Background Report Reference

AP-42 Section Number: 9.2.2

Background Chapter: 4

Reference Number: 12

Title: "Toxaphene Volatilization from a Mature Cotton Canopy" in Agronomy Journal

G.H. Willis et al.

1980

Table 3. Influence of inoculation method on nodule number and fresh weight in two cultivars of *P. vulgaris*. Popayan, 1979A.†

Method of inoculation	Nodule number/plant		Nodule fresh wt.	
	Puebla 152	Carga-manto	Puebla 152	Carga-manto
			mg/plant	
Uninoculated	5.1 a*	0.5 a	73 a	17 a
Inoculated (no pellet)	5.6 a	6.3 a	55 a	66 a
Inoculated (lime pelleted)	8.1 a	6.1 a	80 a	91 ab
Granular inoculant (0.5 g/m)	17.5 a	40.4 b	93 ab	194 bc
Granular inoculant (1.0 g/m)	21.8 a	32.5 b	102 ab	205 bc
Granular inoculant (2.0 g/m)	51.2 b	116.5 d	151 b	458 d
Granular inoculant (4.0 g/m)	48.5 b	88.2 c	143 b	240 c

* Numbers not followed by the same letter are significantly different at the 5% level. † Measured 45 days after planting.

perior to that of seed applied inoculants (Tables 2, 3). At La Selva, PCNB-treated seeds inoculated with granular preparations, achieved nodule numbers at least equal to those of pesticide free seeds which had been inoculated and lime pelleted. While most previous studies with granular inoculants have emphasized soils of higher pH, and used slow-growing rhizobia (Stoddard, 1976; Bezdicek et al. 1978), this method of inoculation appears to have considerable promise for the acid bean-producing soils of Latin America, and should be studied further.

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Toxaphene Volatilization from a Mature Cotton Canopy[1]

G. H. Willis[2], L. L. McDowell[3], S. Smith[2], L. M. Southwick[2], and E. R. Lemon[4]

ABSTRACT

To fully understand the pollution potential of pesticides more knowledge concerning the mechanisms and rates of pesticide exchange between environmental compartments is needed. The momentum balance method was used in a field study to characterize toxaphene (chlorinated camphene) volatilization from cotton (*Gossypium hirsutum* L.) plants for 5 days following aerial application at 2.24 kg/ha. The momentum balance method uses accurate measurements of windspeed profiles, temperature gradients, and atmospheric pesticide concentration gradients above the plant canopy to provide data for calculating vertical flux densities of pesticides. The calculated volatile loss for the 5-day period was 358 g/ha, which represented a loss of 26% of the toxaphene present in the canopy. Although typical volatile loss patterns suggested that flux densities were highest during mid-afternoon, there was evidence that volatility rates were also high when leaves were drying after heavy dew or light rain. Based on comparisons of the amounts of toxaphene transported from nearby cotton fields via surface runoff in earlier studies and the amounts lost by volatilization in this study, it was concluded that aerial transport is the pathway of greater loss.

Additional index words: Aerodynamic profile method, Momentum balance method, Aerial pesticide application, *Gossypium hirsutum* L., Flux density, Pesticide, Insecticide, Chlorinated camphene.

INTEREST in the environmental fate of pesticides has led researchers to attempts to characterize pesticide distribution into various environmental compartments, and the mechanisms and rates of exchange between compartments. Volatilization and aerial transport are recognized as major pathways of pesticide movement in the environment (1, 14, 18, 25, 27) and much has been learned about pesticide volatility from soil and water environments (3, 5, 8, 9, 19, 21). However, only a few studies have been concerned with measuring pesticide flux rates under field conditions (8, 22, 23, 28), especially from plant surfaces (22, 23). Volatile losses from plants need further study since many insecticides are applied foliarly, e.g., toxaphene (chlorinated camphene) applied to cotton (*Gossypium hirsutum* L.) to control the tobacco budworm (*Heliothis virescens* Fabricius) and the cotton bollworm (*Heliothis zea* Boddie).

