Table IV. Agreement in Endrin Content of Fat Samples Analyzed by Spectrophotometric and Bioassay Methods

Sample	Endrin in Diet, P.P.M.	Endrin Content, P.P.M. Bio-assay	Spectrophotometric
Steer, body fat	0.00	<0.02	<0.1
	0.10	0.17	<0.1
	0.25	0.20	0.2
	0.75	0.34	0.4
Lamb, body fat	0.00	<0.02	<0.1
	0.10	0.04	<0.1
	0.25	0.08	0.1
Hog, body fat	0.10	0.02	<0.1
	0.25	0.09	0.1
	0.75	0.22	0.1
Steer, body fat, after feedoff	2.00	0.47	0.3
Lamb, body fat, after feedoff	0.75	0.07	<0.1

weeks was sufficient to allow elimination of the endrin to levels below those detectable by the specific method in the case of hogs and lambs.

Limited data obtained with the confirmatory bioassay indicated that small amounts—less than 0.1 p.p.m.—of endrin were deposited in the tissues at all levels of endrin intake.

The endrin content of roasts and steaks is given in Table III. Only the higher levels of endrin intake resulted in a deposition in this type of tissue in the case of steers and hogs. No detectable endrin was present in steaks and roasts of lambs. Fat determination made on steer steaks and roasts showed that steaks had a higher fat content which may account for the higher residue shown in Table III. In confirmation of this, a steak and a roast from a steer receiving 2.00 p.p.m. of endrin were divided into gross fat and protein fractions and analyzed separately. Practically all of the endrin—nearly 1.0 p.p.m.—was located in the fat.

All meat cuts which appeared to contain significant amounts of endrin at the end of the 12-week feeding period were analyzed again after cooking. The results indicated little or no decrease in endrin content. An increase was actually noted in several cases, possibly owing to the loss of moisture during cooking.

Of all liver and kidney tissues examined, only the liver tissue of steers receiving the two higher levels of endrin showed definite amounts of endrin at the end of the 12-week feeding period (Table III).

The analyses by the biological method were included in the study as a means of detecting any toxic materials produced by the metabolism of the endrin. Data in Table IV reveal that there are no consistent differences between the two sets of analytical results—good evidence that no metabolites more toxic than endrin are produced.

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INSECTICIDE RESIDUES

Endrin Content of Milk and Body Tissues of Dairy Cows Receiving Endrin Daily in Their Diet

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Dairy cows were given daily doses of endrin ranging from 0.1 to 2.00 p.p.m. total dietary concentration for 12 weeks. During and after the endrin intake period, milk samples were analyzed for endrin residues. Various tissue samples were also analyzed for endrin content at the end of the 12-week period. Small amounts of endrin were secreted in milk at all levels of intake. Concentrations of endrin up to 1.00 p.p.m. were found in the body fat.

SPRAY RESIDUES remaining on forage crops at the time they are eaten by livestock constitute a potential hazard to the consumer of milk and meat products. Before any material can be considered for such uses, information on its storage in animal tissues and its secretion in milk must be obtained.

Bateman and coworkers (*3*) studied the fate of toxaphene consumed by dairy cows and found residues in milk in concentrations ranging from 2 to 13 p.p.m. Biddulph *et al.* (*4*) and Shepard *et al.* (*11*) found that cows eating DDT-treated forage secrete DDT in milk and store it in fatty tissues. Harris *et al.* (*8*) and Ely *et al.* (*6*) obtained similar results with dieldrin. Davidow, Radomski, and Ely (*5*), Harris *et al.* (*8*), and Ely *et al.* (*7*) demonstrated the presence of heptachlor epoxide, a toxic metabolite of heptachlor, in the milk of cows receiving this insecticide. The following report describes experiments

in which endrin (1,2,3,4,10,10 - hexachloro - 6,7 - epoxy - 1,4,4*a*,5,6,7,8,8*a*-octahydro - 1,4 - *endo* - *endo* - 5,8 - dimethanonaphthalene), a chlorinated hydrocarbon, was incorporated in the diet of dairy cows, followed by analyses for endrin residues in the milk and eight types of body tissues. A more complete report listing all relevant data has been prepared in mimeograph form (*12*).

