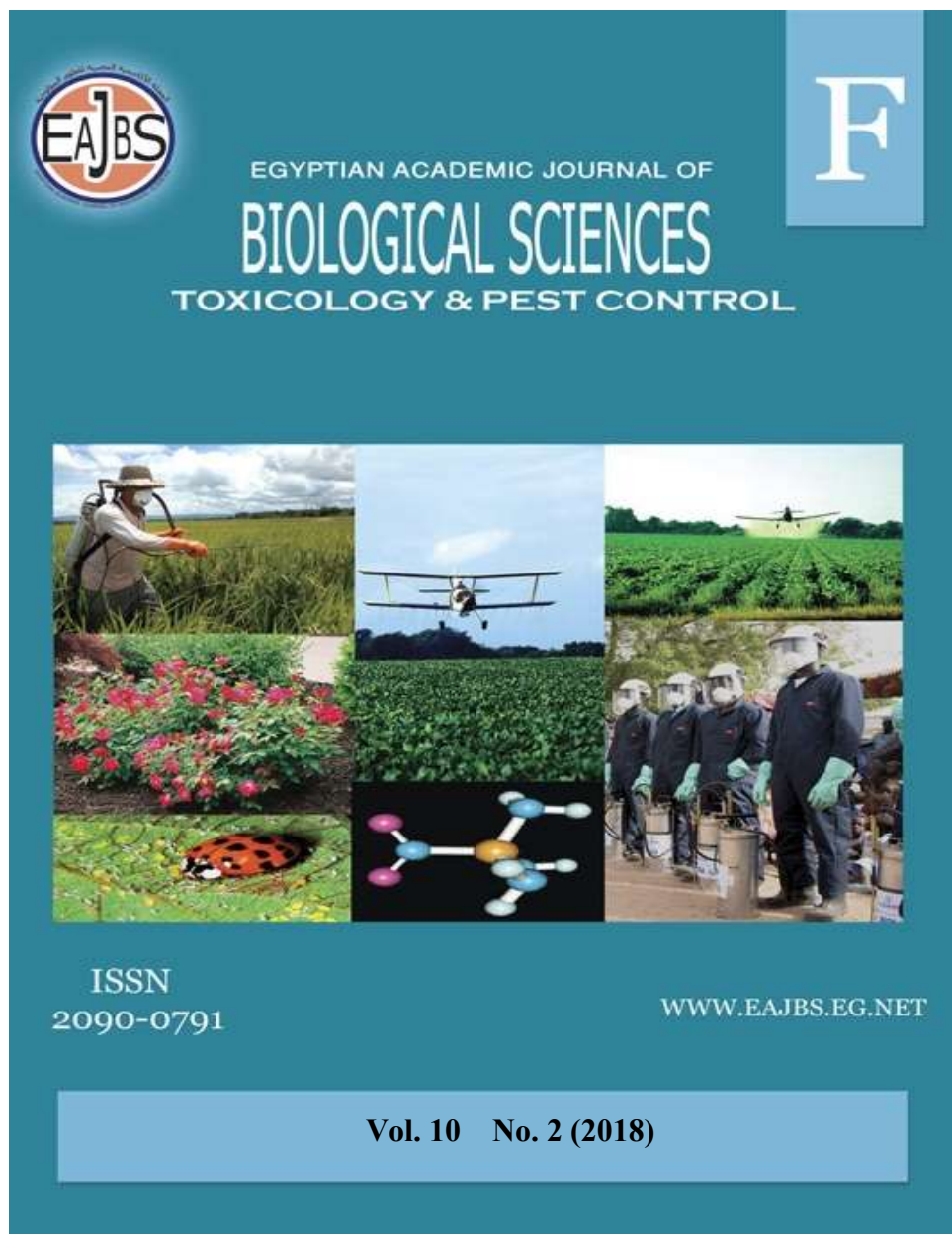


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**Toxicological and Biological Effects of some Synthetic Organic Compounds on Pink Bollworm, *Pectinophora gossypiella* (Sound.) and Cotton Leafworm, *Spodoptera littoralis* (Boisd.) Laboratory Strains**

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**ABSTRACT**

Laboratory experiments have been conducted in the Plant Protection Research Institute (Sharkia Branch) Bollworms Research Department to evaluate the effect of to study the toxic and biological effects of three synthetic organic compounds, pyrazole: (A= 3-amino-5-hydrazino-4-phenyl azo -1*H*-pyrazole, B= 3-hydrazino-4- phenyl azo -5-hydroxy-1*H*-pyrazole and C= 1-(5-hydroxy-4- phenyl azo -1*H*-pyrazole-3-yl ) 3, 5-dimethylpyrazole) against the 1<sup>st</sup> instar larvae of pink bollworm, *Pectinophora gossypiella* (Sound.) and 2<sup>nd</sup>, 4<sup>th</sup> instar larvae of cotton leafworm, *Spodoptera littoralis* (Boisd.) under constant conditions of (26± 1°C. and 70 ± 5% R.H.). Data revealed that synthetic pyrazole compounds had highly toxic effect against newly hatched larvae of pink bollworm followed by 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis*. On the other hand, the latent effects of the pyrazoles compounds on the two insects species were presented in increasing the duration of larval and pupal stages, Significantly decreased on larval and pupal weight of the two insects, male and female longevities and also reduced the fecundity, While the highest reduction in laid eggs recorded 103.83 eggs/female in pink bollworm followed by 123.33 eggs/female for 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis* as compared with 287.25 egg/female for pink bollworm and 503.33 & 663.33 egg/female with untreated control, Also, results showed the tested chemical compounds on the hatchability rate caused decreased significantly with *P. gossypiella* recorded 61.48 % with compound (A) & 55.57 ,68.21 % reduction in 2<sup>nd</sup>, 4<sup>th</sup> instar larvae of *S. littoralis* compared 88.00% for untreated moths of *P. gossypiella* and 63.00 , 97.47 % in 2<sup>nd</sup>, and 4<sup>th</sup> instar larvae for untreated moths of *S. littoralis*, respectively

