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Insecticide Residues on 1972 U.S. Auction-Market Tobacco*

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The objections to and the implications of high pesticide residues on tobacco have been discussed in several reports (1, 2, 3). Pesticide residues are now a factor in determining quality; therefore, tobacco containing high concentrations of pesticides will be considered lower quality.

Since 1970, U.S. tobacco producers have had to certify that DDT and TDE were not used on their crops in order to qualify for Federal price support. The purpose of this certification was to force farmers to eliminate all use of DDT and TDE on tobacco. Although the 1970 crop showed a large reduction in DDT-TDE levels (4) from those reported in 1968 (5), there was little doubt that these insecticides were still being applied by some farmers. In 1972, warnings were issued that false certification on the use of DDT and TDE could lead to prosecution; and the Agricultural Stabilization Conservation Service of the U.S. Department of Agriculture analyzed tobacco samples selected randomly at the auction market before sale to determine if a false certification had been made.

This paper reports the results from our 1972 sampling of auction market tobaccos and is part of a long-term survey of pesticide residues on tobacco and tobacco products. Our survey is unrelated in purpose and sampling procedures to the regulatory actions of the U.S. Department of Agriculture.

EXPERIMENTAL PROCEDURE

Flue, air, and fire-cured tobaccos were sampled at the time of sale. Each sample is a composite of 50 leaves; that is, five leaves from each of 10 farmers. At least one sample was obtained from each stalk position (top, middle, and bottom) from each market. Burley was sampled at sales, from hogsheads, and at a large manufacturing plant during the stemming operation.

The tobacco composites were ground in a Wiley Mill, mixed thoroughly, and analyzed by the acetonitrile-water extraction and gas chromatographic procedures previously reported (4, 6). The tobacco was analyzed for total DDT[a], total TDE[b], total endosulfan[c], endrin, and toxaphene. The moisture content of each sample was determined, and the residue values were corrected to 13 % moisture content.

RESULTS AND DISCUSSION

Flue-Cured-Tobacco. The residue levels for the flue-cured markets are summarized in Table 1. The average over all belts for DDT + TDE was 0.85 ppm. This value is about 14 % of the 1970 mean of 6.1 ppm (4).

Frequency distributions for various concentration ranges are shown in Table 2 for the 1968 and 1970 crops (4, 5) as well as the 1972 crop. These data show that in 1968 about 99 % of the samples contained DDT + TDE in excess of 10 ppm, whereas in 1972 none of the samples contained 10 ppm. In 1972 the eastern North Carolina and North Carolina-South Carolina border belts had the greatest number of samples with DDT + TDE residues greater than 1 ppm (26.6 and 30.5 %, respectively). About 17 % of the samples from the old North Carolina-Virginia belt and approximately 10 % of those from Georgia-Florida belt contained more than 1 ppm of DDT + TDE in 1972. None of the samples from the middle North Carolina belt had residues greater than 0.5 ppm.

We thought that, after the ban on DDT and TDE use on tobacco in 1970, endosulfan use might increase. However, residues of endosulfan dropped from 2.6 ppm over all belts in 1970 to 0.75 ppm in 1972.

Over 90 % of the flue-cured samples were positive for toxaphene in 1972. This contrasts with the 1970 sampling where less than 30 % contained toxaphene (4). The objections to toxaphene residues have been previously discussed (7). Whether these residues are due to direct application or accidental contamination from use of toxaphene on other crops is not known. Nevertheless, tobacco producers should be informed

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[a] Total DDT is the sum of p,p'-DDE, o,p-DDT, and p,p'-DDT.

[b] Total TDE is the sum of p,p'-TDEE, o,p-TDE, and p,p'-TDE.

[c] Total endosulfan is the sum of endosulfan I, endosulfan II, and endosulfan sulfate.

Table 1. Ranges and means for insecticide residues in flue-cured tobacco samples from the 1972 U.S. auction markets.

