

Separation of Dicofol (Kelthane) and its Dichlorobenzophenene Degradation Product from a Standard Florisil Column

by NORMAN L. MORGAN
*California Department of Agriculture
State of California
Sacramento, California*

It is necessary that an analytical method be developed to distinguish between dicofol and its p,p'-dichlorobenzophenone degradation product because both may exist on agricultural products prior to GLC determinations. It has been proven that dicofol breaks down, 15 - 85%, (1, 2) to p,p'-dichlorobenzophenone upon injection into gas chromatographs. This may lead the analyst to an incorrect conclusion as to the amount of dicofol on the agricultural product if any of its p,p'-dichlorobenzophenone degradation product is present originally. Therefore it is necessary to separate dicofol and p,p'-dichlorobenzophenone prior to GLC analysis. An analytical method has now been developed that will separate dicofol and p,p'-dichlorobenzophenone using a Florisil column.

Method

Apparatus and Reagents

(a) Gas Chromatograph - A dual column Jarrell Ash gas chromatograph equipped with dual electron capture detectors. One column

was fitted with a 3/16" by 5' glass column packed with 10% DC-200 on 60/80 mesh Gas Chrom Q. A second 3/16" by 5' glass column was packed with equal portions 10% DC-200 and 10% QF-1 on 60/80 mesh Gas Chrom Q. The instrument was operated at 190°C. Nitrogen flow rate was 200 ml at 20 psi.

(b) Florisil-60/100 mesh, as received from the Floridin Company (preheated to 1200°F) was placed in an open bottle and heated 18 hours at 130°C and held at 130°C until ready to use.(3)

(c) Petroleum ether, AR grade - Purified by redistillation, retaining 30-60°C fraction.

(d) Ethyl ether (Mallinckrodt, 0848, Ether Anhydrous) - Redistilled before use.

(e) Eluting mixture No. 1 - 100 ml purified ether (d) diluted to 1 liter with redistilled petroleum ether (c).

(f) Eluting mixture No. 2 - 250 ml purified ether (d) diluted to 1 liter with redistilled petroleum ether (c).

Preparation of Florisil Column

A standard 19 mm x 24" chromatographic column, equipped with an Ultramax valve, was dried thoroughly. The column was filled with 4" of Florosil; tapping the column while pouring. A $\frac{1}{2}$ " of anhydrous Na_2SO_4 was added to the top of the Florisil. As soon as the Florisil was cooled, the column was prewet with petroleum ether, running about 20-25 ml through the column and discarding the eluate. The column was not permitted to drain dry, but kept covered with petroleum ether.

Experimental

Several extracts of head lettuce and alfalfa hay were separately fortified with 2.0 ppm of dicofol and 1.0 ppm of p,p'-dichlorobenzophenone. The fortified extracts were transferred to a prewet Florisil column with a small amount of petroleum ether. Just before the petroleum ether layer reached the top of the sodium sulfate, 150 ml of eluting mixture No. 1 (e) were added to remove dicofol. Similarly, just before mixture No. 1 was exhausted from column, 150 ml of eluting mixture No. 2 (f) were added to remove p,p'-dichlorobenzophenone. Flow was regulated to about 5 ml per minute.

The eluates were concentrated and injected into a Jarrell Ash gas chromatograph. The dicofol was calculated by taking the area under the curve of dicofol and its degradation products, and comparing the areas with known concentrations. The p,p'-dichlorobenzophenone was calculated by taking the peak heights and comparing the heights with known concentrations.

Results

Table 1 gives the percent recoveries of dicofol and of p,p'-dichlorobenzophenone from a standard Florisil column. Recovery of dicofol from fortified extracts of alfalfa and lettuce in eluting mixture No. 1 were 96, 97, 95, and 98% respectively, at the 2.0 ppm level. Recovery of p,p'-dichlorobenzophenone from fortified extracts of alfalfa and lettuce in eluting mixture No. 2 were 94, 97, 95, and 96% respectively, at the 1.0 ppm level.

TABLE 1

% Recovery of Dicofol and its Dichlorobenzophenone Degradation Product From a Standard Florisil Column

Crop	Insecticide	Eluting Mixture No. 1	Eluting Mixture No. 2
Alfalfa #1	dicofol	96	3
	p,p'-dichlorobenzophenone	7	94
Alfalfa #2	dicofol	97	2
	p,p'-dichlorobenzophenone	5	97
Lettuce #1	dicofol	95	5
	p,p'-dichlorobenzophenone	6	95
Lettuce #2	dicofol	98	2
	p,p'-dichlorobenzophenone	7	96

References

- 1- Morgan, N. L., This Bulletin, Vol. I, 306(1966)
- 2- Westlake, W. E., et al., This Bulletin, Vol. I, 29(1966)
- 3- Gunther, F. A. et al., Stanford Research Inst. 2; 3(1962)