Total Toxic Aldicarb Residues in Soil, Cottonseed, and Cotton Lint Following a Soil Treatment with the Insecticide on the Texas High Plains

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Aldicarb residues in soil, cottonseed, and cotton lint following an in-furrow soil application of 15 lb/acre of a 10% granular formulation of the insecticide on the Texas High Plains were investigated by a gas chromatographic–flame photometric detection procedure. Aldicarb residues (as the sulfone) as high as 1.65 ppm (average of three laboratories) were detected in soil from dryland fields 3 days following application, decreasing to 0.24 ppm in 1 month and completely disappearing in 4 months. Residues in soil from irrigated fields averaged as much as 0.70 ppm 13 days after treatment and had completely disappeared in 42 days. Residues detected in postharvest cotton from dryland plots averaged 0.07 ppm in the seed and 0.05 ppm in the lint. Postharvest cotton from irrigated fields showed average residues of 0.01 ppm in the seed and 0.01 ppm in the lint. No significant residues were detected in soil between treated rows or in adjacent untreated areas.

In the Texas High Plains a 10% granular formulation of 2-methyl-2-(methylthio)propionaldehyde *O*-(methylcarbamoyl)oxime insecticide (registered as Temik by Union Carbide, also known as aldicarb and UC-21149) was tested in 1971 as a broad spectrum insecticide for the control of a wide variety of cotton pests. Although the systemic action and metabolism of labeled aldicarb in cotton plants have been investigated by Metcalf *et al.* (1966), Coppedge *et al.* (1967), Bull (1968), and Bartley *et al.* (1970), only limited data are available on residues in cottonseed and lint. Information is available on persistence of aldicarb and metabolites in soil (Andrawes *et al.*, 1971; Bull, 1968; Bull *et al.*, 1970; Coppedge *et al.*, 1967), but very little pertaining to movement of the insecticide to adjacent untreated soil is available.

This is a report on residues of aldicarb in soil, cottonseed, and cotton lint following a soil application of the pesticide and in soil from untreated areas.

EXPERIMENTAL SECTION

Type of Soil and Application Rate. A single application of aldicarb 10G was applied to sandy loam soil at the rate of 15 lb per acre (1.5 lb active ingredients) to irrigated and nonirrigated fields utilizing precalibrated Gandy granular applicators. Modified anhydrous ammonia chisels were used to open furrows for the insecticide granules and the Temik was placed 6 to 8 in. to the side and 5 to 6 in. deep around the cotton, which was in the seedling stage.

Sampling Procedure. *Soil.* Soil samples, *ca.* 6 in. deep, were randomly collected from cotton fields on three different dates throughout the season with a core-type sampler. Samples were collected from the treated fields, in and between rows. Control samples were randomly collected from untreated fields. Approximately 50 cores were collected from each field.

In order to detect movement of the pesticide from treated fields, soil samples were collected from a creek bottom adjacent to a treated field and also from areas 0.25 mile and 1 mile downstream from a treated field. The same sampling procedure was utilized in collecting these samples as was used for the soil samples from the cotton fields.

Cotton. Seed cotton was collected from treated and untreated fields as bolls matured. No particular system was utilized in collecting cotton samples.

Analytical Procedures. The analytical methods of Union Carbide Chemicals Corporation (1970) were used in this investigation with modifications to improve sensitivity and shorten the procedure. The technique involved the oxidization of aldicarb and its sulfoxide to the sulfone followed by a Florisil (Floridin Co., Philadelphia, Pa.) column cleanup and isolation step to remove interfering compounds and nontoxic oximes formed during the oxidization procedure. The subsequent analysis was performed on a gas-liquid chromatograph equipped with a Melpar flame photometric detector (FPD) (Tracor, Inc., Austin, Tex.) utilizing a sulfur filter. Thin-layer chromatography (tlc), as reported by Bull *et al.* (1967), was employed to confirm some of the larger sulfone peaks.

Extraction. Soil samples were placed on a clean sheet of aluminum foil, mixed thoroughly, and representative 300-g samples were weighed into half-gallon Mason jars. Six-hundred milliliters of a solvent mixture of 1:1 acetone (ACS grade) and distilled water was added to each jar; the jars were sealed with a screw cap and Teflon liner and rotated on a concentric rotator for 2 hr. After rotating, the samples were allowed to stand at least 3 hr, and were then filtered through glass wool into 500-ml graduated cylinders, collecting 300-ml aliquots for analysis. The extracts, representing 150 g of soil, were then transferred to 500-ml Erlenmeyer flasks and stored pending the oxidization step.