Experimental

The dairy cows, 8 Jerseys and 6 Guernseys, were obtained from local dairy herds and from the college farm. They were fed from individual mangers and housed in a modern dairy barn. Except for an exercise period each day, the cows were kept stanchioned throughout the experiment.

The ration consisted of mixed grass and alfalfa hay and a grain mixture of ground oats and barley with 2% salt and 1% of bone meal added. Sufficient feedstuffs were allotted to last through the entire experiment. Endrin analyses made on the various components of the ration indicated that no residual endrin was present.

The lower levels of endrin fed were of the same order of magnitude as those expected from actual field use of endrin on forage crops. To increase the likelihood that positive results would be obtained, endrin levels above those expected to occur in the field were included in the feeding experiment. The concentrations fed, therefore, were 0.0, 0.1, 0.25, 0.75, and 2.00 p.p.m.

Rather than attempt to accurately contaminate the entire ration each day, the toxicant was added to the grain ration in quantities equivalent to those which would be consumed, if the entire ration were contaminated. The grain was weighed and fed just prior to the evening milking. The toxicant, in acetone solution, was distributed over the grain in numbered pails assigned to each cow. The grain was always consumed by each animal. A glass syringe, graduated in milliliters, was used to measure the endrin solution. These solutions were prepared so that the desired level of fortification could be attained by adding 1 ml. of solution per pound of feed. Two cows were used as controls, three received endrin at 0.1-p.p.m., four at 0.25-p.p.m., three at 0.75-p.p.m., and two at the 2.00-p.p.m. level.

Throughout the experiment, records were kept on feed consumption, milk production, butterfat content, and body weights. Milk samples were taken 3 days prior to the beginning of the feeding experiment, 3 days after feeding started, and again at 1, 2, 4, 8, and 12 weeks, at which time endrin feeding was stopped. Further samples were taken 4 and 6 weeks after this date. On the day milk samples were scheduled, the 24-hour milk production of each cow was collected in numbered milk cans. The freshly drawn milk was weighed, well mixed, and a subsample was withdrawn for butterfat determination. Between the morning and evening milkings, the milk was kept in ice water storage. A representative 2-quart sample for endrin analysis was taken and stored in Pliofilm bags inside paper cartons at below zero temperatures.

At the end of the 12th week of endrin feeding, eight cows were slaughtered and their tissues sampled. These included one control cow, two on the 0.1-p.p.m., two on the 0.25-p.p.m., two on the 0.75-p.p.m., and one on the 2.00-p.p.m. feeding level. The remaining cows were continued on an endrin-free diet for 6 weeks before slaughtering, and milk samples were taken at 4- and 6-week intervals. Tissue samples taken for endrin analysis consisted of brain, heart, liver, kidney, renal fat, body fat, steak, and roast. The latter meat cuts were typical round steaks and shoulder roasts, respectively. Attempts were made to make all tissue samples as representative as possible. Portions of both kidneys were used and renal fat was taken from both sides of the carcass. Body fat was obtained from the various areas of deposition over the outside of the carcass. The agreement between duplicates and between the two methods of analysis indicates that these attempts were successful.

Endrin Analysis. All of the milk and body tissue samples were analyzed by a spectrophotometric method for endrin as described by Bann *et al.* (*2*). This method involves refluxing of tissue samples in strong alkali to saponify the fats present, chromatography of the fat-free extracts to remove additional interferences, and the development and measurement of the color characteristic of endrin. The sensitivity of the specific method was 0.01 p.p.m. for the 600-gram milk samples and 0.1 p.p.m. for the 50-gram tissue samples.

To determine whether endrin was converted to toxic metabolites not detected by the specific method, about 20% of the samples were analyzed by a nonspecific biological assay method using mosquito larvae. Samples were saponified and chromatographed in the same way as those prepared for the spectrophotometric method. The mosquito larvae were then exposed to graded amounts of the extracts and the resulting mortalities compared with those encountered with known concentrations of endrin. The sensitivity of the bioassay method was considered to be 0.002 p.p.m. for milk and 0.02 p.p.m. for tissue samples. The bioassay procedure as described would not measure metabolites that were unstable to alkali or removed by the chromatography.

