoxys to runoff water is influenced by all the factors discussed

in the previous two sections, as well as cultural practice,^{232,233} the nature and rate of application, the slope of the field,²³² rainfall intensity, storm duration, type of formulation (e.g., 2,4-D ester formulations appear to wash off more readily than the amine salts though most remain in the surface 3 in. of soil),¹⁷⁷ microbiological activity in soils,^{229,231,232} presence of surfactants,¹³⁶ organic matter (activated charcoal is often used to protect moderately and overly sensitive plants from injury,²³⁴⁻²³⁶ as well as to prevent leaching), and the geological structure of the terrain (water table disposition, type of rock, etc.). Bailey et al.²²⁸ have presented a model to predict pesticide runoff. Other large pesticide inputs may arise from applications for aquatic weed control (see Volume II), industrial wastes, accidental spills, airdrift, dead animals, animal excreta, food chain, etc. Runoff is considered the major source,^{237,238} although industrial discharges, deliberate or accidental or directly or via sewer systems, are also important.²³⁹

Persistence in aquatic systems^{225,240,241} depends on the water type, organic particulate matter, rain, sunlight (see Chapter 3), temperature, microbial degradation, volatilization, and oxygen content of the water. Accumulation in bottom sediments may also be a factor,^{242,243} but in general, not for the phenoxys. Microbial activity is the major means for detoxification of the phenoxys in soils, but is relatively unimportant in natural waters, but dominates in bottom mud sediments and in sludge.^{244,245}

The ionic strengths of 50% of the surface waters of the U.S. are less than 0.01 *M*, and 97% are less than 0.05 *M*.²⁴⁶ The maximum ion strength of the water supply of 100 large cities in the U.S. was calculated²⁴⁷ to be less than 0.01 *M* (compare to ~0.7 *M* for sea water). Estuarine water will have a strength between 0.01 to 0.7 *M*. To make liquid/liquid extractions from such diverse sources comparable, the ionic strengths of these waters must be adjusted before analysis, and this is done either by adding saturating salt or excess acid. The presence of particulate matter will also influence quantitation (see Chapter 1). Particulate matter has been defined as "dissolved", "semicolloidal", and "colloidal" if the diameters are <0.001, 0.001 to 1, and 1 to 100 μm , respectively. Material passing through a 0.45- μm diameter millipore filter has been classified²⁴⁸ as "soluble". Clearly, adsorption or desorption of pesticides on or from semicolloidal matter may still occur, e.g., 2,4,5-T is sorbed on humic acid colloid.²⁵⁰ Natural surface waters range from clear spring, to black swamp containing much dissolved organics, and to reddish-colored river water with high amounts of iron. The pH can vary from 3.0 to 9.6.²⁵¹⁻²⁵³ The colloidal and suspended particles originate from municipal, industrial, and agricultural wastes, but soil erosion, especially from land without vegetation or tree cover,²⁵⁴ is the dominant source. Hence, this should be reflected in the chemical composition of the particles in the small rivers of areas of characteristic geology. Thus, kaolinite and vermiculite dominate in the acid soils of the southeast U.S.; illite and other clay-sized micas prevail in the Northeast and Midwest; montmorillonite is common in the neutral and alkaline soils of the Midwest and Western states.²⁵⁵ In the lower reaches of the large river systems, all particulate types are expected. In general, the muddier the river and the finer the silt load, the more likely is adsorption of pesticides going to occur, with an apparent decrease of residues in solution.

Petroleum spills have been reported in rivers,²⁴⁹ but partitioning into surface oils is more important for chlorinated hydrocarbons than for the phenoxys unless they are just freshly sprayed.

B. Fate of the Phenoxys in Aqueous Solution: Nonbiological Processes

In Chapter 3, we saw how 2,4-D esters hydrolyzed quickly at pHs > 4 to form anions (pH > 4) and free acid (pH < 4). We also noted how the photochemistry of the anion and the esters in aqueous solution is very different from that in organic solvents.

Zepp et al.²⁵⁶ showed that the disappearance of 2,4-D butoxyethanol ester from the Withlacoochee River in southern Georgia at pH 8.1 matched that observed in distilled water of the same pH. The rates of hydrolysis were much slower at pH values 4 to 8. Thus, anion formation is the first event in basic waters. In acidic waters, the structure of the ester determines the preferred dissipation mode. Photodegradation is the most important process for the butoxyethanol ester. Vaporization is most important for the butyl, octyl, and methyl esters. Photodegradation is also important for the methyl ester. There was a concentration dependence of the photolysis products. At concentration <1 ppm, 2,4-dichlorophenol and hydroxylation-dechlorination species were produced; at >300 ppm, the esters formed emulsions and reductive dechlorination occurred (see Chapter 3). Since 2,4-D anion also undergoes hydroxylative-dechlorination,²⁵⁷ this is expected to be the major photochemical fate of the phenoxys in aquatic environments at the concentrations usually encountered. The UV-B region of sunlight determines the rate of photolysis in surface waters. There are dramatic differences in intensity between summer (maximum) and winter (minimum) in the middle latitudes.^{258,259} Intensities also depend on time of day, reaching their maximum values when the sun is at its zenith.²⁶⁰ The calculated photolysis rate for the 2,4-D butoxyethanol ester is greatest between 9 a.m. to 2:30 p.m. on a typical clear day in the southern U.S. (see Figure 1A). The photolysis half-life of 2,4-D butoxyethyl ester is also dependent on season and distance away from the equator (see Figure 1B). These curves are based upon average intensities derived from the sun and the whole sky on cloudless days at sea level. Clouds decrease the intensity of UV-B radiation up to 50% for overcast conditions.²⁶¹ However, UV-B intensity increases 15 to 20%/km increase in elevation.²⁵⁹ The above estimations are valid only near the water surface in the absence of microorganisms. If 2,4-D butoxyl ethyl ester is mixed completely in pure water, the expected degradation with change in depth at various latitudes is shown in Figure 1C. However, the other absorbing species in natural waters will decrease the light intensity. On the other hand, photosensitization may also occur, and much work needs to be done to gauge if the latter process is important. Zepp et al.²⁵⁶ found that sterilized river water of pH 4.7 degraded 1 ppm of butoxy ethyl 2,4-D ester faster than a similar irradiation in pure distilled water at the same pH and suggested photosensitization as the cause.

A comparison of hydrolysis, direct photolysis, and vaporization dissipation at 25°C for various esters of 2,4-D is shown in Table 4. It is interesting that Struif et al.²⁶² also found that 2,4-D and 2,4,5-T esters hydrolyzed faster in basic solution than in acid, and at pH 7, the half-life was 5 to 30 days at 20°C for most esters; at 0°C, it was about 30 to 180 days. In addition, the adsorption of 2,4-D and 2,4,5-T onto flocculated humic acid was low at pH 7, but the esters adsorbed more strongly, especially as the length of side chain increased. Furthermore, the adsorption enhanced the rate of hydrolysis of the esters. In contrast, Shcherbakov and Poluboyarinova²⁶³ found that the free 2,4-D was produced from the butyl ester after 9 to 10 days in natural waters. Butler et al.²⁶⁴ also obtained similar findings to Zepp et al.²⁵⁶ Truchelut and Williams²⁶⁵ demonstrated that Silvex and 2,4-D persisted equally long in basic waters. Silvex was more persistent and hence more effective than 2,4-D in neutral waters. Hattula and Raisanen²⁷¹ observed that degradation of MCPA at 0°C in oxygen-free water was faster at pH 5 than at pH 6 or 7.