Many studies concerning pesticide disappearance (volatilization, degradation, sorption) from plant surfaces suggest that pesticide loss usually follows a decreasing exponential curve. However, close examination of the data revealed that first-order kinetics do not always best describe such losses (6, 25). The rate of pesticide loss from plant surfaces depends on several factors—plant (form and wettability of leaf cuti-

[1] Joint contribution from Soil and Water Pollution Research Unit, USDA-SEA-AR, P. O. Drawer U, Baton Rouge, LA 70893 cooperating with the Louisiana Agric. Exp. Stn.; USDA Sedimentation Laboratory, Oxford, MS 38655; and the Microclimate Project, USDA-SEA-AR, cooperating with Dep. of Agronomy, Cornell Univ., Ithaca, NY 14853. Received 13 May 1979.

[2] Soil scientist and chemists, USDA, Baton Rouge, La.

[3] Soil scientist, USDA, Oxford, Miss.

[4] Soil scientist, USDA, and professor of soil science, Cornell Univ., Ithaca, N.Y.

cle, and canopy structure), pesticide (chemical nature and formulation), and meteorological (rainfall, wind, and temperature).

The objective of this study was to characterize the volatile loss of toxaphene from a mature cotton canopy under field conditions.

MATERIALS AND METHODS

The study was conducted in a 25-ha cotton field about 17 km northwest of Clarksdale, Miss. The plant canopy completely covered the soil surface and was uniformly level with no aerodynamic obstructions within the experimental area. The fetch-height ratio was at least 100:1 in all directions. Toxaphene was applied aerially (low volume) as an emulsifable concentrate at a rate of 2.24 kg/ha on 14 and 20 Aug. and on 6 Sept. 1974. The study was aborted twice because of rainfall 29 hours after application on 14 August and 2 hours after application on 29 August. The study was conducted during daylight hours for 5 days after application at 0745 hours on 6 September.

We used the aerodynamic profile (momentum balance) method for calculating toxaphene flux densities. This method is based upon accurate measurements of windspeed profiles and atmospheric pesticide concentration gradients above the plant canopy. The theory, assumptions, and limitations of this method have been discussed previously (7, 10, 11, 13, 16) and will be mentioned only briefly here. The vertical flux density of a pesticide is calculated as the product of a pesticide transfer coefficient, Kp, and the pesticide concentration gradient, $\Delta p/\Delta z$. The aerodynamic method assumes that vertical fluxes of momentum and pesticide are equal and that the momentum transfer coefficient, Km, determined from windspeed profiles, can be substituted for the pesticide transfer coefficient. The aerodynamic method is valid only during adiabatic conditions. Thus, diabatic correction functions are necessary to adjust the flux calculations for conditions of thermal stability. Two such stability correction functions are the KEYPS (15) and Davis (17) methods. The vertical flux density, F, is given by:

$$F = k^2 (c_1 - c_2)(u_2 - u_1)/\{\ln[(z_2 - d)/(z_1 - d)]\}^2 \psi^2$$

where c_1, c_2, u_1, and u_2 are pesticide concentrations and windspeeds at heights z_1 and z_2, respectively; k is von Karman's constant (0.41); d is the crop displacement height; and ψ is the correction factor that depends upon the thermal stability of the atmosphere.

Toxaphene concentration gradients above the canopy were obtained by an air-sampling system similar to that reported by Caro et al. (2). Air samples were collected at two masts spaced 15-m apart in the approximate center of the field. The samplers (250-ml gas washing bottles containing 100 ml ethylene glycol) were positioned 185, 210, 235, 285, and 335 cm above the soil surface on each mast. The height of the plant canopy was 165 cm above the soil surface. Air was drawn by vacuum pump through the samplers at a continuously monitored rate of 6 liters/min. Two-hour sampling periods were used throughout the study, except on 9 and 10 September when 4-hour periods were used. After each sampling period the ethylene glycol was quantitatively transferred from the sampler with 25 ml H_2O to a 300-ml amber bottle. Approximately 100 ml hexane was then added to the sample and the bottle was capped with a Teflon[a]-lined lid and refrigerated until extraction. The samples were extracted by mixing the contents of the bottle for 1 hour with a magnetic stirrer, and transferring the mixture to a 500-ml separatory funnel, from which the ethylene glycol-water fraction was drained. The hexane was washed once with H_2O, dried with anhydrous Na_2SO_4, and adjusted to a volume appropriate for gas chromatographic analysis. The trapping and extraction efficiencies were 90 and 92%, respectively. The lower detection limit for toxaphene in air was about 15 ng/m³.