To validate the two analytical methods, control samples were analyzed frequently with and without fortification with endrin. The recoveries under these circumstances were about 80%.

Results and Discussion

Effect of Endrin Feeding on Experimental Animals. All cows receiving endrin gained weight. Their physical appearance during the experiment was that of well-fed dairy cattle. Milk production was normal under the conditions imposed.

Results of Analyses. The results of the milk analyses are listed in Table I. A measurable concentration of endrin in the milk of cows receiving daily diets containing endrin at 0.25 p.p.m. and above is indicated. The secretion of endrin began to be apparent within 1 week after intake started and except for the 2.00-p.p.m. intake level, had disappeared from the milk within 1 month after intake ceased. An increase in the dietary level of endrin did not result in a corresponding increase in endrin secretion. Furthermore, within the limits imposed by this study, the endrin content of milk reaches a plateau within a month after intake begins and remains relatively close to this level for the remainder of the exposure.

The bioassay results are listed in Table II. According to this more sensitive method, some detectable endrin is present even at the lowest level of intake. These results confirm those obtained on the duplicate samples by the specific method.

Table III lists the results of the analyses of the tissue samples. Measurable quantities of endrin were found in the fat tissue at the 0.25-p.p.m. and higher dietary intake levels, with the maximum concentration being 1 p.p.m. At the end of the endrin-free diet (feed off) period, all of the samples which had contained endrin were back to normal, indicating a rapid turnover of the stored insecticide. Of the other tissues analyzed, only liver and roast from the two higher level diets contained definite amounts of endrin at the end of 12 weeks.

In Table IV, some analyses of various tissue samples use both bioassay and the specific method; these show good agreement.

The insecticide content of milk fat and of body fat of dairy cows receiving insecticide in their diet are expected to be about the same. As the average milk fat content of the milk of cows involved in this study was 5.3%, the ratio of insecticide in milk *vs.* the insecticide in body fat should be 1 to 19. The actual ratio varied from 1 to 5 to 1 to 20, with an average of 1 to 12.

One of the complications which might arise in the ingestion of insecticide residues by livestock is the conversion of the original insecticide to another toxic

Table I. Spectrophotometric Determination of Endrin Content of Milk from Treated Cows

Endrin in Diet, P.P.M.	No. of Animals[a]	Endrin Content of Milk, P.P.M.[b]								
		Before endrin fed	During Endrin Feeding					After Endrin Feeding Ceased		
			3/7 wk.	1 wk.	2 wk.	4 wk.	8 wk.	12 wk.	4 wk.	6 wk.
0.00	2 (1)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
0.10	3 (1)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
0.25	4 (2)	<0.01	<0.01	0.01	<0.01	0.01	0.02	0.02	<0.01	<0.01
0.75	3 (1)	<0.01	<0.01	0.01	0.01	0.02	0.04	0.02	<0.01	<0.01
2.00	2 (1)	<0.01	0.01	0.07	0.08	0.10	0.10	0.08	<0.01	0.03

[a] Numbers in parentheses refer to numbers of animals retained for feedoff studies.
[b] Data corrected for apparent endrin content of control samples and rounded to nearest 0.01 p.p.m. Values below this level are indicated.

material by the animal's body metabolism. Such a conversion has been noted in the case of heptachlor, where an epoxide analog has been found deposited in the fat (*8*) and secreted in the milk (*5*) of animals ingesting heptachlor. This was confirmed by Ely *et al.* (*7*), using a similar heptachlor intake level. These authors did not find epoxide in the milk when field residue levels were fed, although another group of investigators (*8*) have shown that even low levels of heptachlor result in the secretion of heptachlor epoxide in milk. Another example of the epoxidation is the conversion of aldrin to dieldrin in the body of some animals (*1*). Apparently, toxic metabolites are not formed when endrin is ingested by dairy cows, as confirmatory analyses of a representative portion of the milk and tissue samples by a biological assay method did not reveal residues of greater magnitude than those indicated by the specific method.