Insecticide	Belt	Range (ppm)	Mean[a] (ppm)
DDT	Ga.-Fla.	0 –0.50	0.20
	N.C.-S.C. border	0.06–4.23	0.49
	Eastern N.C.	0.07–4.21	0.91
	Middle N.C.	0.10–0.30	0.19
	Old N.C.-Va.	0.09–1.86	0.25
	All belts	0 –4.23	0.40
TDE	Ga.-Fla.	0 –3.88	0.39
	N.C.-S.C. border	0.08–5.40	0.69
	Eastern N.C.	0.05–0.86	0.22
	Middle N.C.	0.05–0.22	0.11
	Old N.C.-Va.	0.06–3.34	0.44
	All belts	0 –5.40	0.45
DDT + TDE	Ga.-Fla.	0 –4.11	0.58
	N.C.-S.C. border	0.18–7.62	1.18
	Eastern N.C.	0.12–4.43	1.13
	Middle N.C.	0.18–0.41	0.30
	Old N.C.-Va.	0.15–5.21	0.69
	All belts	0 –7.62	0.85
Endosulfan	Ga.-Fla.	0 –6.69	1.73
	N.C.-S.C. border	0 –1.89	0.29
	Eastern N.C.	0 –4.20	0.42
	Middle N.C.	0 –1.39	0.19
	Old N.C.-Va.	0 –7.77	1.06
	All belts	0 –7.77	0.75
Toxaphene	Ga.-Fla.	0 –3.91	0.71
	N.C.-S.C. border	0 –7.94	1.26
	Eastern N.C.	0.09–8.82	1.93
	Middle N.C.	0.33–0.81	0.56
	Old N.C.-Va.	0.15–5.38	0.51
	All belts	0 –8.82	1.00
Endrin	Ga.-Fla.	0 –0.06	<0.01
	N.C.-S.C. border	0 –0.41	0.02
	Eastern N.C.	0 –0.01	<0.01
	Middle N.C.	0 –0	<0.01
	Old N.C.-Va.	0 –0	<0.01
	All belts	0 –0.41	<0.01

[a]Number of samples from each belt are 21 for Georgia-Florida (Ga.-Fla.), 36 for North Carolina-South Carolina border (N.C.-S.C.), 15 for eastern North Carolina, 12 for middle North Carolina, and 24 for old North Carolina-Virginia (N.C.-Va.) belts.

that its presence on tobacco is undesirable, and every effort should be made to ensure that is no longer be used. The eastern North Carolina and border belts had the greatest number of samples with toxaphene levels over 1 ppm.

Endrin residues remained at or near the detection limit. In the North Carolina-South Carolina border belt only two samples contained more than 0.01 ppm (0.11 and 0.41 ppm). Therefore, except for occasional low levels, endrin residues no longer seem to be a problem on flue-cured tobacco.

Table 2. Frequency distributions for various concentrations of insecticides in tobacco from the 1968, 1970, and 1972 U.S. flue-cured auction market.

Insecticide	Concentration range (ppm)	Samples within range for 1968[a] (%)	1970[b] (%)	1972[c] (%)
DDT	0 –0.099	0	0	9.3
	0.10–0.49	0	41.1	73.1
	0.50–0.99	1.2	22.3	8.3
	1.0 –2.99	3.6	20.5	6.5
	3.0 –9.99	25.0	11.6	2.8
	= > 10	70.2	4.5	0
TDE	0 –0.099	0	0.9	19.4
	0.10–0.49	0	35.7	63.9
	0.50–0.99	0	23.2	6.5
	1.0 –2.99	0	17.0	6.5
	3.0 –9.99	2.4	12.5	3.7
	= > 10	97.6	10.7	0
DDT + TDE	0 –0.099	0	0	0.9
	0.10–0.49	0	11.6	63.9
	0.50–0.99	0	27.7	15.7
	1.0 –2.99	0	31.3	12.0
	3.0 –9.99	1.2	15.2	7.4
	= > 10	98.8	14.3	0
Endosulfan[d]	0 –0.249		22.3	55.6
	0.25–0.49		6.3	11.1
	0.50–0.99		10.7	15.7
	1.0 –2.99		30.4	11.1
	3.0 –9.99		28.6	6.5
	= > 10		1.8	0
Toxaphene[e]	0 –0.49			57.4
	0.5 –0.99			23.1
	1.0 –2.99			9.3
	3.0 –4.99			6.5
	5.0 –9.99			3.7
	= > 10			0

[a]84 samples analyzed in 1968.
[b]112 samples analyzed in 1970.
[c]108 samples analyzed in 1972.
[d]Samples were not analyzed for endosulfan in 1968.
[e]Samples were not analyzed for toxaphene in 1968; in 1970, its presence or absence was determined but it was not quantitated.