C. Biodegradation of the Phenoxys

We will not consider here the responses of cultures of defined species of microorganisms (see Volume II); we shall focus here on the responses of undefined mixed cultures.

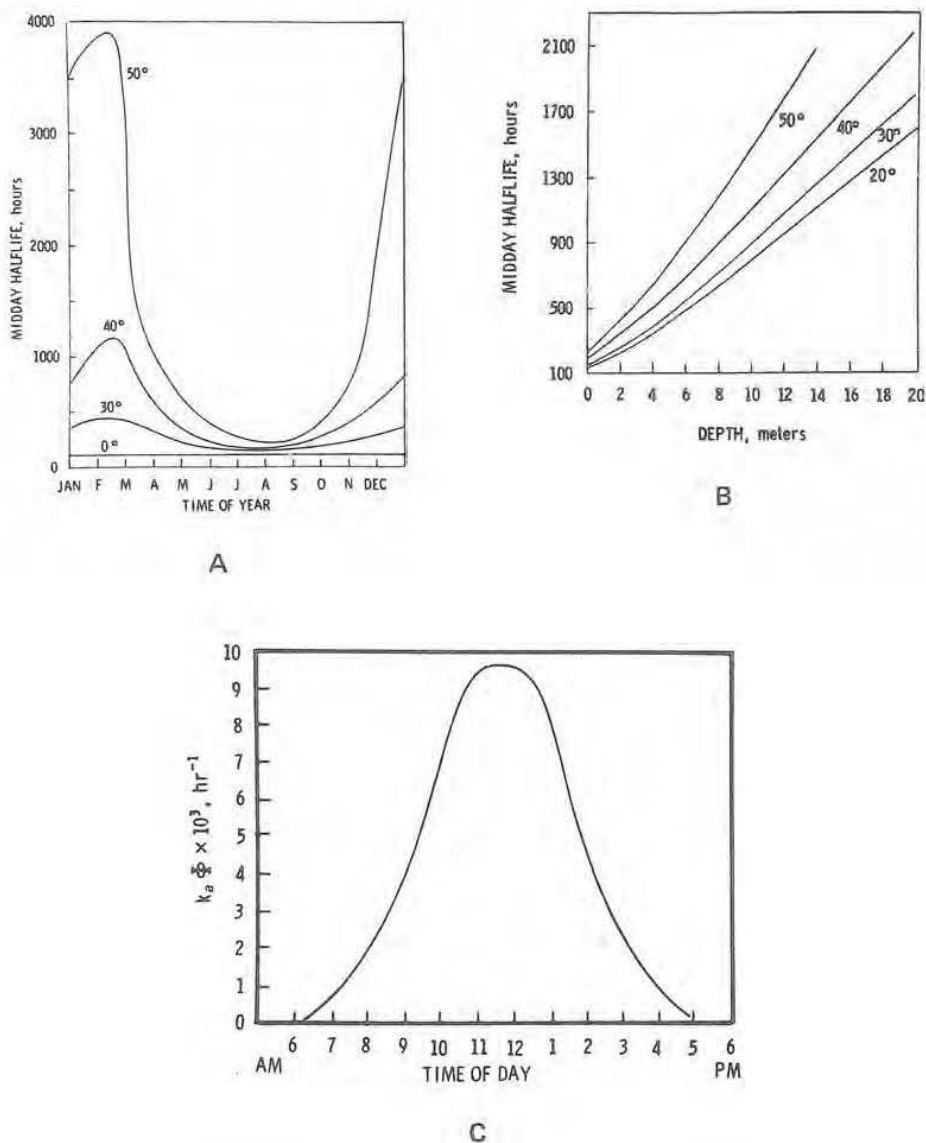


FIGURE 1. Dependence of 2,4-D butoxyethyl ester.²⁵⁶ (A) Photolysis rate upon time of day in the southern U.S.; (B) photolysis half-life upon season and northern latitude; and (C) photolysis half-life at several northern latitudes in completely mixed pure water (depth dependence). (Reprinted with permission from Zepp, R. G., Wolfe, N. L., Gordon, J. A., and Baughman, G. L., *Environment. Sci. Technol.*, 9, 1144, 1975. Copyright by the American Chemical Society.)

Okey and Bogan in 1962,²⁶⁶ using an activated sludge system, found that 2,4-D was quickly degraded. Cultures adapted to degrade 2,4-D could not degrade 2,4,5-T. Aly and Faust,¹⁴⁸ using bottom mud sediments collected from two similar lakes, one which had been treated with 2,4-D, reported that added 2,4-D disappeared in 35 days from

Table 4
COMPARISON OF HYDROLYSIS, PHOTOLYSIS,
AND VAPORIZATION HALF-LIVES FOR
SEVERAL 2,4-D ESTERS AT 25°C^{a,256}

Ester	Hydrolysis (days)		Direct photolysis ^{b,c,d} (days)	Vaporization ^d (days)
	pH 9	pH 6		
Methyl	0.04	44	29	21.7
1-Butyl	0.2	220	—	1.1
1-Octyl	0.2	220	—	11.5
2-Butoxyethyl	0.02	26	16	895

^a For water body 1-m deep.

^b For September in southern U.S. (latitude 34° N); average value for 1-m deep pure water.

^c Expressed in terms of 12-hr days.

^d Calculated for a completely mixed water body.

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the mud of the lake which had been pretreated, but it persisted for 65 days in mud from the untreated lake. Biodegradation was much slower than in soils. Butyl esters were degraded to 2,4-D and *n*-butanol within 9 days. In lake water, 2,4-D persisted up to 120 days. Fully adapted microorganisms could metabolize 2,4-D in 24 hr. Schwartz²⁶⁷ in 1967 found only a small fraction of 2,4-D was degraded by a mixed microbial population in dilute mineral salts. DeMarco et al.²⁶⁸ in 1967 observed that 2,4-D could be biodegraded within 6 days in warm aerobic waters but persisted as long as 80 days in cold deoxygenated waters. Hemmett²⁶⁹ and Hemmett and Faust²⁴⁴ studied the aerobic, microbial degradation of the phenoxys using domestic activated sewage sludge as the source of microorganisms. The zero-order rate constants of 2,4-D degradation were dependent on the number and age of the microorganisms, 2,4-D concentration, and length of adaptation time. It was also discovered that 68% of the 2,4-D was used in the respiration process, whereas 32% was incorporated into the cells as a reserve energy source. Hemmett,²⁶⁹ using free acids and dimethyl amine salts, found that free 2,4-D was degraded in 11 weeks, and its dimethyl amine salt was degraded in 8 weeks; 2,4-dichlorophenol, 4-CPA, and the isopropyl and the butyl esters of 2,4-D were also degraded. However, 3,4-D, 2,4,5-T, 2-(2,4,5-TP), 2-CPA, MCPA, and the isooctyl ester of 2,4-D, as well as 3,4-dichlorophenol, 2-methyl-4-chlorophenol, and 3-methyl-4-chlorophenol, were not attacked. At low concentrations of 2,4-D, the first-order kinetics were obeyed, but zero-order kinetics were observed at microorganisms/substrate (w/w) >10. The rate of degradation of 2,4-D was increased by an order of magnitude when the temperature was increased from 10 to 25°C. The butoxy ethanol ester of 2,4-D was removed²⁶⁴ gradually after 2 weeks when the ester was incubated with planktonic algae. Photolysis and hydrolysis also occurred. Robson²⁷⁰ reported that microbes adapted to MCPA also degraded 2,4-D in apparent contradiction to the results of Hemmett.²⁶⁹ Watson²⁷² in 1977 found that increased suspended matter in winter in natural waters accelerated the in vitro decomposition of added 2,4-D sodium salt. Decomposition also began earlier in mud samples than in water. Investigations by the U S. Environmental Protection Agency (EPA) have shown that 2,4-D in water can be degraded microbiologically within 1 week under favorable conditions.²⁷³

