200

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NICOTINE INSECTICIDES. PART II--SEARCH FOR ACTIVATORS

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This is the second of a series of investigations on nicotine insecticides conducted by this Bureau in cooperation with the Bureau of Agricultural and Industrial Chemistry. Part I of this series (E-646) reported a study of complex salts containing nicotine, usually combined with a metal. In the investigation described herein an effort was made to find activators for nicotine, partly to stretch the limited supply of this insecticide and partly to make its use more economical.

As before, the samples studied were prepared at the Eastern Regional Research Laboratory of the Bureau of Agricultural and Industrial Chemistry, for testing against plant-feeding insects by the Bureau of Entomology and Plant Quarantine at its Sanford, Fla., laboratory.

Previous attempts by other investigators to increase the toxicity of nicotine insecticides were concerned largely with the use of various alkaline materials to liberate the alkaloid from nicotine sulfate. For example, dolomite and lime have been called chemical accelerators by Headlee and Rudolfs (5), while Thatcher and Streeter (9) have classed them as active carriers. In a few cases, however, nonalkaline materials have been used, such as agar-agar (6), karaya gum (4), lecithin (1), and hydrocarbon oils (3).

In this investigation the entomological tests were conducted on several species of leaf-feeding larvae. In order to minimize losses of nicotine by volatilization from the dusts in shipment and on storage, the adjuncts to be evaluated as activators were not tested with nicotine alkaloid, but in combination with (1) nicotine bentonite in bentonite and (2) nicotine sulfate in pyrophyllite as the diluents.

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24 hours for 3 days. The deposit used depended upon the species being tested and also upon the susceptibility of that species at the time the tests were made, the desired mortalities being between 20 and 80 percent. Each sample was tested on from 2 to 6 species of insects. The melon worm and southern armyworm were used in all tests. Since these tests were only preliminary, no replications were made.

The second step was foliage-injury tests, made on 4 or 5 varieties of plants in an outdoor garden with the samples that showed some increase in toxicity as dusts over the standard materials against insects. The plants used were bean, collard, corn, cotton, okra, potato, pumpkin, sweetpotato, and tomato. Materials were sprayed on the plants twice, at an interval of 7 days and at a concentration of 0.1 percent of nicotine. Records of the amount of injury were taken on the third, seventh, and fourteenth days after the application of the first spray.

The third stage of testing was with sprays. All materials that showed some promise in dust tests, and which did not cause foliage injury, were used in spray tests on leaf sections which were fed to larvae in petri dishes and in cloth-covered vials.

Discussion of Results

The adjuncts which in the dust test, with either carrier, gave mortality of the melon worm or the southern armyworm, or both, 10 percent above that given by the standard were considered as activators of the nicotine. The following materials were found to be in this class:

2,2'-Allylidenebis(5,5-dimethyl-1,3-cyclohexanedione
2-Butoxyethanol
2-Chloroallyl lactate
p-Dichlorobenzene
Diethylene glycol monobutyl ether
Diethylene glycol monomethyl ether
5,5-Dimethyl-1,3-cyclohexanedione
Dodecanoic acid
2-Ethoxyethanol

Ethyl n-butylacetoacetate
o-Phenylphenol
12-Hydroxystearic acid
Kojic acid

Lauryl acetoacetate
Lecithin
Nitrobenzene
2-Nitrobiphenyl
Paraffin
Pentachloroethane
Pentachlorophenol
Polylactic acid (115% lactic acid)
Stearic acid
Tetrahydrofurfuryl lactate

The samples containing pentachlorophenol and nicotine were the only ones that gave higher mortalities than the standards in every test. In subsequent tests 97 percent of the melon worms and 100 percent of the armyworms were killed with rather light deposits of dust mixtures containing only pentachlorophenol as the toxicant. Swingle, Phillips, and Gahan (8) found this material to be toxic to 9 of 10 species against which it was tested, and it is also toxic to subterranean termites (10).

