Determination of O,O-Diethyl O-*p*-Nitrophenyl Thiophosphate (Parathion) in Pineapples

R. Santini, Jr., and Evangelina Sotelo[1]

INTRODUCTION

In experiments carried out by the Entomology Department of this Station, the new synthetic insecticide O,O-diethyl O-*p*-nitrophenyl thiophosphate, better known as "parathion"[2] has been found very satisfactory for the control of the larva which is the causative agent for the disease known as pineapple gomosis. Because this insecticide is highly toxic it was necessary to determine whether it left any residue on the fruit. After a thorough search of the literature it was decided to apply the analytical method developed by Averell and Norris (1)[3], a procedure that had already been applied to apple, pear, grape, tobacco, cabbage, brussels sprouts, corn, tomato, cherry, pea vine, wax bean, grapefruit, and soils.

EXPERIMENTAL PROCEDURE

There are three major steps in the determination of minute quantities of parathion by the Averell and Norris procedure. The first step is the reduction with zinc dust in acid solution to the amino compound. The second step is the diazotization of the amino compound with sodium nitrite, removal of the excess nitrite with ammonium sulfamate, and coupling with N (1-naphthyl) ethylenediamine dihydrochloride to produce an intense magenta color. The third step is the evaluation of the color developed with a Coleman spectrophotometer and a standard transmission-concentration curve prepared by using a stock solution of parathion in benzene. Aliquots of this solution containing from 20 to 200 μg. were taken to prepare the curve (fig. 1). All readings were taken at a wavelength of 555 mμ, which is the absorption maximum obtained in the transmittance-wavelength curve of the colored solution (fig. 2).

DETERMINATION OF PARATHION IN PINEAPPLES

PREPARATION OF SAMPLE

Two hundred and fifty grams of pineapple were triturated in a Waring blendor for 2 minutes with different aliquots of a solution containing parathion dissolved in ethyl alcohol (0.3575 gm./l.). One hundred and twenty-five grams of the mashed sample were then poured into a cylindrical separatory

[1] Associate Chemist and Research Assistant, respectively, of the Agricultural Experiment Station, University of Puerto Rico, Río Piedras, P. R.

[2] Also sometimes as "E 605" or "compound 3422."

[3] Numbers in parentheses refer to Literature Cited, p. 15.

funnel. Two hundred grams of benzene were added and the parathion extracted by placing the separatory funnel in a horizontal shaker for 1½ hours. The bottom layer was drawn off and the benzene stored in a well-stoppered flask. One hundred grams of the benzene extract were weighed

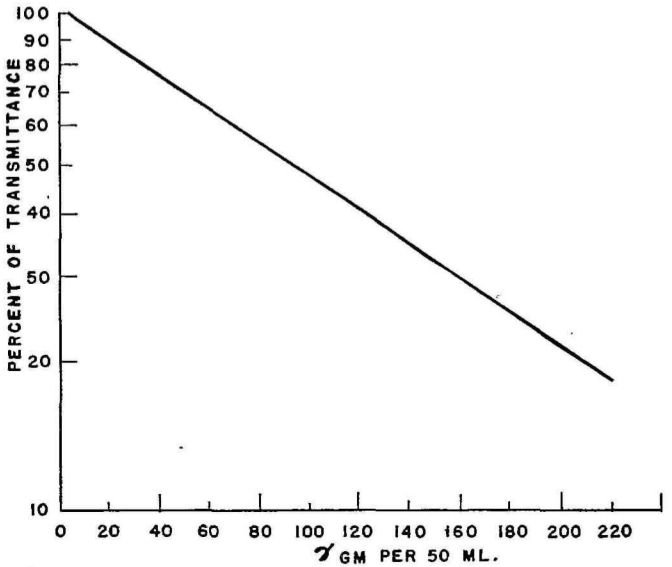

FIG. 1.—Standard transmission-concentration curve prepared using a stock solution of parathion in benzene.

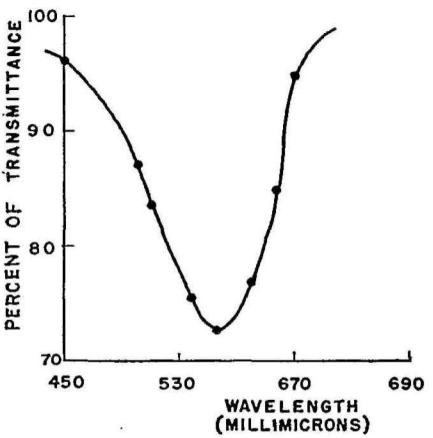

FIG. 2.—Readings obtained at the absorption maximum of the transmittance-wavelength curve of the colored solution used.

TABLE 1.—*Results of the determination of parathion in pineapple*

Sample No.	Parathion added	Parathion found on analysis
	P.p.m.	*P.p.m.*
1	0.1	0.2
2	.3	.3
3	.4	.5
4	.7	.8
5	.9	.9
6	1.2	1.2
7	1.5	1.2
8	1.7	1.4
9	2.3	2.1
10	2.4	2.0
11	3.0	2.9

into a 300-ml. tall-form beaker and decolorized with 11 gm. of clay-supercel mixture using the procedure of Averell and Norris.

DETERMINATION

The decolorized benzene solution was concentrated to 10 ml. in a 300-ml. tall-form beaker on a steam bath. The last 10 ml. of benzene were removed at room temperature by passing air over the surface, evaporation being discontinued as soon as the residue was dry. This residue was taken up in a mixture of 10 ml. of alcohol and 10 ml. of water; 2 ml. of 5N HCl and 0.2 gm. of zinc dust were added. Each beaker was covered with a watch glass, the contents boiled 5 minutes, allowed to cool, and then filtered through a No. 42 Whatman filter paper into a 50-ml. volumetric flask.

The beaker, residue, and paper were washed until the total volume of the filtrate was about 40 ml., when 1 ml. of 0.25-percent $NaNO_2$ was added, the flask was agitated, and then let stand for 10 minutes. Next 1 ml. of 2.5-percent ammonium sulfamate was added and the mixture was again agitated and let stand for 10 minutes. Finally, 2 ml. of 1-percent N (1-naphthyl) ethylenediamine dihydrochloride was added and the mixture diluted to 50 ml. with water. The blank solution, consisting of pineapple containing no parathion, was now used to set the spectrophotometer scale at 100-percent transmittance. The transmittance of each sample was then determined.

RESULTS

Table 1 shows the results obtained.

SUMMARY

Reliable results are obtained using the Averell and Norris procedure for the determination of very small qualities of parathion in pineapple.

RESUMEN

Se obtienen resultados satisfactorios cuando se usa el método Averell y Norris, para determinar cantidades pequeñas de *parathion* en la piña.

LITERATURE CITED

1. Averell, P. R. and Norris, M. V., Analy. Chem. **20** (8) 753–56, 1948.