D. Runoff of the Phenoxy in Water

The first reports²⁷⁴ of extensive 2,4-D pollution in water came in 1945, when a company disposed of 2,4-D and 2,4-dichlorophenol into the local sewage system. Within 17 days, phenolic tastes and odors were reported downstream in shallow wells, and they persisted for 3 years. In Colorado at the Rocky Mountain Arsenal, 2,4-D wastes were discharged²⁷⁵ into lagoons from 1943 to 1957. Ground-water contamination was first reported in 1951 when crops were damaged by irrigation well water. It took 7 to 8 years for the contaminant to migrate 3.5 mi and eventually to affect an area of 6.5 mi.²

The statement of Westlake and Gunther²⁷⁶ in 1966 still holds true: "While we know a great deal about the fates of many registered pesticide chemicals, we know very little about the natures and fates of the many products in the immense tonnages of wastes in the dumps of pesticide manufacturers. These compounds will slowly leach from already existing "dumps" into our waters and soils for hundreds of years."

In 1961, Winston and Ritty²⁷⁷ predicted that the phenoxy were not likely to be a major pollution hazard in watersheds, since bacterial action was supposed to degrade them within a few days. Some of the phenoxy have been shown to persist for over a year, however.²⁷⁸ Low levels in drainage water have been detected immediately after spraying.²⁷⁹⁻²⁸² For example, very small quantities of 2,4-D were found²⁸³ in irrigation water 20 to 25 mi downstream from the point of application to soil at 1.9 to 3.0 lb/acre. The maximum concentration of 2,4-D found in the water was 25 to 61 ppm, and this was reduced to traces within 30 to 60 min. Most of the pesticide front had been swept downstream and the ensuing slow leaching process from soil ensured great dilution by the water. Adjacent streams running near a forest sprayed by helicopter with the dimethylamine salt of 2,4-D showed levels up to 7 ppb during the spraying.²⁸⁴ The propylene glycol butyl ether ester of Silvex (8 lb/acre) applied directly to floating alligator weed (*Alternanthera philoxeroides*) produced maximum residues of 1.6 ppm. Similarly, applied dimethyl amine salts of 2,4-D and Silvex (4 lb/acre) on alligator weed and water hyacinth (*Eichornia crassipes*) caused maximum residue levels between 0.65 to 1.0 ppm. The maximum concentrations did not appear immediately, but occurred one to 2 weeks after treatment, probably because of the absorption of the herbicides and their subsequent release by plants.²⁸⁴ 2,4-D and Silvex²⁸⁵ are often applied as liquid emulsions or granules to control submerged aquatic weeds (1.5- to 3.6-ppm initial concentrations in 4 ft of water). A 1.33-ppm application of 2,4-D granules produced²⁸⁶ a maximum level of 0.067 ppm in water 18 days after treatment. Even as early as 1965, approximately 100,000 lb 2,4-D granules were applied²⁸⁷ to lakes in the Tennessee River system. Low levels have also been found in water following use of granules.²⁸⁸ Residues have also been found after spraying for ditchbank vegetation control and usually ranged from a few to 100 ppb.^{289,290} In more modern studies, the levels of 2,4-D in the Nakwasina River watershed in southeast Alaska after spraying of 400 acres of cutover forest with the butyl ester of 2,4-D (2 lb/acre) were²⁹¹ far below the lethal doses to fishes and aquatic invertebrates. Low 2,4-D and 2,4,5-T levels in irrigation waters were noted²⁹² in 1973 by a publication of the U.S. National Information Service. However, Synek et al.²⁹³ in a Czech study could detect MCPA sodium salt 11 months after application in soil and ground water, and in some places it could be detected for up to as long as 29 months. Phenols and enhanced chloride ion were also noted. Dodecyl/tetradecyl amine salts of 2,4-D (4.48 kg/ha) were sprayed to control water hyacinth over 7000 acres along the Hillsboro perimeter canal in the Loxahatchee National Wildlife Refuge, Florida in 1971.³¹⁸ The highest 2,4-D levels in water and hydrosol were 37 ppb and 5 µg/kg at day 1 and days 3 to 15 after treatment, respectively. Of 60 fish samples, 3 had residues >10 µg/kg, and 16 had residues of 10 µg/kg.

Breast muscle and liver of Florida gallinules (*Gallinula chloropus*) had residues ~ 0.30 and 0.675 mg/kg, respectively, 1 day after treatment. After 4 days, no residues in these tissues could be detected. There was no effect on the egg hatching or fledgling development in boat-tailed grackle (*Cassidix mexicanus*). Perhaps the most extensive study on the ecological consequences of 2,4-D and 2,4,5-T was carried out by continuous application²⁹³⁻²⁹⁵ of massive quantities of the mixed butyl esters of 2,4-D/2,4,5-T (4950 kg/ha) to a square mile area of Eglin Air Force Base Reservation, Florida, over 8 years, 1962 to 1970; 2,3,7,8-tetrachloro-dibenzo-*p*-dioxin impurities contained in sprayed 2,4,5-T were found, even though this was 6 to 10 years after the last spraying of 2,4,5-T and residues were found in soils, animals, insects, and birds, despite the warm climate and an annual rainfall of 1550 mm. The top soil contained 337 ng/kg dioxin, 5% of the calculated applied dioxin. Herbicide residues were low. In another U.S. study, 2,4-D alkanolamine salts and Silvex propylene glycol butyl ether ester applied²⁹⁶ at 5.51 ppm in furrow irrigation water caused injury to sugar beets and soybeans at 1.1 ppm. On sprinkler irrigated plots, 0.22 ppm decreased soybean yield. Sprinkler irrigation of corn containing 1.1 ppm Silvex left a 10-ppb residue on foliage. Furrow and sprinkler irrigation with 2.21 ppm 2,4-D left a residue of ≤ 10 ppb in sugar beet roots. Tharaldsen²⁹⁷ reported that water samples from a Norwegian stream flowing through bushy vegetation sprayed aerially with 2,4,5-T butyl glycol ester (2,4,5-T "Bayer") contained ≤ 3 ppb of the ester, 1 to 44 hr after spraying. In a 1971 study,²⁹⁸ nine ponds in Florida, Georgia, and Missouri were treated with 2.24 to 8.96 kg/ha 2,4-D dimethylamine salt. Initially at the first two locations, residues were 0.345 to 0.692 mg/l and declined to ≤ 5 μ g/l after 28 days. In the Missouri ponds, the initial levels were ~ 0.6 mg/l and declined to < 5 μ g/l after 56 days. Residues in mud were never < 50 μ g/kg over 56 days. The highest level in mud was 170 μ g/kg (first and third days). Residues in the mud of one Missouri pond could be detected for 28 days after treatment. Of 307 fish samples, 45 contained residues 10 μ g/kg to 1 mg/kg; however, no fish were killed. 2,4,5-T was detected in runoff water on Texas rangeland²⁹⁹ after spraying of 1:1 2,4,5-T/picloram triethylamine salts. This was mostly because of plant wash-off. Herbicide content in Houston black clay soil was low (0 to 238 ppb) for at least 2 years. There were high concentrations on grass (50 to 70 ppm) immediately after treatment, but these decreased rapidly. High concentrations of 400 to 800 ppb were found in water if heavy rains fell just after treatment, but were low (< 5 ppb) if no major storms occurred for 1 month after treatment. No damage to cotton or sorghum in adjacent fields was observed. In contrast, less than 1 ppb of 2,4,5-T could be detected³⁰⁰ when 1:1 2,4,5-T/picloram (1.12 to 2.24 kg/ha) was applied every 6 months for 3 years. The initial residues on grass were ~ 28 to 113 ppb, but these declined quickly. No 2,4,5-T residues were detected in a field lysimeter sampled for year after treatment. Soderquist and Crosby³⁰¹ also monitored MCPA residues in a California rice field. Water residues declined to 10 ppb within 14 days; plant residues, beginning at 20 ppm, decreased to 1 ppm in 20 days. Microbial degradation was postulated as the prime degradative mode. Davis, in a Ph.D. thesis,³⁰⁹ investigated the disappearance of the propylene glycol butyl ether ester and the triethylamine salt of 2,4,5-T from field plots and lysimeters. The mean concentration of 2,4,5-T in impounded water was ~ 40 ppb, with negligible amounts in fresh runoff. The ester persisted longer in impounded water than in field plot soil. No residues were found in water 7 months after application. Residues were detected in the lysimeter 1 to 16 days after spraying. No residues on the soil surface were found after 90 days. In a Russian study,³⁰² it was found that 2,4-D dimethyl amine salt (2.4 to 3.6 kg/ha) persisted 20 days in water and 30 days in bottom sediments after application. In another Russian investigation,²⁶³ although the 2,4-D esters appeared to be hydrolyzed to 2,4-D in 9 to