Windspeed profiles were measured by rotating-cup anemometers (Cardion Electronics Wind Speed Profile System) at six elevations (185, 235, 285, 335, 385, and 435 cm) above the soil surface. The anemometer mast was located halfway between and 15 m downwind (prevailing wind from the south) from the two sampling masts. Crop displacement height (d), determined graphically from neutral profiles (30), was 110 cm above the soil surface. Atmospheric stability correction was determined by the KEYPS and Davis methods using temperature differences measured by aspirated, shielded thermocouples at 185 and 285 cm above the soil surface. Air temperatures and relative humidity were also measured by a maximum-minimum thermometer and a hygrothermograph located in a standard weather shelter.

The toxaphene load in the crop canopy was determined from plant samples collected throughout the study. Each sample consisted of all the leaves, petioles, and bolls within a 0.5 × 0.5-m template centered over a row of plants. The number of samples collected each time varied from two to eight. The plant material, divided into upper and lower plant halves, was placed in glass jars, covered with hexane, and stored for a minimum of 4 weeks. The hexane was decanted and the plant material washed with fresh hexane. The washings were combined in a separatory funnel, any accumulated water was drained, and the hexane was dried with Na_2SO_4. A 50-ml aliquot of the hexane was added to a 2 × 10-cm prewashed (hexane) Florisil column. The toxaphene was eluted with 200 ml of 85:15 (v/v) hexane:ether solution, and the volume adjusted for gas chromatographic analysis. The plant extraction procedure resulted in 94% recovery.

Triplicate soil samples were collected approximately 24 hours before and 1 to 2 hours after each toxaphene application. The surface soil, within the same 0.5 × 0.5-m area from which a plant sample was taken, was excavated to a depth of 2.5 cm. A 10-g subsample of the air-dried soil was Soxhlet-extracted with 200 ml of an azeotropic (41:59 v/v) hexane:acetone mixture. The extract was partitioned in a separatory funnel, washed with H_2O to remove acetone, dried with anhydrous Na_2SO_4, and diluted or concentrated to an appropriate volume for gas chromatographic analysis. The procedure resulted in greater than 95% recovery.

Aliquots (5μl/injection) of the hexane extracts of air, plant, and soil samples were analyzed for toxaphene using a Micro-Tek model GC-2000 R gas chromatograph equipped with a ^{63}Ni high temperature electron-capture detector operated at 290 C, and with inlet and column oven at 220 and 215 C, respectively. The carrier gas (filter-dried N_2) flow rate was 150 cm³/min. The column was a coiled Pyrex glass tube 180 cm long × 6 mm O.D. × 4 mm I.D., packed with 3% OV-1 on 80-100 mesh Chromosorb W (high performance, AW, DMCS). Toxaphene in hexane extracts was quantified by triangulating the last four peaks of the chromatogram (4) and comparing the values obtained with those of standard toxaphene (sample X-16189-49 supplied courtesy Hercules Inc., Wilmington, Del.).

RESULTS AND DISCUSSION

The amounts of toxaphene measured in the crop canopy after application on 14 August and 6 September are shown in Table 1. The 201-g/ha toxaphene load present in the canopy 1 hour after application

Table 1. Amount of toxaphene on cotton plants.

Date (1974)	Toxaphene application		Number of plant samples†	Time of collection	Toxaphene load ($\bar{x} \pm$ s.d.)
	Time	Amount			
	hours	kg/ha		hours	g/ha
13 Aug.	–	–	3	1500	0
14 Aug.	1000	2.24	4	1100	201 ± 145
15 Aug.			4	1000	148 ± 82
28 Aug.			3	1500	–‡
29 Aug.	1000	2.24	4	1100	–‡
5 Sept.			3	1500	935 ± 34
6 Sept.	0800	2.24	8	0900	1,369 ± 243
8 Sept.			2	0900	1,256 ± 93
10 Sept.			2	0900	824 ± 240

† Plant samples collected at the same time at different sites in the test area. ‡ Since the 29 August run was aborted 2 hours after starting, and no volatility calculations were made, these plant samples were not analyzed.