Cottonseed was separated from the lint by processing through a small cotton gin (Continental Moss-Gordon, Prattville, Ala.). Untreated samples were ginned first and the machine was thoroughly cleaned between treated samples. Representative 50-g cottonseed samples were weighed into blender jars, 150 ml of a 50% aqueous acetone (ACS grade) mixture was added, the samples were blended for *ca.* 3 min at medium speed, and the macerate was transferred to half-gallon Mason jars. Another fresh 150-ml portion of the solvent mixture was added to the blender jars and again mixed for *ca.* 1 min to rinse the jars. This procedure was repeated with 150 ml of fresh solvent mixture and the jars were finally rinsed with the same amount of fresh solvent mixture. All of the rinsings were transferred to the Mason jars, and the jars were sealed tightly with screw caps and Teflon liners and rotated on a concentric rotator for 2 hr. The extracts were filtered through glass wool into 500-ml graduated cylinders; 300-ml aliquots were collected and transferred to 500-ml flasks for the oxidization step.

Cotton lint was finely cut with scissors and then 25-g

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samples were weighed into half-gallon Mason jars. Eight-hundred milliliters of a 1:1 ACS grade acetone–distilled water mixture was added and the jars were sealed loosely with screw caps and Teflon liners. The jars were stored overnight in a 10° cooler to allow better penetration of the solvent into the fibers. After the samples warmed to room temperature, the jars were sealed tightly and rotated on a concentric rotator for 4 hr. The extracts were filtered through glass wool into 1000-ml graduated cylinders; 600-ml aliquots were collected and transferred to 1000-ml flasks for the oxidization step.

Oxidization. The procedures utilized for the soil, cottonseed, and cotton lint were essentially the same, with a few minor modifications. These changes were necessary due to the difference in reactions of the samples. The procedures were performed as described below.

Soil extracts were oxidized by adding 5 ml of 40% peracetic acid (Pfaltz and Bauer, Inc., Flushing, N. Y.) and Teflon encased magnetic stirring bars to each sample, stirred continuously while adding an additional 3 ml of the peracetic acid, and stirred again for 30 min. Eighty milliliters of a 10% (w/v) aqueous sodium bicarbonate solution was carefully added to each flask with continued stirring. The extracts were stirred for an additional 30 min to neutralize the excess acid. The extracts were then transferred to 500-ml separatory funnels and the flasks were rinsed with 50-ml portions of ACS grade chloroform, transferring the rinsings to the separatory funnel. The funnels were shaken for 30 sec, venting to release carbon dioxide pressure, and the lower solvent layers were filtered through a layer of *ca.* 150 g of anhydrous sodium sulfate into 500-ml Erlenmeyer flasks. The aqueous layers were extracted an additional three times as above, with fresh 50-ml portions of chloroform, draining each extraction through the sodium sulfate into the flask. After the fourth extraction, the sodium sulfate bed was washed with 50 ml of chloroform into the flask. The flasks were then immersed in a 40–50° water bath and evaporated to dryness with a gentle stream of dry air. The residues were then redissolved in 100 ml of chloroform and stored pending the isolation and cleanup procedure.

Cottonseed extracts were carried through essentially the same oxidization procedure as the soil extracts. However, 4.0 ml of peracetic acid was used and 40 ml of sodium bicarbonate solution was added to neutralize the excess acid after oxidization. The chloroform extraction step was also essentially the same, except the last three extractions were made with 100-ml portions of chloroform, the solvent was evaporated to *ca.* 3 ml in a warm (40–50°) water bath with a gentle stream of dry air, and the residue was redissolved in 50 ml of chloroform.

The cotton lint samples were oxidized and extracted as described for cottonseed, except 7.0 ml of the peracetic acid was used and 70 ml of the 10% aqueous sodium bicarbonate was used to neutralize the excess acid following the oxidization procedure. One 100-ml portion, then three 50-ml portions of chloroform were used to extract the sulfone from the aqueous layer in a 1-l. separatory funnel. The chloroform was evaporated to dryness in a warm (40–50°) water bath with a gentle stream of dry air; then the residue was redissolved in 50 ml of chloroform.