10 days, the ester appeared to accumulate in fish at levels 100 to 300 times higher than in water. They recommended the level of $6 \mu\text{g}/\text{l}$ for fish ponds. The largest loss of 2,4-D (0.56 kg/ha) in runoff from plots receiving simulated rainfall and from a small agricultural watershed on a sandy U.S. coastal plain soil in 1970, 1971, and 1972 was³⁰³ 8.1, 6.2, and $2.5 \mu\text{g}/\text{l}$, over 20, 27, and 34 days, respectively. Residues in the top 0.5 cm of soil decreased from 4.7 to 0.27 ppm in 7 days and to 10 ppb after 34 days. Soil subsurface concentrations were always $<1 \mu\text{g}/\text{l}$. When rains were simulated at day 1, 8, and 35 after application, runoff was 25.2, 5.8, and $0.7 \mu\text{g}/\text{l}$, respectively. These researchers published another study³⁰⁴ on surface runoff of 2,4-D (0.56 kg/ha) from corn (*Zea mays*) plots down a 24.4-m grassed waterway. When soils were moist, the loss of 2,4-D was 2.5% when it rained (simulated) 1 day after application; if the soils were dry, the loss was 10.3%; 30% of the applied 2,4-D reached the end of the runway from the corn plot, regardless of moisture. In another U.S. study, 2,4-D dimethylamine salt (1.5 ppm) concentrations were³⁰⁵ 139 ppb on day 1, but were 4 ppb on day 41. Silvex PGBE ester (2.9 ppm) levels were²⁸⁵ 1.6 ppm for 7 days, declining to 20 ppb at day 182. In a recent study,⁴⁴⁰ Joyce and Sikka found levels of 2,4-D to be well below tolerance in water and blue crabs (*Callinectes sapidus*) after routine water hyacinth (*Eichornia crassipes*) control in the St. Johns River in Florida.

E. Residues of the Phenoxy in Water

From the preceding section, it is clear that persistence in waters is as variable as in soils in spite of the much greater dilution.

Most measured phenoxy residues in streams and lakes are in the parts per billion range.³⁰⁶ Hindin et al.^{307,308} detected 2,4-D esters in late 1960 in the irrigation supply water of the rainshadow area of the Columbia Basin in Washington. Values ranged from 0.3 to 18 ppb; 2,4-D and 2,4,5-T were noted in Lake Hopatcong, N.J. in 1957 to 1958,³¹⁹ and 2,4,5-T was noted in Dutchess County, N.Y. in 1958 to 1960,³²⁰ as well as 2,4-D in Colorado in 1959³²¹⁻³²³ and in 1963.³⁰⁹ Residues <1 ppb were also detected in the western U.S.;³¹⁰ residues of 2,4,5-T and Silvex³²⁴ are rarely found.³¹¹ In spite of these low levels, concentrations as high as 70 ppb of 2,4-D³¹² have been detected after aerial spraying of Oregon forests, and 50-ppb levels were measured³¹³ at Lafayette, Ind.; levels between 0.01 and 0.07 ppb of 2,4,5-T in surface waters were also noted in 1971;³¹⁴ Silvex residues in surface waters and reticulated town waters from 15 western states can range between 0.01 and 0.21 ppb.³¹⁵ Consequently, the "rarity" of residues may in fact reflect the desultory nature of the monitoring.

In Canada, Simpkins and Peake³¹⁶ analyzed some 260 samples of natural waters from the Prairie provinces of Alberta and Saskatchewan in 1972 and found quantities of 2,4-D to be greater than 1 ppb in over half the samples, the highest value being 31 ppb; 2,4,5-T was found in 85 samples at levels >1 ppb; 2,4-DP was not commonly found, but concentration up to 7 ppb were detected. Lee Choi et al.³¹⁷ in 1976 reported on 1973 2,4-D levels in the South Saskatchewan River near Saskatoon, Saskatchewan. Levels in mid-June were around 2 ppb. No 2,4-D was detected at harvest time or in river mud. In both Canadian studies, total phenoxy content was evaluated and confirmed by GC/MS. The figures by Lee Choi et al. were also confirmed by transesterification. In the latter study, many neutral compounds were detected having the same retention times as the *n*-butyl and *i*-butyl esters of 2,4-D, but most of these peaks proved spurious on clean-up.

Adsorbed Silvex appears to be difficult to extract from organic matter³²⁵ and may explain why its residues are comparatively rare in addition to its smaller volume of usage.

All the studies above have reported levels as if all the residues were derived from

homogeneous solutions. However, the samples may or may not have contained particulate matter. Suffet³²⁶ demonstrated that waters with particulate matter adsorbed herbicides. In fact, spiked isopropyl 2,4-D ester at ~ 4 ppm in natural water with a pH of 7.8 at 25°C and of 58 Jackson turbidity units (JTU) showed only 1 ppm on analysis, and this value declined to ~ 0.1 ppm after 1 day, with many adsorption and desorption cycles. Under *p*-value (See Chapter 1, Section IV.D) extraction conditions (pH 2.0, 0.2 *M* orthophosphate buffer), the initial value was even lower (~ 0.6 ppm), but although adsorption and desorption cycles again occurred, the mean value stayed around 0.6 ppm. This behavior indicates that many literature levels could be factors of 6 to 40 too low if the sample was initially heavily turbid. The best sampling technique is to filter the water at the time of collection and analyze the water and particulate matter separately, remembering that "water" still contains much semicolloidal material, even after filtration. If there is suspended matter in a sample, age and storage conditions are crucial. Clearly, analytical and methodological problems still await clarification in this area.