[a] Mention of trade names or commercial materials is for the convenience of the reader and does not constitute any preferential endorsement by the USDA over similar products available.

on 14 August represents 9% of that applied (2.24 kg/ha). The canopy background load was 935±34 g/ha on 5 September because of the previous applications (14 and 29 August). Therefore, the 6 September application added 434 g/ha (935 + 434 = 1,369) to the canopy, which is 19% of the amount applied. Of the total amount of toxaphene measured on 6 September, 84±4% was in the upper half of the canopy. Little, if any, of the aerially applied toxaphene penetrated the 165-cm-thick canopy to reach the soil surface (data not shown).

The apparently small amounts of toxaphene on plants after application could have resulted from several factors: i) application errors; ii) drift, evaporation, and rapid initial volatilization; iii) sampling errors, and iv) analytical errors. The toxaphene was applied by a commercial aerial applicator. We had no convenient way of checking the accuracy of application. Air temperatures at the time of application (29 and 21 C on 14 August and 6 September, respectively) were probably high enough to permit some toxaphene loss by evaporation and volatilization during application and during the hour before plant sample collection (25). Sampling errors are always possible, especially when minimal numbers of samples are collected. We found in later studies that 20 plant samples were required for accurate estimates of pesticide load in a plant canopy. However, the four or eight randomly selected samples collected within 1 hour after application were sufficient to demonstrate that the amounts of aerially applied toxaphene intercepted by the plants were low. Studies by Ware et al. (24) showed that less than 50% (depending on conditions, with amounts varying above and below a mean of 47%) of aerially applied insecticides reached the target zone in Arizona fields. Approximately 48% of the applied toxaphene was intercepted by alfalfa (air temperature = 16 C).

Figure 1 shows typical atmospheric concentration profiles of toxaphene above the canopy surface. As we expected, under normal daytime conditions, the concentrations decreased with elevation (mast 1, $Y = 1{,}408 - 225 \ln X$; mast 2, $Y = 1{,}082 - 163 \ln X$). Figure 1 also illustrates a major problem we encountered throughout the study. Even though the data were collected simultaneously at masts spaced 15-m apart, there was much more concentration variability for mast no. 2 ($r^2 = 0.70$) than for mast no. 1 ($r^2 = 0.92$). We found variability at various times throughout the study at each mast. This problem will be discussed in more detail in later paragraphs.

Figure 2 shows the mean calculated flux density for each 2-hour sampling period on 8 September (2 days after application). Flux density appeared to increase as the temperature increased to a maximum during mid-afternoon, and then decreased as temperatures began to decline.

On 15 August (1 day after application) the flux density was highest during the 0800-1000 hour sampling period (Fig. 3). The cotton plants were wet with a heavy dew that morning, which may have increased volatilization from sunup until the leaves dried. The conditions seem to be analogous with the report of Harper et al. (8) who found that some volatility rates of trifluralin (α,α,α-trifluoro-2,6-dinitro-N,N-dipropyl-*p*-toluidene) from soil were highest at