It appeared that only eight compounds in the list merited further attention. More complete data on these eight materials are given in table 1. With the exception of 2-nitrobiphenyl, these adjuncts were nontoxic when tested as 5 percent dusts without nicotine; 2-nitrobiphenyl was slightly toxic to the southern armyworm. Kojic acid and o-phenylphenol appeared to be the most effective adjuncts for nicotine sulfate in pyrophyllite.

Since none of these eight materials were injurious to plants in phytotoxicity tests, they were tested as sprays. The results are shown in table 2.

In table 3 a comparison of the two carriers as dusts and sprays indicates that the mixtures are slightly more effective as sprays. Furthermore, the indications are that dusts in which the materials are diluted in pyrophyllite, using nicotine sulfate, are more effective than the nicotine bentonite dusts. On the other hand, when the materials are applied as sprays the bentonite group is superior to the pyrophyllite.

Table 4 includes the adjuncts which gave insect mortalities above the standard when used in nicotine dusts or sprays. Since none of these compounds were highly effective, no attempt was made to show the degree of activity towards individual species. The table lists the test insects used.

Several materials appeared to be specific in their activation of nicotine toward different species of insects. The nicotine dusts containing the following materials in both bentonite and pyrophyllite were active agains the melon worm, but not against the southern armyworm: Ethyl acetoacetate, ethyl 2-(2-butoxyethoxyethyl)acetoacetate, ethyl n-lauryl-acetoacetate, furil, lauryl acetoacetate, octadecyl acetoacetate, octyl disulfide, and sulfur. Nicotine sprays containing lauryl acetoacetate were also effective against the southern armyworm in each series. Levulose was effective against the armyworm but was ineffective against the melon worm. Four other materials were activators of the nicotine against one of these two insects in one series

and against the other in the second series. Cyclohexanol, allyl maleate, and p-tolyl p-toluenesulfonate in bentonite were effective against the melon worm, and in pyrophyllite they were effective against the armyworm. Another material, 9,10-epoxystearic acid, in bentonite produced higher mortalities than the standard against the southern armyworm, but in pyrophyllite the melon worm was the susceptible insect.

The adjuncts which were inactive in all tests are listed in table 5.

In this search for an effective activator for nicotine none of the adjuncts sufficiently increased the insecticidal action of the nicotine compounds in these preliminary tests to merit further study on the test insects used.

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Table 1.--Effectiveness of various materials as activators in nicotine dusts when tested against various species of insects

Adjunct and insect 1/	Nicotine bentonite in bentonite				Nicotine sulfate in pyrophyllite			
	Deposit of dust per square centimeter	Extent of feeding	Mortality: Total	Mortality: Above or below standard	Deposit of dust per square centimeter	Extent of feeding	Mortality: Total	Mortality: Above or below standard
	Micrograms		Percent	Percent	Micrograms		Percent	Percent
2,2'-Allylidenebis(5,5-dimethyl-1,3-cyclohexanedione:								
Melon worm	125	Moderate	50	-30	140	Moderate	58	+10
Southern armyworm	180	-- do --	9	- 8	140	Trace	90	+38
Southern beet webworm	70	-- do --	43	+14	---	---	--	---
			Av.	- 8			Av.	+24
Diethylene glycol monomethyl ether:								
Bean leaf roller 2/	97	-- do --	65	+ 9	---	---	--	---
Cabbage looper	200	Normal	(17)3/	+ 0	---	---	--	---
Hawaiian beet webworm	200	Moderate	(67)	+42	---	- ---	--	---
Melon worm	145	-- do --	79	+14	140	Moderate	87	+22
Southern armyworm	180	-- do --	29	+13	160	-- do --	82	+25
Southern beet webworm	200	Trace	(71)	-29	---	---	--	---
			Av.	+ 8			Av.	+24
o-Phenylphenol:								
Bean leaf roller	128	Moderate	58	- 3	---	---	--	---
Melon worm	140	-- do --	61	-30	110	-- do --	83	+43
Southern armyworm	180	-- do --	88	+32	155	-- do --	83	+29
			Av.	- 1			Av.	+36
12-Hydroxystearic acid:								
Bean leaf roller	107	-- do --	55	- 3	---	---	--	---
Melon worm	110	-- do --	83	+14	125	-- do --	43	- 1
Southern armyworm	180	-- do --	72	+16	155	-- do --	75	+21
			Av.	+ 9			Av.	+10