Burley Tobacco. The analysis of the 1972 crop suggested that DDT and TDE use on burley tobacco was rare (Table 3). In past surveys (4, 5) burley tobacco from Kentucky and Tennessee contained high levels of DDT and TDE. Only one 1972 sample from Kentucky, which was sampled from a hogshead, had a concentration greater than 0.25 ppm. This sample contained 8.17 ppm of DDT + TDE; the value suggests that one or more farmers whose tobacco went into this hogshead ignored the ban on DDT and TDE or that their tobacco was contaminated indirectly in some manner. The values for the Kentucky burley crop are in good agreement with those of *Dorough* et al. (10). North Carolina burley continued to have levels of DDT and TDE at or near the limits of detection.

Endosulfan residues on Kentucky and Tennessee burley

Table 3. Range and mean values for insecticide residues in burley tobacco from the 1972 crop.

Pesticide	State[a]	Range (ppm)	Mean (ppm)
DDT	N.C.	0.10–0.21	0.13
	Ky.	0.07–0.68	0.14
	Tenn.	0.08–0.13	0.10
TDE	N.C.	0.01–0.05	0.03
	Ky.	0.04–7.48	0.60
	Tenn.	0.07–0.28	0.17
DDT + TDE	N.C.	0.12–0.26	0.17
	Ky.	0.11–8.17	0.73
	Tenn.	0.17–0.37	0.27
Endosulfan	N.C.	0 –0.33	0.06
	Ky.	0.10–13.77	4.85
	Tenn.	0.22–5.90	1.48
Toxaphene	N.C.	0.28–0.97	0.43
	Ky.	0.26–1.07	0.53
	Tenn.	0.39–3.36	1.24
Endrin	N.C.	0 –0.01	<0.01
	Ky.	0 –0.31	0.05
	Tenn.	0.01–0.04	0.02

[a] Six samples were analyzed from the North Carolina (N. C.) auction market. Fourteen samples were analyzed from Kentucky (Ky.); six were composites of the three stalk positions sampled at the auction market and eight were sampled from hogsheads. Five of the Tennessee (Tenn.) samples were taken during the stemming operation.

tobacco decreased in 1972 from the levels reported in 1970 (4). The Federal Republic of Germany has proposed a tolerance of 5 ppm (8). Five of the 14 samples from Kentucky contained residues over this tolerance, and the residues in over 60% of the samples were greater than 3 ppm. Only one sample from Tennessee contained more than 1 ppm of endosulfan. One of six North Carolina samples contained a detectable amount of endosulfan, and its concentration was 0.33 ppm. Recent work by *Dorough* et al. (9) has shown that burley tobacco treated with endosulfan must remain as long as 21 days before harvest if the 5 ppm tolerance is to be met.

Applications of toxaphene to burley tobacco seem to have increased. In 1970 (4), only 18% of the samples appeared to contain this insecticide. Fifty percent of the 1972 samples had toxaphene concentrations greater than 0.5 ppm, and three values were greater than 1 ppm. In North Carolina only one sample was greater than 0.5 ppm.

Endrin in all burley samples remained near the detection limit of the analytical technique.

Fire and Air-Cured Tobacco. The residue values for fire and air-cured tobaccos are given in Table 4. Except for Tennessee dark air-cured tobacco, the DDT and TDE residues were, in general, similar to the residue levels found on flue-cured and burley tobacco. Although the DDT + TDE residue in Tennessee air-cured tobacco

Table 4. Ranges and means for insecticide residues in fire and air-cured tobaccos.[a]