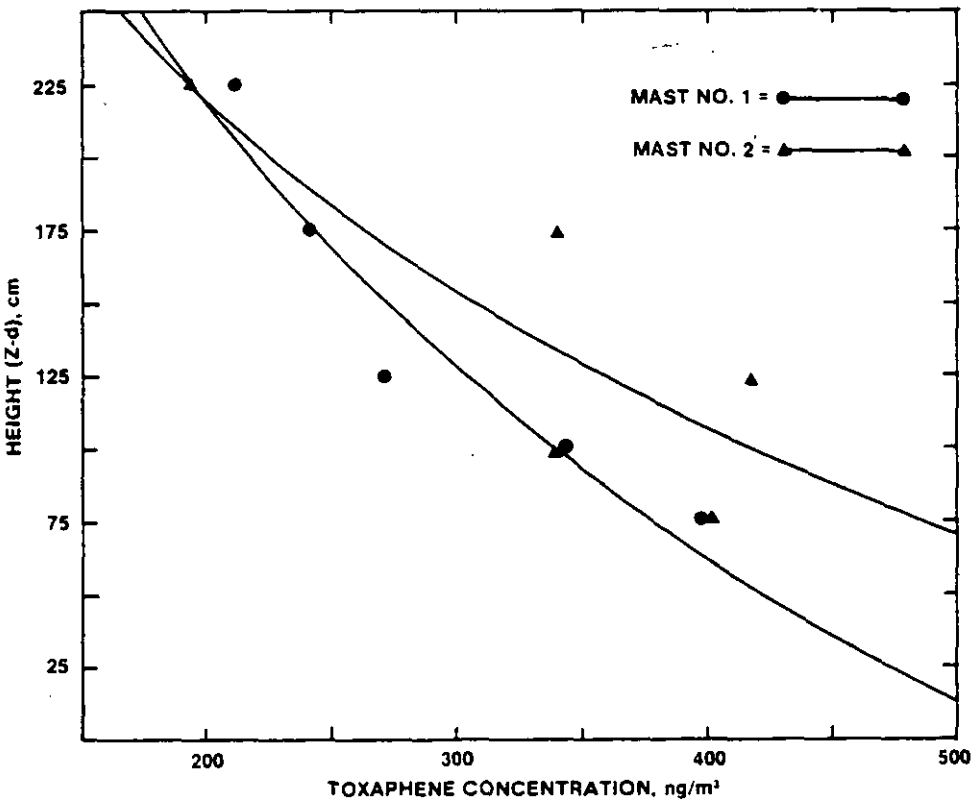

Fig. 1. Atmospheric concentration profiles of toxaphene above the canopy surface, 1300-1500 hours, 14 Aug. 1974.

night in Georgia when the soil surface began to rewet after it had dried during the day. Their data agreed with those of Spencer et al. (20), who reported that pesticide vapor densities decreased when soil water content decreased below the equivalent of 1 molecular layer in thickness. Possibly, conditions were similar on 15 August, i.e., heavy dew resulted in increased pesticide desorption from leaf surfaces and increased volatilization. Also, toxaphene dissolved in the dew may have accumulated at high enough concentrations at the air-water interface to increase volatilization over that from dry leaf surfaces at the same temperature (Cliath, M. M. 1978. Vapor behavior of EPTC in aqueous systems. Ph.D. thesis. Univ. Calif., Riverside). After the leaf surfaces became dry, the flux density decreased and then appeared to increase with temperature until the study was terminated because of a thunderstorm. Mean windspeed during the sampling periods on 15 August was 4.7 km/hour. High volatility rates also occurred on the morning of 9 September, after an overnight shower of 1.3 mm. However, the mean windspeed that morning was 13.0 km/hour, which confounded any effect that wet leaves may have had on flux density. The effect of 0.5 mm rainfall during the afternoon of 8 September was also impossible to separate from wind and temperature effects.

The calculated flux densities[6], shown in Table 2 for masts 1 and 2, did not always agree because of the toxaphene concentration variability. In one case (mast no. 1, 10 September), the data were rejected because of the variability. However, the flux densities calculated at the two masts agreed closely enough to show trends and the order of magnitude of the volatile losses. Variability in this type of data is not unusual. Further, comparisons of different meteorological techniques of estimating flux densities above tall vegetation have shown two-fold differences (12, 29). The flux densities in Table 2 are mean values for daylight hours only. Nocturnal losses could and probably did occur when wind and turbulence were present.

If weather conditions were constant, one would expect the highest flux densities during day 1 and 2 after application, when pesticide concentrations on leaf surfaces are highest. However, in this study, the increased windspeed on 8 and 9 September (due to remnants of a tropical storm) and increased cloud cover on 6, 7, and 8 September (lower temperatures) caused the highest volatility rates to occur on days 3 and 4 after application. As expected, simple linear regression (using mean daily values) showed high correlation between windspeed and toxaphene flux density ($r^2 = 0.85$ and 0.76 for masts 1 and 2, respectively). However, there was no significant statistical relationship between mean daily temperature and flux density. Apparently the effect of higher windspeeds on 8, 9, and 10 September masked temperature effects. Multiple linear regression suggested a combined effect of wind and temperature on flux density (r^2 = 0.98 and 0.79 for masts 1 and 2, respectively).