Although the phenoxys, when pure, are odorless, the corresponding chlorophenols, often the main biodegradation products, are not. The permissible level of 2,4-dichlorophenol is³²⁷ 0.5 ppb. Threshold odor and taste levels are reported to be 0.65 ppb at 30°C³²⁷ and 8 ppb,⁴⁴² respectively; however, 2,4-dichlorophenol appears to be biodegraded faster than 2,4-D under the same conditions. This does not mean to imply that the rate of biodegradation is always fast, e.g., the early fouling of water supplies by chlorophenols from 2,4-D which persisted for 3 years.²⁷⁴ This is the basis of the taste and odor³²⁹ thresholds for 2,4-D (0.12 ppm, 3 ppm) and for 2,4,5-T (14 ppb, 2.92 ppm), respectively. Silvex has an odor threshold of 0.78 ppm,³²⁹ and the threshold for the isopropyl ester of 2,4-D is between 3.1 to 55 ppb;³²⁹ the value for the isooctyl ester of 2,4-D is set at 0.12 ppm.

Removal of pesticides from water supplies has received much attention. It was demonstrated in Chapter 3 that the phenoxys are comparatively resistant to chemical degradation. Silvex is not degraded by HCl or NaOH,³³⁰ and neither chlorine nor KMnO_4 are effective in removing 2,4-D or its esters from surface waters.³³¹ Activated charcoal (see Chapter 1) appears to be the most efficient means,³³⁸ removing at least 80% of 2,4-D at 6-ppm levels,³³² although twice as much charcoal is required to reduce anion levels than esters for a 30-min contact time at 25°C. A 20- to 30-fold excess of activated carbon will cleanse out all phenoxyacetate anions, esters, and phenolates. A 2,4,5-T ester at 10 ppb was removed partially by chlorination (<10%).³³³ Ion-exchange methods proved to be ineffective, only 20 to 30% being removed at 10 to 120 ppm of sodium salt and *n*-butyl ester of 2,4-D.³³⁴ The ion-exchange technique of Renberg, however, appears promising.³³⁵ Reverse osmosis techniques remove only 65% of the 2,4-D sodium salt.³³⁶ Aly and Faust³³⁷ demonstrated that limestone, $\text{Al}_2(\text{SO}_4)_3$, and FeSO_4 were also ineffective to remove phenoxys from water.

In two interesting papers, Moffett et al.^{339,340} examined the effect of spraying bees and spiking their water supply with 2,4-D and 2,4,5-T. After spraying,³³⁹ no residues were detected in the bees or their honey, and no injury was caused. In another later investigation³⁴⁰ (1974), 2,4,5-T at 1000 ppm in the water source of honey bees caused residues of 148 ppm in the bee and 50 ppm in the honey after 12 weeks. After spraying, residues were both ~ 50 ppm; 2,4,5-T was still detectable 480 days after study initiation and could be still found in wax after 650 days. Thus, at these larger doses, 2,4,5-T appears to be persistent.

Tolerances for 2,4-D in potable or fisheries waters in the U.S. are now set at 0.1 ppm;³⁴¹⁻³⁴³ those for 2,4,5-T and 2,4,5-TP are³⁴² 10 ppb, being also dependent on maximum daily air temperatures. In Canada, the total amount of permitted phenoxy acids is 0.1 ppm³⁴⁴ in fishery and potable waters.

Estuarine and oceanic studies of phenoxy residues are scarce, even though 2,4-D is used extensively for control of weeds in tidal areas.^{345,346} In 1964, attempts were made to reclaim large areas of natural oyster beds in the Chesapeake Bay area of the U.S. The highest residues in clams and oysters occurred during the first few days after exposure, ranging in general from 1.45 to 3.00 ppm. No residues were found after 21 days at one location; in another, the residues persisted for 59 days.³⁴⁷ In other Chesapeake Bay studies,³⁴⁸ oysters and clams accumulated maximum 2,4-D residues of 3.8 and 3.6 ppm, respectively. Thus, although shellfish appear to accumulate the phenoxys, slow elimination does occur.

As far as the authors know, no residue study for the phenoxys in mid-ocean, e.g., the Gulf Stream or the Kuro Siwo, has ever been published or attempted.

The topic of herbicide residues in aquatic environments has been the subject of numerous reviews.^{224-226,229,276,284,350-358}

V. RESIDUES ON PLANTS AND CROPS

Herbicidal residues on plants are easily detectable just after spraying. Plants can also absorb herbicides via their roots from the air, "washout", contaminated waters, birds, animal excreta, and similar sources. The residue on the leaf is often a fine balance between leaf penetration (and translocation) and herbicide volatilization, photodecomposition, or wash-off. We have already discussed most of these processes in earlier sections of this chapter. We shall not provide an exhaustive list of residues, since this is adequately covered by other reviews.^{224-226,359} Instead we shall concentrate on the plant residues obtained in some representative studies in the 1970s.

Residues of 15 to 30 ppb of 2,4-D were found³⁶⁰ in potato tubers at harvest after application for enhancement of color and control of tuber size. Some residues were ≥ 110 ppb. At least 70% of the 2,4-D was in conjugated form. Bovey and Baur²¹² in 1972 showed that 2,4,5-T ester in Texas grassland ecosystems did not persist from year to year. Just after spraying (0.5 or 1 lb/acre), the residues on grass were 26 to 144 ppm. After 6 weeks, these had declined to 0.040 to 5.93 ppm; at 26 weeks, they were 0 to 51 ppb. Corn sprayed in Russia³⁶¹ with 2,4-D amine salt (0.5 mg/kg) still contained residues at the 15-leaf stage, but none were detected at harvest time.

A U.S. application²⁹⁶ of 1.1 ppm Silvex propylene glycol butyl ether ester applied via sprinkler to irrigated plots left a 10-ppb residue on corn foliage. The same level in sugar beet roots was also found on application of 2.21 ppm 2,4-D alkanolamine salts via furrow or sprinkler irrigation. Only parts per billion to parts per million levels of 2,4-D and 2,4,5-T were found³⁶² in and on various plants (asparagus, beans, beets, carrots, cole crops, cucumbers, melons, squash, lettuce, leeks, peas, peppers, potatoes, spinach, tomatoes, sweet potatoes, sweet corn, apples, blueberries, cherries, cranberries, grapes, and pears) in a survey dealing with 92 pesticides used in the Maryland, Massachusetts, New Jersey, New York, and Pennsylvania areas in 1972. No residues or metabolites of 2,4-D, MCPA, and MCPB were detected³⁶³ in grain from Russian winter rye treated at 0.6 to 1.4, 0.8 to 1.8, and 1.5 to 2.5 kg/ha, respectively. In India, it was found³⁶⁴ that groundnuts in the Punjab sprayed with 2,4-D at 0.6 to 3.6 kg/ha showed residues (≤ 1.40 ppm) up to 75 days after treatment, even though the residues decreased gradually with time. In a Russian study, 2,4-D butyl esters sprayed in August 1973, at 3 kg/ha on trees and shrubs, produced²¹⁰ residues in the litter layer under treated trees of 1.6 and 0.22 mg/kg at 1 and 10 days, respectively, after application. The levels in aspen, birch, mountain ash, raspberry, and red bilberry decreased 4.9-, 5.9-, 6.1-, 13.9-, and 3.4-fold respectively, during the first 10 days after application.