Table 1.--Continued

Adjunct and insect 1/	Nicotine bentonite in bentonite				Nicotine sulfate in pyrophyllite			
	Deposit of dust per square centimeter	Extent of feeding	Mortality: Total	Mortality: Above or below standard	Deposit of dust per square centimeter	Extent of feeding	Mortality: Total	Mortality: Above or below standard
	Micrograms		Percent	Percent	Micrograms		Percent	Percent
Kojic acid:								
Bean leaf roller	93	Moderate	64	+ 8	---	---	--	---
Melon worm	135	-- do --	40	-22	125	Moderate	67	+26
Southern armyworm	180	-- do --	54	- 2	155	Trace	97	+43
				Av. - 5				Av. +35
2-Nitrobiphenyl:								
Bean leaf roller	117	Trace	53	- 6	---	---	--	---
Melon worm	145	Moderate	50	-41	115	Normal	35	- 6
Southern armyworm	180	Trace	(100)	+53	170	Trace	100	+46
				Av. + 2				Av. +20
Polylactic acid (115% lactic acid):								
Bean leaf roller	98	Moderate	68	+12	---	---	--	---
Melon worm	130	-- do --	61	+46	125	Moderate	46	+14
Southern armyworm	180	-- do --	20	- 9	155	-- do --	76	+11
				Av. +16				Av. +13
Stearic acid:								
Melon worm	125	-- do --	61	-19	170	-- do --	42	- 6
Southern armyworm	190	-- do --	27	+10	150	-- do --	97	+45
Southern beet webworm	70	Trace	75	+46	70	-- do --	48	+ 0
				Av. +12				Av. +13

1/ First-instar larvae of southern armyworm, but fourth instars of other species.

2/ All bean leaf roller data are the averages of three tests.

3/ Figures given in parentheses represent mortalities after 2 days' exposure. All other figures represent kill in 3 days.

Table 2.--Effectiveness of various materials as activators in sprays containing nicotine when tested against the melon worm and the southern armyworm.

Adjunct and insect 1/	Nicotine bentonite in bentonite				Nicotine sulfate in pyrophyllite			
	Nicotine and adjunct concentration	Extent of feeding	Mortality: Total in days 2/	Mortality: Above or below standard	Nicotine and adjunct concentration	Extent of feeding	Mortality: Total in days	Mortality: Above or below standard
	Percent		Percent	Percent	Percent		Percent	Percent
2,2'-Allylidenebis(5,5-dimethyl-1,3-cyclohexanedione):								
Melon worm	0.15	Moderate	79 (4)	+49	0.15	Moderate	75 (4)	+45
	.075	-- do --	63 (4)	+45	.075	Normal	46 (4)	+28
Southern armyworm	.3	-- do --	97 (4)	+27	.3	Trace	100 (2)	+19
	.15	-- do --	67 (4)	+36	.15	Moderate	60 (4)	+ 3
			Av.	+39			Av.	+24
Diethylene glycol monomethyl ether:								
Melon worm	.1	-- do --	87 (6)	- 3	.1	Normal	27 (4)	-25
Southern armyworm	.1	-- do --	38 (4)	+18	.1	--do--	24 (4)	+18
			Av.	+ 8			Av.	- 4
o-Phenylphenol:								
Melon worm	.1	-- do --	83 (6)	- 7	.1	--do--	43 (6)	-16
Southern armyworm	.1	Normal	23 (4)	+ 3	.1	--do--	19 (4)	+13
			Av.	- 2			Av.	- 2
12-Hydroxystearic acid:								
Melon worm	.1	Moderate	96 (6)	+ 6	.1	--do--	70 (6)	+11
Southern armyworm	.1	Normal	31 (4)	+13	.1	--do--	47 (4)	+41
			Av.	+10			Av.	+26
Kojic acid:								
Melon worm	.15	Trace	100 (3)	+61	.15	Moderate	95 (4)	+16
	.075	Moderate	65 (3)	+38	.075	--do--	42 (3)	- 6
Southern armyworm	.3	-- do --	75 (4)	+58	.3	Trace	83 (4)	+ 4
	.15	-- do --	58 (4)	+44	.15	Moderate	23 (4)	-15
			Av.	+50			Av.	- 1