[6] Since both methods of stability correction were in close agreement, the flux density calculations of only one method (KEYPS) are included. The Davis method of correction consistently resulted in calculated volatile losses of 2 to 5 g/ha-day greater than the KEYPS method.

Table 2. Mean† windspeeds, temperatures, and toxaphene flux densities for 6 through 10 Sept. 1974.

Date	Windspeed	Temperature	Toxaphene flux density, Mast No. 1	Toxaphene flux density, Mast No. 2	Toxaphene flux density, Avg.
	km/hour	°C	g/ha-hour	g/ha-hour	g/ha-hour
6 Sept.	6.0	22.8	5.8	3.2	4.5
7 Sept.	7.5	25.1	8.5	8.0	8.3
8 Sept.	12.1	22.6	10.4	11.6	11.0
9 Sept.	11.2	28.1	11.6	8.4	10.0
10 Sept.	9.8	27.0	–	7.7	–

† Mean values for daylight hours only—from 0800 to 1800 hours for 6, 7 and 8 September, and from 0800 to 1600 hours for 9 and 10 September.

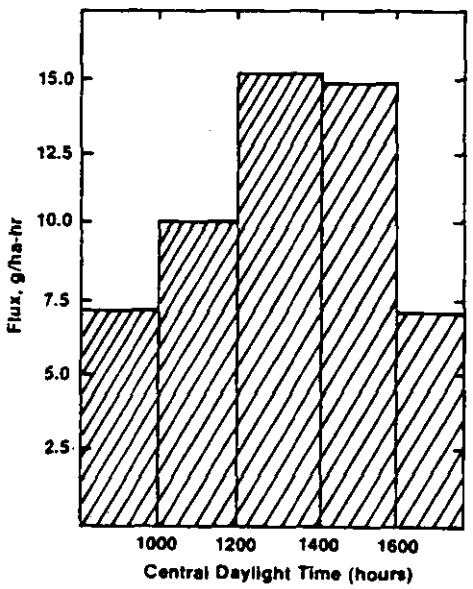

Fig. 2. Calculated toxaphene flux densities, average of masts no. 1 and 2, 8 Sept. 1974.

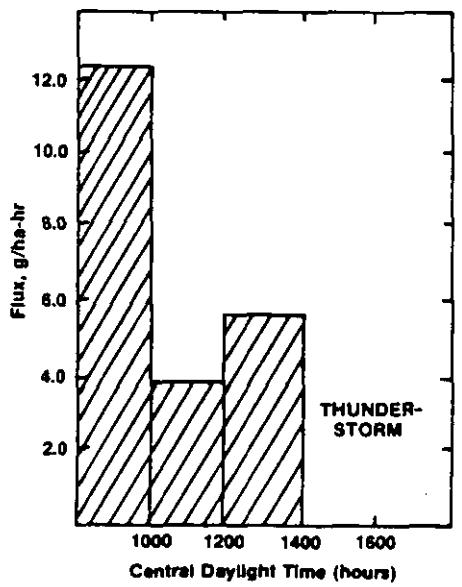

Fig. 3. Calculated toxaphene flux densities, average of mas no. 1 and 2, 15 Aug. 1974.

The calculated volatile loss totals were 341 and 318 g/ha for mast no. 1 and the average of masts nos. 1 and 2 (4 days), respectively, and 358 g/ha for mast no. 2 (5 days). These values represented 25, 23, and 26% of the toxaphene present on the plants when the study was initiated on 6 September. The plant data in Table 1 showed the total 5-day loss [volatilization (day and night), washoff from rain and/or dew, and degradation] was 545 g/ha, or 40% of that present on the plants when the study was initiated. Despite the data variability for both plant loading and volatility, the calculated losses agreed reasonably well.

Studies (26) in cotton fields adjacent to the site of this experiment have shown that about 100 g/ha-year of toxaphene were transported in runoff and sediments during two high rainfall years. The calculated volatile loss of about 350 g/ha of toxephene in 4 to 5 days after application illustrates that volatilization and subsequent aerial transport is probably the major pathway of toxaphene transport from cotton.

ACKNOWLEDGMENT

The authors are grateful for the able assistance of Mike Johnson and George Drake, USDA-SEA-AR, Ithaca, N.Y., in conducting this study.

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