In other Russian studies, 2,4-D and 2,4-dichlorophenol were measured³⁶⁵ in straw, fodder, eggs, milk, meat products, and food at public caterers in the Saratov Region at levels >9.5- to 12-fold the maximum allowable concentration of 2,4-D. Chmil³⁶⁶ in 1975 reported 2,4-D and 2,4-DB levels of 10 $\mu\text{g}/\text{kg}$ from grain and potatoes. In another Saratov Region study, Fedorova et al.³⁶⁷ in 1975 showed that 46% of the cattle contained 2,4-D and its 2,4-dichlorophenol metabolite. In addition, small quantities were found in milk, root crops, straw, hay, and in the eggs of Japanese quail ($\sim 1\%$ of the injected herbicide was found in the yolk and albumin). Both were found in the liver and kidneys of quails. In a 1974 Finnish study, cowberries (*Vaccinium vitis-idaea*) and wild mushrooms growing in young conifer forests sprayed aerially with 2,4-D/2,4,5-T were shown by Mukula and Siltanen³⁶⁸ to contain 0.1 to 5.2 and 0.2 ppm, respectively, of 2,4-D and 0.7 to 15 and 0.15 to 1.8 ppm, respectively, of 2,4,5-T residues. Pleshakov and Serov³⁶⁹ could detect 2,4-D butyl ester (3 to 4 kg/ha) or dimethyl amine salt (4.8 to 7 kg/ha) sprayed on grass for 100 days after spraying. Possible transfer to food products because of feeding of animals was proposed. Anpalov and Mostovi³⁷⁰ in 1976 noted that the residues of the ammonium salt of 2,4-D (1.2 kg/ha) disappeared within 40 days, and fodder residues were halved by fertilization with ammonium nitrate (75 kg N per hectare). Bristol,³⁷¹ in a 1976 U.S. National Bureau of Standards study, showed that red potatoes from North Dakota treated with 2,4-D (2 oz/acre) at tuber set to improve market quality and yield contained 68 to 150 ppb 2,4-D and <2 ppb 2,4-dichlorophenol. When stored at 4°C over 15 months, the levels were <5 ppb, but the 2,4-dichlorophenol levels remained constant. The $t_{1/2}$ of 2,4-D under these conditions was ~ 10 to 14 weeks. In contrast, tuber samples sliced, fortified, processed immediately after harvest, and stored at -20°C contained constant levels of 2,4-D over 15 months, but this time, 2,4-dichlorophenol concentration decreased from 88 to 47% in this period. It was concluded that kinetic studies were required to prove residue dissipation.

In two Oceania studies, Love and Donnelly³⁷² in 1976 showed that apricots grown in Central Otago (New Zealand) on trees sprayed with 2,4-D to control fruit drop still contained 2,4-D for up to 7 weeks. The levels were <0.7 mg/kg at normal harvest. Singh et al.³⁷³ in a 1977 Australian study studied the fate of 2,4-D in lemons which are usually sprayed to reduce losses in storage from stem rot caused by *Diaporthe citri*. The herbicide was applied in various forms at packing time via wax (100 to 500 mg Na-2,4-D/l) or as a double treatment in a benomyl fungicide bulk dip followed by packing line treatment. Only doubly treated fruit contained 2,4-D after 8 weeks, and Na salt < dimethyl amine salt < ethyl ester. Residues declined in storage, but could still be detected after 12 weeks, except for fruit given a single treatment with the sodium salt.

In the U.S., only 50 samples of food were found to be contaminated with 2,4-D (21 to 160 ppm) between 1964 and 1970. Residues were found in 1% or less of dairy products, oils, fats, shortening, and in fruits, 1.9% of leafy vegetables, and in 22.1% of sugar and adjuncts.²⁷³ Very little 2,4,5-T was found³⁷⁴ in food in analyses of raw agricultural products and in the Market Basket survey (1965 to 1968) samples. Of about 10,000 food and feed samples examined from 1964 to 1969, only 25 contained traces of 2,4,5-T (<100 ppb) and 2 contained measurable amounts (0.19 and 0.29 ppm). In the FDA Market Basket Survey, Silvex³⁷⁵ and MCPA were detected at levels of ≤ 100 and ≤ 400 ppb, respectively.¹⁰ Similar levels were found in a Canadian study³⁷⁶ for 1972 to 1973.

The tolerances for 2,4-D in the U.S. in or on potatoes³⁷⁷ and in sugar cane molasses and bagasse³⁷⁸ are now set at 0.2 and 5 ppm, respectively. 2,4-D dimethylamine salt tolerances have been set³⁷⁹ at 0.1 ppm in citrus, cucurbits, forage grasses, forage legumes, fruiting vegetables, stone fruits, avocados, cotton seed, hops, and strawberries;

other tolerances are 0.5 ppm in grain, 20 ppm in forage, and 2 ppm in the milled fractions of barley, oats, rye, wheat (except flour), or feed. The acceptable daily intake set by the FAO/WHO is 300 $\mu\text{g}/\text{kg}$.³⁸⁰ Another FAO/WHO publication recommended tolerances of 20 ppb in grains.³⁸¹

We will not discuss residue and metabolite levels in microorganisms, birds, fish, man, or animals, since these will be considered in Volume II. An excellent book³⁸² is available on the general subject of pesticide residues, as well as the FAO/WHO bulletins^{380,381} and U.S. EPA Reports.^{273,313,374,375,443}

VI. 2,4,5-T AND TCDD POLLUTION

As has been stressed in Chapter 1, TCDD is a very teratogenic impurity found in formulations of 2,4,5-T, Silvex, 2,4,5-TB, pentachlorophenol, and any compounds which utilize tri-, tetra-, or penta-chlorophenols in the manufacturing processes. We will consider in detail in Volume II the toxicological properties of the dioxins. Several reviews and excellent books are now available. Instead, we shall concentrate here on dioxin contamination.

There are four well-known instances of significant environmental TCDD contamination namely, in South Vietnam in the 1960s, northwestern Florida (U.S.) in the 1960s, eastern Missouri (U.S.) (May, 1971), and in Seveso, northern Italy (July, 1976). All were unintentional and uncontrolled.