Cleanup and Isolation of Residues. Many nontoxic oximes of aldicarb are formed in the weathering process and in the oxidization procedure. These and other extraneous materials must be removed, since they interfere in the gas–liquid chromatography (glc) procedure. The following procedures discussed were used to accomplish this.

All of the soil, cottonseed, and lint samples were processed utilizing varied amounts of unactivated 60/100 mesh Florisil packed in a chromatographic column with the following dimensions: i.d., 10 mm; length, 630 mm, equipped with No. 2A Teflon stopcocks and 250-ml reservoirs. The activity of the Florisil was checked periodically to determine if the aldicarb sulfone appeared in the correct fraction.

Soil. The columns were prepared by placing a plug of glass wool in the bottom and weighing exactly 5.0 g of the unactivated Florisil into the columns with tapping to settle the adsorbent, which was then prewet with 25 ml of chloroform. When the solvent level reached the top layer of the adsorbent, the oxidized sample was transferred to the column and allowed to elute dropwise. The sample flask was rinsed with 100 ml of a 4% acetone: 96% diethyl ether solvent mixture; the rinsings were then transferred to the column and allowed to elute dropwise. When the solvent level reached the top of the adsorbent layer, the stopcocks were closed and receivers changed. The first two eluates were discarded. One-hundred-and-fifty milliliters of a 1:1 (v/v) acetone–diethyl ether mixture was added to the column, eluted dropwise, and collected in 250-ml Erlenmeyer flasks. This fraction contained the aldicarb sulfone. The flasks were then immersed in a 40–50° warm-water bath and the solvent was evaporated to dryness with a gentle stream of dry air. The residues were redissolved in *ca.* 5 ml of benzene and transferred to 15-ml centrifuge tubes. The flasks were rinsed two additional times with *ca.* 5-ml fresh portions of benzene; the rinsings were transferred to the tubes after each rinse. The benzene was evaporated to 1.0 ml in a 40–50° water bath to ensure complete removal of the other solvents for the glc–FPD analysis.

Cottonseed. Chromatographic columns were prepared as described in the previous section with the following exceptions. 4.0 g of the deactivated Florisil was used. The Florisil was prewet with 15 ml of benzene. The 50 ml of chloroform extracts was transferred to the columns and eluted dropwise as above. The second elution was made with 100 ml of a 2% acetone–diethyl ether solvent mixture and the eluate was discarded. The third elution was made with 50 ml of a 10% acetone:90% diethyl ether solvent mixture and the eluate was discarded. A fourth elution was made with 100 ml of a 20% acetone:80% diethyl ether solvent mixture. This eluate which contained the aldicarb sulfone was retained for glc analysis.

The purified extracts were evaporated essentially as described previously, except the solvent was evaporated to *ca.* 1 ml and then transferred to centrifuge tubes and evaporated as before.

Lint. Chromatographic columns were prepared as described previously for cottonseed. After transferring the oxidized sample to the column and eluting dropwise through the column, the following changes in the elution procedure were made.

Flasks were rinsed with 15 ml of benzene, transferred to the columns, and eluted dropwise with 50 ml of a 20% acetone:80% diethyl ether solvent mixture. The first two eluates were discarded.

The receivers were then changed to clean, dry 250-ml Erlenmeyer flasks, 100 ml of a 20% acetone:80% diethyl ether solvent mixture was added to the columns, the stopcocks were opened, and the solvent was eluted dropwise through the columns. This fraction contained the aldicarb sulfone. The evaporation procedure was the same as described previously, except the eluates were evaporated to *ca.* 0.5 ml, transferred to centrifuge tubes with benzene, and diluted or concentrated to the desired volume for the glc–FPD analysis.

Gas–liquid chromatographic analyses were made on a Tracor Model MT-220 Gas Chromatograph equipped with a Melpar Flame Photometric Detector (FPD), utilizing a 394-mμ sulfur interference filter. A ¼ in. × 12-ft aluminum column packed with 5% Carbowax 20M on 60/80 mesh Gas Chrom Q (Applied Science, State College, Pa.) was used for the cottonseed and lint samples; the same was used for soil, except a 3% Carbowax column was used. The injector, detector, and column were maintained at