Insecticide	Type	Market	Range (ppm)	Mean (ppm)
DDT	Fire-cured	Tenn.	0.10–0.19	0.14
		Va.	0.07–0.13	0.10
	Dark air-cured	Tenn.	0.17–2.99	1.03
		Va.	0.06–0.16	0.10
	Light air-cured	Md.	0.08–0.10	0.09
TDE	Fire-cured	Tenn.	0.07–0.21	0.21
		Va.	0.03–0.10	0.08
	Dark air-cured	Tenn.	0.08–6.64	2.43
		Va.	0.11–2.63	0.56
	Light air-cured	Md.	0.04–0.08	0.05
DDT + TDE	Fire-cured	Tenn.	0.18–0.40	0.26
		Va.	0.11–0.24	0.17
	Dark air-cured	Tenn.	0.25–9.63	3.46
		Va.	0.17–2.79	0.67
	Light air-cured	Md.	0.12–0.16	0.14
Endosulfan	Fire-cured	Tenn.	5.49–9.42	7.34
		Va.	0 –5.09	2.45
	Dark air-cured	Tenn.	7.11–13.03	10.32
		Va.	0 –2.09	0.63
	Light air-cured	Md.	0 –4.00	1.34
Toxaphene	Fire-cured	Tenn.	0.26–0.51	0.40
		Va.	0.20–0.50	0.35
	Dark air-cured	Tenn.	0.52–5.78	1.69
		Va.	0.21–0.98	0.36
	Light air-cured	Md.	0.13–0.23	0.19
Endrin	Fire-cured	Tenn.	0.01–0.71	0.26
		Va.	0	<0.01
	Dark air-cured	Tenn.	0.06–0.30	0.17
		Va.	0	<0.01
	Light air-cured	Md.	0	<0.01

[a] Six samples were analyzed from each market.

decreased from an average of 10.6 ppm in 1970 (4) to 3.5 ppm in the present survey, some use of these insecticides was apparent. The only other sample with a DDT + TDE residue greater than 1 ppm was collected on a Virginia market.

Dark-air and fire-cured tobaccos from Tennessee had the highest endosulfan residues, and all the samples from this area contained residues of endosulfan over the tolerance of 5 ppm proposed by the Federal Republic of Germany. Endosulfan in one Virginia fire-cured tobacco also exceeded the proposed tolerance.

Endrin residues were not present or were found only in trace amounts except on samples from Tennessee. Fire and dark air-cured tobaccos from Tennessee averaged 0.26 and 0.17 ppm, respectively; endrin residues in two of the five samples were over 0.5 ppm.
Toxaphene residues were highest in Tennessee air-cured samples which averaged 1.69 ppm. Tennessee fire-cured samples averaged 0.4 ppm. No other sample of fire or air-cured tobacco had a toxaphene residue greater than 1 ppm.
Toxaphene and endrin are insecticides used on cotton. The residues found on tobacco may be due, at least in part, to drift during application of these insecticides to cotton planted in close proximity to tobacco fields.

CONCLUSIONS

The U.S. tobacco industry has taken positive steps to make U.S. tobacco acceptable in world markets, and the strong effort has had a marked effect in reducing the residue levels of chlorinated hydrocarbon insecticides. Except for isolated cases, tobacco farmers seem to have stopped using DDT and TDE. It is possible that some tobacco sampled in 1972 was grown in 1971 or before and stored by the farmer until the time of sale in 1972. Such tobacco could be a source of pesticide residues in the 1972 samples.
Compared with former crops the residue levels of DDT and TDE are now very low, but the imposition of a tolerance of 0.1 ppm as proposed by the Federal Republic of Germany (8) would certainly be unrealistic. Sixty-four percent of the flue-cured, 96% of the burley, and 83% of the fire and air-cured samples contained DDT + TDE at concentrations between 0.1 and 0.5 ppm. In view of recent reports (2, 11, 12, 13) showing uptake of DDT and other insecticides by the tobacco plant from the soil and assuming that residues of 0.5 ppm are the result of uptake from the soil, the question remains unanswered as to how long such residue levels will persist even without any treatment.

SUMMARY

Average residue levels of DDT + TDE in flue-cured tobacco decreased from 6.1 ppm in 1970 to 0.85 ppm in 1972. DDT + TDE residues in burley also dropped sharply from previous levels. In 1972 one sample from Kentucky contained 8.17 ppm; all other burley samples were less than 0.25 ppm. DDT + TDE residues also declined in fire-cured and air-cured types; of these samples Tennessee dark air-cured tobacco contained the highest average residue (3.5 ppm of DDT + TDE). In 1972 over 90% of the flue-cured samples were positive for toxaphene. Since each of our samples was a composite of tobacco from 10 farmers, we cannot conclude from this result that 90% of the individual piles contained toxaphene. Significant amounts of toxaphene were found in other types also; for example, 50% of the 1972 burley samples had toxaphene concentrations greater than 0.5 ppm. Average endosulfan levels decreased between 1970 and 1972 in flue-cured and burley tobaccos. However, in all of the dark air and dark fire-cured samples from Tennessee endosulfan residues exceeded 5 ppm. Average endrin residues were at or near the low detection limit in all samples except fire-cured and dark air-cured tobacco from Tennessee; these averaged 0.26 and 0.17 ppm, respectively.