Table 2.--Continued

Adjunct and insect 1/	Nicotine bentonite in bentonite				Nicotine sulfate in pyrophyllite			
	Nicotine and adjunct concentration	Extent of feeding	Mortality: Total in days 2/	Mortality: Above or below standard	Nicotine and adjunct concentration	Extent of feeding	Mortality: Total in days 2/	Mortality: Above or below standard
	Percent		Percent	Percent	Percent		Percent	Percent
2-Nitrobiphenyl:								
Melon worm	0.1	Trace	93 (4)	+20	0.1	Moderate	75 (6)	+16
Southern armyworm	.1	Moderate	71 (4)	+51	.1	Normal	20 (4)	+14
				Av. +36				Av. +15
Polylactic acid (115 percent lactic acid):								
Melon worm	.1	-- do --	93 (6)	+20	.1	Normal	63 (6)	+ 4
Southern armyworm	.1	Normal	27 (4)	+ 7	.1	Moderate	20 (4)	+14
				Av. +14				Av. + 9
Stearic acid:								
Melon worm	.15	Trace	100 (3)	+61	.15	-- do --	86 (4)	+ 7
	.075	Moderate	81 (3)	+64	.075	Normal	42 (3)	- 6
Southern armyworm	.3	Trace	95 (3)	+78	.3	Trace	70 (4)	- 9
	.15	Moderate	48 (4)	+34	.15	Moderate	22 (4)	-16
				Av. +59				Av. - 6

1/ First-instar larvae of southern armyworm, but fourth instar of other species.

2/ Figures given in parentheses represent number of days exposure.

Table 3.--Comparison of the adjuncts as a group when used as dusts or sprays in bentonite and pyrophyllite against insects listed in tables 1 and 2

Carrier	Dust tests		Spray tests	
	Average mortality above or below standard	Tests	Average mortality above or below standard	Tests
	Percent	Number	Percent	Number
Bentonite	+22	15	+36	20
	-17	12	- 5	2
Pyrophyllite	+26	14	+17	15
	- 4	3	-13	7

Table 4.--Adjuncts showing activating action against some species of insects when included in nicotine mixtures