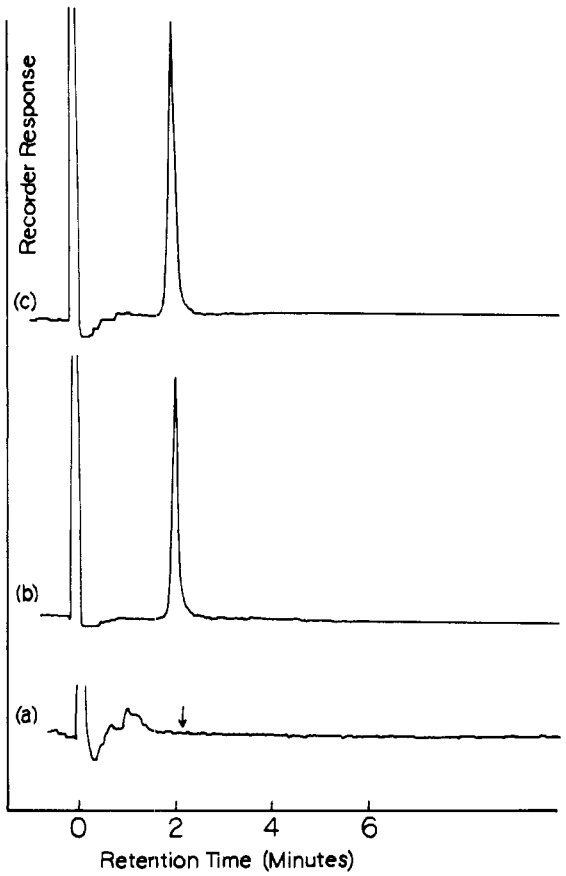

Figure 1. Gas chromatographic spectra of (a) untreated soil, (b) aldicarb sulfone standard, 10.42 ng, and (c) soil fortified with 0.19 ppm of aldicarb on a Carbowax column. All residues were oxidized to the sulfone before chromatography. See Gas Chromatographic Analysis section for complete operating parameters.

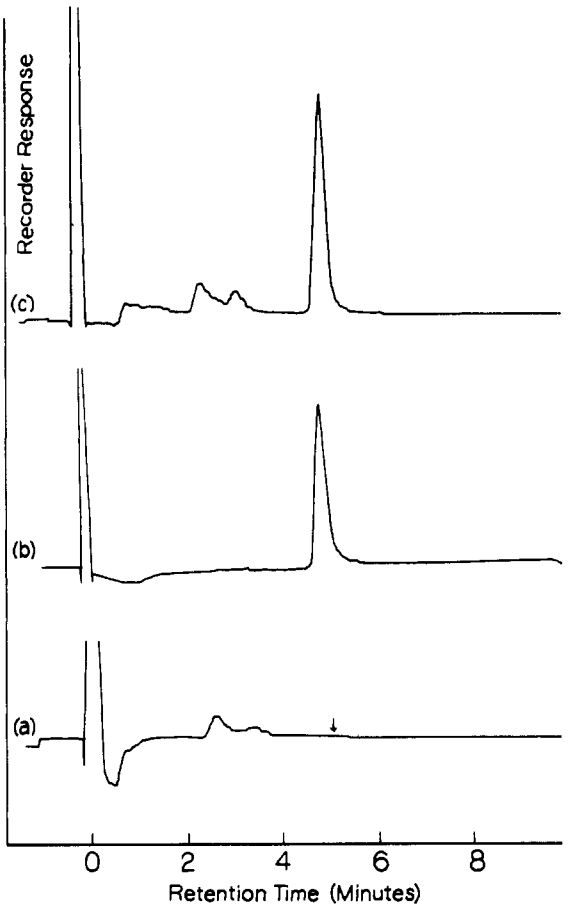

Figure 2. Gas chromatographic spectra of (a) untreated cottonseed, (b) aldicarb sulfone standard, 10.42 ng, and (c) cottonseed fortified with 0.11 ppm of aldicarb on a Carbowax column. All residues were oxidized to the sulfone before chromatography. See Gas Chromatographic Analysis section for complete operating parameters.

250, 200, and 200°, respectively, for the 5% column and 300, 160, and 160° were maintained for the 3% column. Hydrogen, nitrogen, oxygen, and air flow rates were 100, 57, 15, and 50 ml/min, which produced maximum response for the sulfone on the gas chromatograph. Electrometer setting was adjusted to obtain half-scale deflection with a 10-ng injection of an aldicarb sulfone standard. Recorder chart speed was 30 in./hr.

Glass inserts in the column inlet were replaced with metal inserts lightly packed with silanized glass wool. The high inlet temperatures and metal inserts were utilized to promote thermal breakdown of the sulfone to the nitrile.