A. South Vietnam

Dioxin was dispersed aerially by the U.S. Air Force as a contaminant of Agent Orange, a 1:1 mixture of butyl esters of 2,4,5-T/2,4-D. Between 1961 and 1970, but primarily (89%) during the 4 years from 1966 to 1969, an estimated total of more than 110 kg of dioxin was applied³⁸³ unintentionally to approximately 10^6 ha of South Vietnam (i.e., 6% of its land area). The overall dose was thus 106 mg/ha; 66% of this sprayed area received 70 mg/ha, 22% received 140 mg/ha, 8% received 210 mg/ha, 3% received 280 mg/ha, and the remaining 1% area received >280 mg/ha. Most of the defoliation program (90%) was directed at forests (upland and coastal), and the remainder was directed at agricultural lands (primarily upland). The region around Saigon was the recipient of 53% of the spraying, but on an areal basis, this region received three times as much as the average. The approximate temporal distribution of the estimated 111 kg of dioxin was (in kilograms: 1961 (0), 1962 (0), 1963 (1), 1964 (2), 1965 (4), 1966 (16), 1967 (30), 1968 (22), 1969 (31), and 1970(5). The total population of South Vietnam at the time was approximately 17.6×10^6 . Thus, on a per capita basis, the average maximum dioxin exposure was 6 mg/person; around Saigon, the average was 25 mg/person. In terms of the top 10 cm of soil and assuming a weight of 1325 kg/m³ for normal soil, this represents 80 ng/kg. Dioxin was soon found to be concentrated in food chains leading to man by residue analysis of fish, shellfish, and crustacea collected in South Vietnam.³⁸⁴ In addition, during the height of spraying season in 1968 to 1970, the number of stillbirths at Tay Ninh Hospital, serving the most heavily sprayed area, was twice the rate in comparable regions or when there was no spraying. Saigon's Children's Hospital records showed trebled incidence of spina bifida and pura cleft palate during heavy spraying.³⁸⁵ These epidemiological data were confirmed independently by the U.S. Department of Defense^{386,387} for all sprayed areas. In spite of the suggestive nature of the above, it does not prove cause and effect. However, the segment of the population most likely to bear the brunt of the spraying was also the one most likely to be underrepresented in the above statistics.^{388,389}

The description of the train of events after aerial spraying is worthwhile to summa-

rize: leaves of trees yellowed, shriveled, and fell, and crops withered, rotted, and became inedible. Fish in ponds and rivers and small animals and fowls in farmyards died. Some abortions and monstrous births in animals were observed. Vomiting and nausea seemed to be only transitory. Chloracne was also reported. Fatigue, weariness, and dizziness were induced. Long illnesses were experienced by children and elderly people.³⁸⁸ Such events evoked swift reaction from scientists^{390,391} and the popular press. Thus, for example, many articles and letters in *Science* have appeared on the issue.^{388,390-397}

The Report by the National Academy of Sciences^{387,394} also predicted that the mangrove forests would take 100 years to recover and found after great controversy that between 0.5 to 2 million m³ of merchantable timber was lost, that the phenoxy herbicides in soils barely persisted beyond 1 year, that only one growing season was lost after spraying, and that spraying did not have a "lasting detrimental effect" on plant nutrients in soils.

The reports of the abortions and stillbirths in Vietnam came just before it was found³⁹⁸ in 1970 that 2,4,5-T, containing about 30 ppm TCDD, was teratogenic and fetocidal in two strains of mice and in one strain of rats. In 1963, following the thalidomide tragedy, the U.S. President's Advisory Committee recommended the initiation of a large scale screening program to determine the safety of pesticides and other industrial chemicals.³⁹⁹ In 1964, the National Cancer Institute awarded a contract to Bionetics Research Laboratory to conduct carcinogenicity, mutagenicity, and teratogenicity tests on many pesticides including 2,4,5-trichlorophenol and 2,4,5-T. Bionetics reported in the late 1960s that 2,4,5-T was teratogenic to mice.

Public disclosure that TCDD was the active teratogenic agent in a very widely used herbicide triggered a number of administrative and legal actions as well as intensified toxicological and environmental studies of 2,4,5-T and TCDD.⁴⁰⁰ Congressional hearings were initiated in April, 1970, on the "Effects of 2,4,5-T on Man and the Environment".⁴⁰¹ The U.S. Departments of Agriculture (USDA), Interior, and Health, Education and Welfare announced jointly on April 20, 1970 the suspension of home uses of 2,4,5-T as a liquid formulation and of all uses in and near water.⁴⁰² This was soon followed by the cancellation on May 1, 1970 of all registered uses of 2,4,5-T for certain food crops.⁴⁰³ Use of 2,4,5-T on range and pasture land, along rights-of-way, and in forest areas were not affected by the action. This was soon challenged by two manufacturers.⁴⁰⁴ An advisory committee appointed by the Secretary of Agriculture in a report dated May 7, 1971 recommended that 2,4,5-T be reinstated when tolerances in foods were established and that levels of TCDD be kept low. The newly formed EPA, the regulatory body for registering pesticides, declined to rescind the cancellation on the grounds that data on the effects of 2,4,5-T and TCDD did not warrant this action.⁴⁰⁵ In 1973, EPA gave public notice of its intent to complete environmental and human monitoring projects and to determine TCDD toxicity,⁴⁰⁶ as well as to hold a hearing on all uses of 2,4,5-T. In March 1975, it released a "Dioxin Implementation Plan"⁴⁰⁷ to sample the total environment for 2,4,5-T and Silvex. Results of the analyses of 85 beef fat samples and 43 beef liver samples carried out by several collaborating laboratories showed that three fat samples contained 20 to 60 ng/kg TCDD, but only one liver sample contained TCDD, near the limit of detection.⁴⁰⁸ The Council for Agricultural Science and Technology (CAST) took the opposite position: the amount of TCDD in 2,4,5-T and Silvex was not enough to change the toxicological properties of these preparations or to endanger human health or affect plants or animals in the environment. Unfortunately, synergistic interactions were not considered in this report.^{358,409} On August 11, 1978, the USDA prohibited spraying of 2,4,5-T within a quarter mile of streams on national forest lands. However, it amended this distance

to within 200 ft of streams in September, 1978, after intensive lobbying by forestry groups. Spraying within 1 mi of permanent dwellings was also prohibited.⁴¹⁰ A fuller account of the early stages of the 2,4,5-T controversy can be read in the article by Bevenue and Kawano.⁴¹¹ On February 28, 1979, the EPA suspended the use of 2,4,5-T and Silvex for use in forestry, rights-of-way, and, in the case of Silvex, for home and garden use, as well as other minor uses. This action was based on results from an epidemiological study of spontaneous abortions in the Alsea forestry region of Oregon, an area that has been continuously treated with these two herbicides.⁴⁴¹ An appeal by Dow Chemical against the banning was unsuccessful. At this time (January 1981) a decision on the complete ban on the use of these herbicides awaits the outcome of cancellation hearings being held in 1980-1981.

In 1970, the U.S. Air Force terminated its defoliation program in Southeast Asia and approximately 8.7×10^6 l of Agent Orange were stored at Gulfport, Miss. and on Johnston Island in the Pacific Ocean.^{293-295,393} Analyses for the TCDD contest produced figures ranging from 0.1 to 47 ppm, the average being around 1.86 ppm. The subsequent fate of Agent Orange has already been alluded to in Chapters 2 and 3.