ZUSAMMENFASSUNG

In „flue-cured"-Tabak verminderten sich die durchschnittlichen Rückstandsmengen an DDT+TDE von 6,1 ppm im Jahre 1970 auf 0,85 ppm im Jahre 1972. In Burley-Tabak gingen die Rückstände an DDT+TDE im Vergleich zu Befunden früherer Jahre ebenfalls stark zurück. 1972 enthielt eine Probe aus Kentucky 8,17 ppm; die Gehalte aller anderen Proben von Burley-Tabak waren kleiner als 0,25 ppm. Die Rückstände an DDT+TDE verringerten sich auch in „fire-cured"- und „air-cured"-Tabaken; bei diesen Proben hatte dunkler „air-cured"-Tabak aus Tennessee den höchsten Durchschnittswert (3,5 ppm DDT+TDE). Im Jahre 1972 war bei über 90% der Proben von „flue-cured"-Tabak Toxaphen nachweisbar. Da sich jede dieser Proben aus Lieferungen von zehn Anbauern zusammensetzte, kann aus diesem Ergebnis nicht geschlossen werden, daß 90% der einzelnen Lieferungspartien Toxaphen enthielt. Auch in anderen Tabakarten wurden signifikante Mengen Toxaphen gefunden, zum Beispiel wiesen die Proben der Burley-Tabake des Jahrgangs 1972 zu 50% Toxaphenmengen über 0,5 ppm auf. Die durchschnittlichen Endosulfangehalte von „flue-cured"- und Burley-Tabaken gingen zwischen 1970 und 1972 zurück. In allen dunklen „air-cured"- und dunklen „fire-cured"-Proben aus Tennessee lagen die Rückstände jedoch über 5 ppm. Im Durchschnitt lag die Höhe der Endrinrückstände in allen Proben – abgesehen von „fire-cured"- und dunklen „air-cured"-Tabaken aus Tennessee, deren Werte im Durchschnitt 0,26 bzw. 0,17 ppm betrugen – bei oder in der Nähe der unteren Nachweisgrenze.

RESUME

La teneur résiduelle moyenne de DDT+TDE dans les tabacs «flue-cured» a décru de 6,1 ppm en 1970 à 0,85 ppm en 1972. Les mêmes résidus ont egalement fortement diminué dans les tabacs Burley. En 1972, un échantillon de tabac du Kentucky contenait 8,17 ppm, alors que tous les autres échantillons de Burley restaient au-dessous de 0,25 ppm. Les résidus de DDT+TDE ont également décru dans les types «fire-cured» et «air-cured». Parmi ces échantillons, le tabac foncé «air-cured» du Tennessee contenait le plus grand résidu moyen (soit 3,5 ppm de DDT+TDE). En 1972, plus de 90% des échantillons «flue-cured» ont une réaction positive au toxaphène. Chacun de ces échantillons étant com-

posé du tabac de 10 planteurs, nous ne pouvons affirmer que 90% des tabacs individuels contiennent du toxaphène. D'autres variétés de tabac contiennent également de grandes quantités de toxaphène; par exemple, 50% des échantillons des tabacs Burley de l'année 1972 ont des résidus de toxaphène excédant 0,5 ppm. Les niveaux moyens d'endosulfan ont décru de 1970 à 1972 dans les tabacs «flue-cured» et Burley. Cependant dans tous les tabacs foncés «air-cured» et «fire-cured» provenant du Tennessee les résidus d'endosulfan dépassent 5 ppm. Dans tous les échantillons, les résidus moyens d'endrin se trouvent à la limite de détection ou au-dessous, exception faite des tabacs foncés «air-cured» et «fire-cured» du Tennessee. La teneur moyenne pour ceux-ci est de 0,17 et 0,26 ppm respectivement.

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