A series of control samples consisting of solvent check, untreated sample material, and aldicarb fortified sample material was carried through the entire procedure along with the unknown samples. Quantitation was based on peak heights of the sulfone peak, once linearity was ascertained. Average recovery value of the fortified soil was 93.5%, with a range of 90.0 to 97.5%; recoveries were 95.5% for the cottonseed and 90.3% for the cotton lint. Only one recovery value was determined for the cotton lint and seed because of the small number of samples analyzed. All residues reported were corrected for these values. No interfering peaks were detected in the solvents, reagents, or blank sample material.

RESULTS

Figure 1 depicts chromatographic tracings obtained from (a) untreated soil, (b) aldicarb sulfone standard (10.42 ng), and (c) soil fortified with 0.19 ppm of aldicarb and carried through the entire procedure.

Figure 2 shows chromatographic tracings from (a) untreated cottonseed, (b) aldicarb sulfone standard (10.42 ng), and (c) cottonseed fortified with 0.11 ppm of the pesticide and carried through the extraction, cleanup, and glc analytical procedures.

Figure 3 is a chromatographic tracing of (a) untreated cotton lint, (b) aldicarb sulfone standard (10.42 ng), and (c) cotton lint fortified with 0.10 ppm of the pesticide and processed in the same manner as the unknown samples.

The lower limits of sensitivity utilizing this procedure were determined to be 0.01 ppm for soil in the Brownsville and Gulfport Laboratories and 0.02 ppm for Union Carbide. For cottonseed and lint, the lower limits were 0.01 ppm for Brownsville, 0.02 ppm for Union Carbide, and 0.05 ppm for the Gulfport Laboratory. No materials contributing interfering peaks were detected in any of the "blank" sample material.

Table I presents residue data on the accumulation and disappearance of aldicarb and metabolites in treated soils at various intervals after soil application of this pesticide. Some of the soil samples were also analyzed by the Environmental Protection Agency's Monitoring Laboratory in Gulfport, Miss., and Union Carbide's Residue Laboratory at South Charleston, W. Va. The results of these analyses are also listed in this table.

Residues of aldicarb and metabolites in soil were averaged for the three laboratories and for row samples from dryland fields 1 and 4. An average of 0.99 ppm was obtained with a range of 0.23 to 1.65 ppm. Results from the three laboratories were also averaged for soil samples from irrigated fields, but an average could not be made of the various fields since different sampling intervals were involved. Average residues for row samples from field no. 2 were 0.10 ppm 18 days following application, completely

Table I. Total Toxic Aldicarb Residues (as Temik Sulfone) in Soil Treated with Aldicarb 10G in the Texas High Plains (1971)

Field no.	Sampling location	Treatment date	Sampling date	Residue, ppm[a] Brownsville	Union Carbide	Gulfport
			Dryland			
1	Row	6/30/71	7/2/71	0.23	0.50	0.26
	Row	6/30/71	7/27/71	0.05		
	Row	6/30/71	11/22/71	0.00		0.00
	Middle	6/30/71	7/2/71	0.00	0.02[b]	
	Middle	6/30/71	7/27/71	0.00		0.00
	Middle	6/30/71	11/22/71	0.00		0.00
4	Row	6/25/71	6/28/71	1.49	1.80	1.65
	Row	6/25/71	7/27/71	0.39		0.10
	Row	6/25/71	11/22/71	0.00		0.00
	Middle	6/25/71	6/28/71	0.00	<0.02	
	Middle	6/25/71	7/27/71	0.00		
	Middle	6/25/71	11/22/71	0.00		
7		Untreated	6/25/71	0.00	<0.02	0.00
		Untreated	7/27/71	0.00		0.00
		Untreated	11/22/71	0.00		
			Irrigated			
2	Row	6/10/71	6/28/71	0.07	0.12	0.10
	Row	6/10/71	11/22/71	0.00		0.00
	Middle	6/10/71	6/28/71	0.00	<0.02	
	Middle	6/10/71	11/22/71	0.00		0.00
3	Row	6/16/71	6/28/71	0.43	0.37	0.27
	Row	6/16/71	7/27/71	0.06		0.00
	Row	6/16/71	11/22/71	0.00		0.00
	Middle	6/16/71	6/28/71	0.00	<0.02	
	Middle	6/16/71	7/27/71	0.00		
	Middle	6/16/71	11/22/71	0.00		0.00
	East Creek[c]	6/16/71	7/14/71	0.00		
	East Creek[d]	6/16/71	7/14/71	0.00		
	Creek Bottom[e]	6/16/71	7/14/71	0.00		
5	Row	6/15/71	6/28/71	0.78	0.78	0.54
	Row	6/15/71	7/27/71	0.00		0.00
	Row	6/15/71	11/22/71	0.00		0.00
	Middle	6/15/71	6/28/71	0.00	<0.02	0.00
	Middle	6/15/71	7/27/71	0.00		
	Middle	6/15/71	11/22/71	0.00		0.01[b]
6	Row	6/14/71	6/25/71	0.35	0.61	0.28
	Row	6/14/71	7/20/71	0.06		
	Row	6/14/71	7/26/71	0.00		
	Row	6/14/71	11/22/71	0.00		0.00
	Middle	6/14/71	6/25/71	0.00	<0.02	
	Middle	6/14/71	7/20/71	0.00		
	Middle	6/14/71	7/26/71	0.00		
	Middle	6/14/71	11/22/71	0.00		0.00
8		Untreated	6/25/71	0.00	<0.02	
		Untreated	7/26/71	0.00		0.00
		Untreated	11/22/71	0.00		0.00