B. Northwest Florida

At Eglin Air Force Base in northwestern Florida the U.S. Air Force carried out a spray equipment test program between 1962 and 1970.²⁹³⁻²⁹⁵ The area of particular interest is a formerly forested, unpopulated 37.25-ha grassland tract (Grid Number 1 of Test Area Number C-52A). Over a 3-year period 1962 to 1964, 39,540 kg of 2,4,5-T was sprayed. If the TCDD contamination was the same as in the formulations left over from the Vietnam War, about 180 g of dioxin was applied leading to a rate of 4.9 g/ha. In terms of the top 10 cm of normal soil, this represents 3.7 $\mu\text{g/kg}$. Dioxin has been found in all collected soil samples and in many animals, birds, fish, and insects in spite of the warm weather and heavy annual rainfall (1550 mm) and despite the fact it was 12 years after the last spraying. The top 10 cm of soil contained 337 ng/kg of dioxin, implying a half life of ~ 3 years. Two other field testing sites were also used, namely, western Kansas (Garden City) and northwestern Utah (Air Force Logistics Command Test Range Complex). Soil types varied from a sandy loam at Eglin, silt loam at Garden City, and a clay loam at the Air Force Logistics Command Complex. The mean rainfalls were 155, 40, and 15 cm, respectively. TCDD residues in these soils varied from 375 ppt to 15 ppb at the earliest time of analysis. TCDD appeared to degrade faster in Kansas soils than in Florida soils and degrade least rapidly in Utah desert soils. The ranking of the first two was a surprise, since it might be expected that biodegradation would be most rapid in the Florida climate. TCDD did not leach appreciably below 15 to 30 cm. Preliminary studies did indicate however, that added lime, fertilizer, and organic matter enhanced degradation under Florida conditions. Degradation was possibly also enhanced by tilling and by increases in temperature. No bioaccumulation was observed. The reports were subjected to critical scrutiny by Commoner and Scott,⁴¹² who concluded that the half life of TCDD in all three field sites was the same, namely, 190 to 330 days and that tillage, photodecomposition by sunlight, or addition of nutrients did not shorten persistence, at least for as long as 3 years. Their evaluation agrees with the finding that TCDD remains firmly bound to its original location in the soil and does not leach up or down with water. This implies the chief degradation occurs by soil microorganisms. Commoner and Scott also pooled compatible data, and they concluded that bioaccumulation and bioconcentration did occur, a conclusion diametrically opposite to that of Young. It was asserted that the Air Force data could not be properly used to prove this thesis, since the data were not based on a series of trophic food levels as determined from whole-

body analyses. There also appears to be a concentration dependence, the critical concentration in mouse liver being ≥ 500 ppt. It is also possible that natural selection for resistance took place over the many generations exposed. In addition, the technique used to obtain the analytical data was not clearly stated. The Air Force studies also note that TCDD is difficult to release from activated charcoal once it is bound.

C. Eastern Missouri

This incident occurred during May 1971⁴¹³⁻⁴¹⁵ when a large (68 m³) of waste oil, unwittingly contaminated with ~ 330 mg/kg of dioxin, was spread as a dust palliative at three horse arena farms and along one stretch of road. If the oil weight was 1000 kg/m³, the maximum amount of dioxin was ~ 22 kg. Soil samples taken of the most afflicted farm 3 months after the event at a depth of about 15 cm were found to contain an average of 32 mg/kg dioxin. If the half-life was 463 days in soil, about 13% disappears in the 3-month interval, and the initial concentration was thus ~ 37 mg/kg. It is reasonable to assume that none of the dioxin had leached up or down, and assuming a compacted soil weight of 2650 kg/m³, the initial rate of application should have been ~ 147 kg/ha. Moreover, the total area contaminated at this application rate could not have exceeded about 0.15 ha. In terms of the top 10 cm of normal soil, this represents 110 mg/ka. After 3 days, people became ill, but children were more susceptible; livestock and wildlife died; many humans were hospitalized, especially for chloracne. Livestock continued to die as late as January, 1974, even though the soil of the horse arena at one of the farms was removed. TCDD was deduced to be present when 2,4,5-trichlorophenol was positively identified. The oil sludge sprayed had been collected from many industries, and it was shown that the dioxin had originated from a hexachlorophene producer in 1970 and 1971. The producer had contracted disposal of these residues to a chemical distributor, who subcontracted this disposal to the salvage oil company that sprayed the horse arenas. The fluid still left in the storage tank also contained TCDD.

D. The Seveso Incident

The chemical reactions leading to the explosion in the ICMESA plant producing 2,4,5-trichlorophenol have already been noted in Chapter 1. The explosion⁴¹⁶ released a chemical cloud which blew over the cities of Seveso and Cesano Maderno near Milan in Meda, Italy on July 10, 1976. The plant owned by L. Givaudan and Company, a subsidiary of Hoffman-La Roche, produced 2,4,5-trichlorophenol for shipment to plants in Clifton, N.J. and in Switzerland for use in the manufacture of hexachlorophene. The accident occurred during the Saturday lunch hour. On overheating in the reaction vessel, the safety valve blew, releasing about 10% of the contents horizontally from the reactor building into the atmosphere, and the resultant cloud was carried toward Seveso. It was estimated to contain 500 kg of 2,4,5-trichlorophenol and 2 kg of TCDD, and it spread quickly in a southerly direction, initially covering an area, about 5 km long and 700 m wide. The affected areas were subsequently (after a 2-week delay) divided into three zones:⁴¹⁷ Zone A (an area of approximately 115 ha in the town of Seveso just south of the plant in which residues of 50 ppm TCDD were found on tree leaves and $50 \mu\text{g}/\text{m}^2$ to $1 \text{ mg}/\text{m}^2$ in the soil; 800 people were evacuated), Zone B (in an area of 255 ha of population 4000, showed 5 to $50 \mu\text{g}/\text{m}^2$ TCDD in soil; 1300 children and pregnant women were evacuated), and Zone C (further south, with a population of 11,000 and an area of 1400 ha which contained light residues over a large area). Thus, over 320 ha of Zones A and part of B, the application rate of TCDD was ~ 8 g/ha. In terms of the top 10 cm of normal soil, this represents $5.9 \mu\text{g}/\text{kg}$.

The Regional Health Minister appointed three committees to cover the areas of med-

icine and epidemiology, veterinary medicine, and chemical analysis and decontamination. Approximately 2000 people were exposed; 300 were treated for skin disorders including burns (caused by the phenol), and 46 people were hospitalized. Over 1100 animals were killed directly, and up to 255 ng/g of TCDD was found in the liver of dead rabbits from Zone A. The medical committee undertook to test and assist 11,000 people from the area over the next 5 to 10 years. The workers in the ICMESA plant were also monitored, and an epidemiological study involving 200,000 people in the area was commenced. A review of news items and reports in the Italian press during July 10, 1976 to September 10, 1976 has been published by the Rachel Carson Trust for the Living Environment.⁴¹⁸ Other reports^{419,422-431,433,435-439} should be consulted for the detailed history of the Seveso Incident. As of 1977, the TCDD had penetrated into the soil at least 30 cm. To decontaminate the area, it was proposed⁴¹⁹ to incinerate the top soil and plants;⁴²⁰ Calvin suggested that carbon granules and cow manure be spread to adsorb the TCDD and also suggested that photodecomposition would be the preferred mode. Crosby et al.^{420,421} recommended that the topsoil be sprayed with an olive oil emulsion to induce reductive dechlorination in sunlight. The TCDD could be thereby detoxified to less toxic dioxins and also made more volatile so it could be diluted in the air by evaporation. This was the recommendation followed, and this halved the vegetation residues in 24 hr. However, rain intervened and the rest of the residues were washed into the soil. The TCDD was analyzed by a mass fragmentographic technique, originally proposed by Baughman and Meselson⁴³² in 1973. A review of analytical methods for TCDD is available.⁴³⁴

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