The rate of output of an insecticide in milk is an important consideration in selecting an insecticide for forage crop treatments. An insecticide that has less tendency to be secreted in milk certainly presents a potentially less health hazard than one that is secreted rapidly. Endrin is secreted at a lower level than dieldrin or heptachlor, although there is some contradictory evidence for the latter insecticide (Table V).

Endrin is accumulated in the fat of small animals (rats) at a rather rapid rate (*10*). At 1-p.p.m. dietary level, the ratio of storage *vs.* intake is 15 to 1. This ratio decreases with increasing intake. The current study indicates that in the dairy cows the ratio of storage in body fat *vs.* intake is about 1 to 2, and seems to be constant at all levels tested.

In Table VI, the residues obtained with some insecticides at rates giving economic pest control and the corresponding residues in milk are compared with their toxicity. Endrin gives the largest safety margin.

Table II. Bioassay Determination of Endrin Content of Milk from Treated Cows

Endrin in Diet, P.P.M.	Endrin Content, P.P.M.[a]			
	During Endrin Feeding			After feeding ceased, 6 wk.
	1 wk.	8 wk.	12 wk.	
0.00	<0.002 (1)	<0.002 (1)	<0.002 (1)	<0.002 (1)
0.10	0.002 (1)	0.003 (3)	0.003 (2)	<0.002 (1)
0.25	0.003 (1)	0.007 (1)	0.02 (4)	0.002 (1)
0.75	0.009 (1)	0.03 (1)	0.03 (2)	0.002 (1)
2.00	0.03 (1)	0.05 (1)	0.06 (1)	0.02 (1)

[a] Figures in parentheses refer to number of animals.

Table III. Endrin Content of Various Cow Tissues after 12 Weeks of Endrin Intake and after 6 Additional Weeks without Endrin

Endrin in Diet, P.P.M.	No. of Animals[a]	Endrin Content, P.P.M.[b]											
		Brain, 12 wk.	Heart, 12 wk.	Kidney, 12 wk.	Liver		Renal Fat		Body Fat		Steak, 12 wk.	Roast	
					12 wk.	18 wk.	12 wk.	18 wk.	12 wk.	18 wk.		12 wk.	18 wk.
0.00	2 (1)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
0.10	3 (1)	<0.1	<0.1	<0.1	<0.1	...	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	...
0.25	4 (2)	<0.1	<0.1	<0.1	<0.1	...	0.2	<0.1	0.1	<0.1	<0.1	<0.1	...
0.75	3 (1)	<0.1	<0.1	<0.1	0.1	<0.1	0.3	<0.1	0.4	<0.1	<0.1	0.1	<0.1
2.00	2 (1)	<0.1	<0.1	<0.1	0.2	<0.1	0.8	0.1	1.0	<0.1	<0.1	0.1	<0.1

[a] Numbers in parentheses refer to number of animals retained for feedoff studies.
[b] Data corrected for apparent endrin content of control samples and rounded to the nearest 0.1 p.p.m. Values below this level are indicated. Only those samples which contained significant amounts of endrin at 12 weeks were analyzed at the end of the feedoff period.

Table IV. Endrin Content of Cow Tissue, Bioassay and Spectrophotometric Methods

Sample	P.P.M.	Endrin Content, P.P.M.	
		Bioassay	Spectrophotometric
Steak	Control	<0.02	<0.1
	0.25	<0.02	<0.1
Roast	0.75	0.07	0.1
	2.00	0.06	0.1
Liver	0.25	<0.02	<0.1
	0.75	0.11	0.1
	2.00	0.15	0.2
Renal fat	0.10	0.08	<0.1
	0.25	0.09	<0.1
	2.00	0.68	1.0

Table V. A Comparison of the Tendency of Endrin, Dieldrin, and Heptachlor to be Secreted in Milk

Insecticide	Intake in Diet, P.P.M.	Output in Milk, P.P.M.	Ratio Intake/Output	Reference
Endrin	0.25	0.02	0.08	This paper
	0.75	0.04	0.05	This paper
Dieldrin	0.40	1.0	2.5	(*6*)
	1.64	0.5	0.3	(*8*)
Heptachlor	0.24	0.3[a]	1.2[a]	(*8*)
	0.20	0.4[a]	2.0[a]	(*8*)
	1.20	0.0	...	(*7*)

[a] Secreted as heptachlor epoxide.