[a] All residues were corrected for moisture content and for aldicarb recovery from fortified samples. [b] Lower limit of sensitivity. [c] One-fourth mile down creek from Field no. 3. [d] One mile down creek from Field no. 3. [e] Even with and North of Field no. 3.

disappearing in 4 months. Field no. 3 showed average residues of 0.36 ppm 12 days after treatment, declining to 0.03 ppm after 47 days, and disappearing completely in 4 months. Average residues of 0.70 ppm were detected in row samples from field no. 5 after 13 days; no residues were detected in the 42-day and 4-month samples. In row samples from field no. 6, average residues were 0.41 ppm 14 days following treatment, 0.06 ppm after 36 days, and no residues after 4 months. Average residues found in soil between rows of both dryland and irrigated fields and in soil from adjacent untreated areas were below the detectable limits of the procedure. One laboratory reported 0.02 ppm, their lower limits of detectability, in one soil sample collected between treated rows 3 days following treatment; the remaining samplings showed no detectable residues.

Table II presents data on the accumulation of aldicarb and metabolites in cottonseed and lint collected from cotton plants grown in irrigated and dryland fields treated with the insecticide. Residues (average of the three laboratories) in the lint ranged from 0.01 (irrigated) to 0.05 ppm in cotton from a field receiving no irrigation. Cottonseed samples showed essentially identical residues, ranging from 0.01 ppm in an irrigated field to 0.07 ppm in cotton from a nonirrigated field.

DISCUSSION

Aldicarb was found to be a compound of short residual life. One month following treatment, average soil residues in treated rows decreased to approximately 15% of residues detected 3 days after treatment of dryland fields. In one irrigated field, average residues detected in soil from

Table II. Total Toxic Aldicarb Residues (as Temik Sulfone) in Cotton Lint and Seed of Cotton Plants Grown in Soil Treated with Aldicarb 10G

Sample no.	Sampling date	Type of land	Residue, ppm[a]					
			Lint			Seed		
			Browns-ville	Gulf-port	Union Carbide	Browns-ville	Gulf-port	Union Carbide
1	9/30/71	Dry	0.16	0.00	0.14	0.19	0.10	0.12
2	9/30/71	Dry	0.08	0.00	0.17	0.07	<0.05	0.21
3	9/30/71	Dry	0.00	0.00	<0.02	0.00	0.00	<0.01
4	9/30/71	Dry	0.05	0.00	0.04	0.04	<0.05	0.04
5	9/30/71	Dry	0.03	0.00	0.07	0.09	0.05[b]	0.08
		Avg	0.06	0.00	0.08	0.08	0.04	0.09
6	9/30/71	Irrigated	0.08	0.00	0.05	0.06	<0.05	0.04
7	9/30/71	Irrigated	0.02	0.00	0.02[b]	0.02	0.00	0.02[b]
8	9/30/71	Irrigated	<0.01[b]	0.00	0.02[b]	<0.01[b]	0.00	<0.02
9	9/30/71	Irrigated	0.00	0.00	<0.02	<0.01[b]	0.00	<0.02
10	9/30/71		0.00	0.00	<0.02	0.00	0.00	<0.02
		Avg	0.01	0.00	0.02	0.02	0.00	0.02

[a] Corrected for aldicarb recovery from fortified samples. [b] Lower limit of sensitivity.