Adjunct	Hawaiian beet web-worm	Melon worm	Bean leaf roller	Cabbage looper	Southern armyworm First instar	Southern armyworm Fourth instar	Southern beet web-worm	Fall web-worm
2,2'-Allylidenebis(5,5-dimethyl-1,3-cyclohexanedione)	--	xx	--	--	xx	--	xx	--
Allyl maleate	--	xx	xx	--	xx	xx	--	--
2-Butoxyethanol	x	xx	xx	x	xx	xx	x	--
2-Chloroallyl lactate	--	xx	xx	--	xx	--	--	--
p-Tolyl p-toluenesulfonate	--	xx	x	--	xx	--	--	--
Cyclohexanol	--	xx	xx	x	xx	x	x	--
p-Dichlorobenzene	--	xx	xx	--	xx	--	--	--
Diethylene glycol monobutyl ether	x	xx	x	x	xx	xx	--	--
Diethylene glycol monomethyl ether	xx	xx	xx	x	xx	x	x	--
5,5-Dimethyl-1,3-cyclohexanedione	--	xx	--	--	xx	--	x	--
Dodecanoic acid	--	x	--	--	xx	--	xx	--
2-Ethoxyethanol	x	xx	x	x	xx	x	x	--
Ethyl acetoacetate	--	xx	xx	x	x	x	x	x
Ethyl 2-(2-butoxyethoxy ethyl)-acetoacetate	x	xx	x	xx	x	x	xx	x
Ethyl n-butyl-acetoacetate	--	xx	x	x	xx	xx	xx	x
Ethyl n-lauryl-acetoacetate	--	xx	xx	x	x	x	xx	--
Furil	--	xx	xx	xx	x	xx	xx	x
o-Phenylphenol	--	xx	x	--	xx	--	--	--
12-Hydroxystearic acid	--	xx	x	--	xx	--	--	--
Kojic acid	--	xx	xx	--	xx	--	--	--
Lauryl acetoacetate	--	xx	x	x	x	x	xx	x
Lecithin	x	x	x	x	xx	xx	x	--
Levulose	--	x	x	--	xx	--	--	--
Nitrobenzene	--	xx	x	x	xx	x	x	--
2-Nitrobiphenyl	--	xx	x	--	xx	--	--	--
Octadecyl acetoacetate	--	xx	x	x	x	x	xx	x
Octyl disulfide	xx	xx	x	x	x	xx	x	--
9,10-Epoxystearic acid	--	xx	x	--	xx	--	--	--

Table 4.--Continued

Adjunct	Hawwaiian beet web-worm	Melon worm	Bean leaf roller	Cabbage looper	Southern armyworm		Southern beet web-worm	Fall web-worm
					First instar	Fourth instar		
Paraffin	--	xx	xx	--	xx	--	--	--
Pentachloroethane	--	xx	x	--	xx	--	--	--
Pentachlorophenol	--	xx	xx	--	xx	--	--	--
Polylactic acid (115% lactic acid)	xx	xx	xx	--	xx	xx	--	--
Stearic acid	--	xx	--	--	xx	--	xx	--
Sulfur	--	xx	xx	x	x	x	xx	x
Tetrahydrofurfuryl lactate	--	xx	xx	--	xx	--	--	--

1/ x denotes material was tested but no activation occurred; xx denotes that activation occurred on this species.

Table 5.--Adjuncts showing no activating action in nicotine mixtures and insects against which they were tested

Adjuncts	Hawaiian beet webworm	Melon worm	Bean leaf roller	Cabbage looper	Southern armyworm		Southern beet webworm	Fall web-worm
					First instar	Fourth instar		
Acme white oil (80-85 sec. Saybolt)	- 1/	x 1/	x	x	x	x	-	-
Agar-agar	-	x	x	-	x	-	-	-
Beeswax	-	x	x	-	x	-	-	-
Benzyl disulfide	-	x	x	x	x	x	x	-
2,2'-Benzylidenebis(5,5-dimethyl-1,3-cyclohexanedione)	-	x	-	-	x	-	x	-
Bis(1-carballyloxyethyl)chloromaleate	-	x	x	-	x	x	-	-
Bis(1-carballyloxyethyl)maleate	-	x	x	-	x	x	-	-
Bis(2-chloroethyl)ether	x	x	x	x	x	x	-	-
2-(2-Butoxyethoxy)ethyl acetoacetate	-	x	x	x	x	x	x	x
2-Butoxyethyl phthalate	-	x	x	-	x	-	-	-
Butyl disulfide	-	x	x	x	x	-	-	-
Camphor	-	x	-	-	x	-	x	-
Caprylic acid	-	x	-	-	x	-	x	-
2-Carbododecoxy-5-hydroxy-1,4-pyrone	-	x	x	x	x	x	x	-
Castor oil	-	x	x	-	x	-	-	-
Chloramine T	-	x	x	-	x	-	-	-
2-Chloroallyl-alpha-acetoxypropionate	-	x	x	-	x	-	-	-
Chlorobenzene	-	x	x	x	x	x	x	-
o-Chlorobenzyl disulfide	-	x	x	x	x	x	x	-
2-Chloroethanol	-	x	x	-	x	-	-	-
2-Chloroethyl lactate	-	x	-	-	x	-	x	-
2-Chloromethyl-5-hydroxy-1,4-pyrone	-	x	-	-	x	-	x	-
2-Chloromethyl-5-methoxy-1,4-pyrone	-	x	x	x	x	x	-	-
Cottonseed oil	-	x	x	x	x	x	x	-
p-Cymene								