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Table VI. A Comparison of Rates of Application to Alfalfa, Residues at Harvest, Residues in Milk, and Acute Oral Toxicity to Rats of Endrin, Toxaphene, and DDT

Insecticide	Rate of Appl., Lb./Acre	Residue at Harvest, P.P.M.	Residue Expected in Milk, P.P.M.	Acute Oral Toxicity to Rats, Mg./Kg. (9)	Relative Safety Margin[a]	Reference
Endrin	1/8–1/4	0.15	0.02	18	9	This paper
Toxaphene	2	120	2.5	90	0.36	(3)
DDT	2	12.1	3.3	225	0.7	(4)

[a] Relative safety margin = LD_{50} per p.p.m. in milk × 100.

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INSECTICIDE RESIDUES

Field Persistence Comparisons of Residues of the Insecticide, Diazinon, in Lemons and Valencia Oranges and Effects on Juice Flavor

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Residues of the insecticide, Diazinon, were determined by an ultraviolet spectrophotometric procedure on and in lemons and oranges. The persistence of these residues is illustrated by the half-life values of 12 to 13 days for lemons and 16 to 17 days for Valencia oranges; Diazinon residues are short-lived compared to most other insecticides and acaricides as residues on citrus fruits. Triangular-type tests of juice from Diazinon-treated fruits showed no detectable flavor changes; citrus peel appears to be an efficient barrier against penetration into citrus juices by odors or flavors from outside sources.

THE COMPOUND *O,O*-diethyl *O*-(2-isopropyl-4-methyl-6-pyrimidinyl) phosphorothioate, or Diazinon, is a useful insecticide against certain insects (*4, 12*) and shows promise in the control of some California citrus pests—notably soft scale, *Coccus hesperidum* L. (*3*). In the present paper, magnitudes and half lives of residues of Diazinon on and in lemons and Valencia oranges treated in the field with commercial formulations are compared with residues from a number of other insecticides and acaricides.

Residue Studies

An analytical method suitable for determining magnitudes of residues of Diazinon on and in citrus tissues was proposed by Harris (*13*) and modified by Blinn and Gunther (*1*) for use in this study. This procedure is based on the ultraviolet determination of 2-isopropyl-4-methyl-6-pyrimidinol, a hydrolytic product of Diazinon. The method is semispecific, as only compounds that hydrolyze to a pyrimidinol will respond.

Materials and Methods. Average-sized Valencia orange trees were sprayed on June 4, 1956, either with 2.0 pounds of a 25% wettable-powder formulation of Diazinon per 100 gallons of water or with 2.0 pounds of a 25% emulsifiable-concentrate formulation of Diazinon per 100 gallons of water. Identical sprays were applied to average-sized lemon trees on November 12, 1956. Applications were made as conventional sprays, using a high-pressure reciprocating pump and manually operated spray guns. Final sprays were applied at the rate of approximately 1700 gallons per acre for the oranges and 1125 gallons per acre for the lemons.

Mature orange fruit samples for assay of residues were collected immediately before treatment, within 4 hours after the spray deposit had dried, and then 1, 4, 7, 14, 21, and 28 days after treatment. Mature lemon fruit samples for assay of residues were collected before treatment and 1, 4, 14, 21, and 28 days after treatment. One fruit was picked from each quadrant of each of eight trees in each plot, and the resulting 32 fruits were processed as a unit. The three replicates for each treatment were processed separately.

The fruits were peeled, and 1-pound subsamples of the minced peel and of the minced pulp were processed separately with petroleum ether in a manner previously described (*5*), to afford final stripping solutions. Aliquots of stripping solutions were assayed by the ultraviolet spectrophotometric procedure (*1*).

Results. Field-replicated residue values for Diazinon and field-treated lemons and Valencia oranges are collated in Table I and presented graphically in Figures 1 and 2. No Diazonon was found in the pulp (edible portion) of