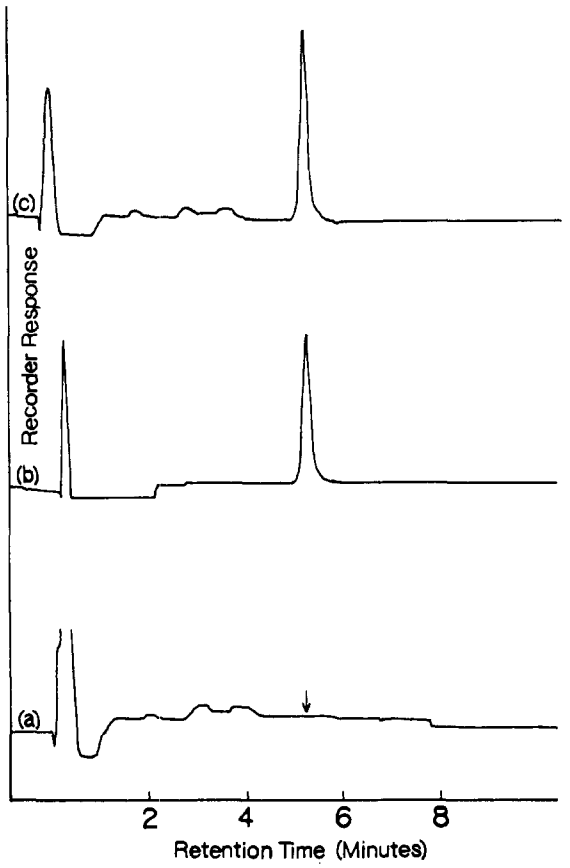

Figure 3. Gas chromatographic spectra of (a) untreated cotton lint, (b) aldicarb sulfone standard, 10.42 ng, and (c) lint fortified with 0.10 ppm of aldicarb on a Carbowax column. All residues were oxidized to the sulfone before chromatography. See Gas Chromatographic Analysis section for complete operating parameters.

treated rows 47 days after treatment declined to approximately 8% of residues found in soil samples collected 12 days following application. Average residues in soil from treated rows in a second irrigated field showed no detectable residues 42 days after application. Four-month post-treatment soil samples showed no detectable residues in either dryland or irrigated fields. No significant movement of aldicarb and metabolites was noted, as indicated by the lack of detectable residues in soil collected between treated rows or in soil from untreated areas a greater distance from treated fields. Evidence of systemic action was indicated by the detection of aldicarb and/or metabolites in postharvest samples of cottonseed and lint.

On the basis of the results obtained in these tests, it was concluded that residues of aldicarb and metabolites do not persist for long periods of time and therefore would not carry over from one growing season to another. There was little lateral movement of the pesticide through the soil. Irrigation would be a factor only in causing a more rapid disappearance of residues, but not contributing to any appreciable movement of the pesticide and/or metabolites to adjacent untreated areas.

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LITERATURE CITED

Andrawes, N. R., Bagley, W. P., Herrett, R. A., *J. Agr. Food Chem.* **19**, 731 (1971).
Bartley, W. J., Andrawes, N. R., Chancey, E. L., Bagley, W. P., Spurr, H. W., *J. Agr. Food Chem.* **18**, 446 (1970).
Bull, D. L., *J. Econ Entomol.* **61**, 1598 (1968).
Bull, D. L., Lindquist, D. A., Coppedge, J. R., *J. Agr. Food Chem.* **15**, 610 (1967).
Bull, D. L., Stokes, R. A., Coppedge, J. R., Ridgeway, R. L., *J. Econ. Entomol.* **63**, 1283 (1970).
Coppedge, J. R., Lindquist, D. A., Bull, D. L., Dorough, H. W., *J. Agr. Food Chem.* **15**, 902 (1967).
Metcalf, R. L., Fukuto, T. R., Collins, C., Borck, J., Burk, J., Reynolds, H. T., Osman, M. F., *J. Agr. Food Chem.* **14**; 579 (1966).
Union Carbide Corporation, "Aldicarb-FPD-Soil and Aldicarb-FPD-Cots" Analytical Methods Series, 1970.

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