Table 5.- Continued

Adjuncts	Hawaiian beet webworm	Melon worm	Bean leaf roller	Cabbage looper	Southern armyworm First instar	Southern armyworm Fourth instar	Southern beet webworm	Fall webworm
Dibutyl phthalate	-	x	x	-	x	-	-	-
Dibutyl sebacate	-	x	x	-	x	-	-	-
Diethyl maleate	-	x	x	-	x	-	-	-
9,10-Dihydroxystearic acid	-	x	x	-	x	-	-	-
Dodecyl amine	-	x	-	-	x	-	x	-
Furoin	-	x	x	x	x	x	x	x
Gelatin	-	x	-	x	x	-	-	-
Guayule gum	-	x	-	x	x	x	-	-
Gum arabic	-	x	x	-	x	-	-	-
10-Hendecenoic acid	x	x	x	x	x	x	x	-
2(10-Hendecenoxy)-5-hydroxy-1,4-pyrone	-	x	-	-	x	-	x	-
5-Hydroxy-2-thiocyanomethyl-1,4-pyrone	-	x	-	-	x	-	x	-
Linseed oil	-	x	x	-	x	-	-	-
1-Methylallyl alpha-acetoxypropionate	-	x	x	-	x	-	-	-
2-Methylallyl alpha-acetoxypropionate	-	x	x	-	x	-	-	-
2-Methylallyl lactate	-	x	x	-	x	-	-	-
2,2'-Methylenebis(5,5-dimethyl-1,3-cyclohexanedione)	-	x	x	-	x	-	-	-
Monohydroxy stearic acid polymer	-	x	x	-	x	-	-	-
p-Nitrobenzyl disulfide	x	x	x	x	x	x	x	-
Octadecyl disulfide	-	x	x	x	x	x	x	-
2-Octanol	-	x	x	-	x	-	-	-
Pentachlorophenyl p-toluenesulfonate	-	x	x	x	x	-	-	-
Petrolatum	-	x	x	-	x	-	-	-
Phenyl alpha-acetoxypropionate	-	x	x	-	x	-	-	-

Table 5.--Continued

Adjuncts	Hawaiian beet webworm	Melon worm	Bean leaf roller	Cabbage looper	Southern armyworm		Southern beet webworm	Fall web-worm
					First instar	Fourth instar		
Polyvinyl alcohol	-	x	-	-	x	-	x	-
Pyrogallol	-	x	x	-	x	-	-	-
Resorcinol	-	x	x	-	x	-	-	-
2,2'-Salicylidenebis(5,5-dimethyl-1,3-cyclohexanedione)	-	x	-	-	x	-	x	-
Soluble starch	-	x	x	-	x	-	-	-
Sulfoxystearic acid	-	x	x	-	x	-	-	-
Tetrahydrofurfuryl alpha-acetoxypropionate	-	x	x	-	x	-	-	-
Tetralin	-	x	x	-	x	-	-	-
Triethylene glycol acetoacetate	-	x	x	x	x	x	x	x
Triethylene glycol bis(2-ethyl butyrate)	-	x	x	-	x	-	-	-
Turpentine	-	x	x	-	x	-	-	-
Xylylstearic acid	-	x	x	-	x	-	-	-

1/ x denotes test was made; - denotes that no test was made.