**INTRODUCTION**

Cotton, *Gossypium hirsutum* (L.) is considering an important crop all over the world which it is strategic crop by sharing in Egyptian national income. Cotton plants like other field crops are liable to attack by several species of insect pests during its growing season. In Egypt during the late cotton season, cotton plants suffer from the infestation with pink bollworm, *P. gossypiella*. Also, caused the most damage for major parts of the pest complex on cotton in Egypt. The loss caused by *P. gossypiella* to cotton arises to one million kantar annually Metwally *et al.*, (1980). The level of resistance to some insecticides has increased and the new group of chemicals was needed to manage resistant populations in cotton. Insecticidal

pyrazoles was a new family of insecticides that act on the gamma – amino butyric acid (GABA) receptors of insects by blocking the passage of chloride ions, inducing disruption of the central nervous system Cole *et al.*, (1993). The fipronil is the first compound of phenyl pyrazole insecticide class to be registered for commercial use which is toxic to both piercing – sucking and chewing insects Eric *et al.*, (2001) and also Scott and Sondgrass (2000) also found pyrazoles has shown excellent activity against *Spodoptera litura*, tarnished plant bug and European corn borer larvae.

Therefore, the present study aims to throw some light on the toxic and latent effect of some synthetic organic compounds (pyrazole) against the newly hatched larvae of the *P. gossypiella* and 2<sup>nd</sup>, 4<sup>th</sup> instar larvae of cotton *S. littoralis* under laboratory conditions.

## MATERIALS AND METHODS

### Tested Chemical Compounds:

The tested chemical compounds synthesized at the laboratory of the faculty of science, Zagazig University. The tested chemical compounds were as follows:

**A=** 3-amino-5-hydrazino-4- phenyl azo -1*H*-pyrazole

**B=** 3-hydrazino-4- phenyl azo -5-hydroxy-1*H*-pyrazole

**C=** 1-(5-hydroxy-4- phenyl azo -1*H*-pyrazole-3-yl ) 3, 5-dimethylpyrazole

3-Amino-4-arylazo-2-pyrazolin-5-one and 4-arylazo-3,5-diamino-pyrazoles have been synthesized (Elnagdi and Abdalla 1973).

### Experimental Insect:

**a- *P. gossypiella*:** A susceptible strain of the pink bollworm was supplied by the Bollworm Research Department, Plant Protection Research Institute, Agric. Res. Center (Sharkia Branch), reared for many generations away from any insecticides treatments and kept in the incubator at 26± 1°C. and 70 ± 5% R.H, on the artificial diet under laboratory conditions have been described by (Abd El-Hafez, *et al.*, 1982). The larvae for both tested insects were used for carried all experiments.

**b- *S. littoralis*:** A laboratory strain of *S. littoralis* larvae was reared in the laboratory away from any insecticidal contamination at the department of cotton leafworm, Branch of Plant Protection Research Institute at Zagazig, Sharqia Governorate under constant conditions 26± 1°C. and 70 ± 5% R.H. to provide insects used in the present investigation. The neonate hatched larvae resulted from laid egg-masses for each adult female were placed on leaves of castor bean oil, *Ricinus communis* in cylindrical glass jars El-Defrawi *et al.*, (1964).

### Acute Toxicity of Some Tested Chemical Compounds Against *P. Gossypiella* and *S. Littoralis*:

***P. gossypiella*:** To evaluate the acute toxicity of the three tested chemical compounds; 3-amino-5-hydrazinopyrazole(A),3-hydrazino-5-hydroxypyrazole (B) and 1-(5-hydroxy -4- phenyl azo -1 *H*-pyrazole-3-yl )3, 5-dimethylpyrazole (C) against newly hatched larvae of *P. gossypiella* and the 2<sup>nd</sup>, 4<sup>th</sup> instar larvae of *S. littoralis*. Serial aqueous dilutions of the three tested compounds in distilled water were prepared from the stock solution as four dilutions. The concentrations of each compound gave about 20 – 80 % larval mortality of *P. gossypiella* and the 2<sup>nd</sup>, 4<sup>th</sup> instar larvae of *S. littoralis*. The stock solution of each tested chemicals prepared were achieved by adding one gram from each compound to 25ml acetone (solvent) as first concentration then the forward concentrations were 50.00, 25.00, 12.50 and 6.25%, respectively. All the concentrations prepared for each compound were used against *P. gossypiella* and the 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis*.

***P. gossypiella*:** Ten grams of semi-artificial diet was poured into conventional Petri-dishes (9cm diameter) were treated with different concentrations of the three tested chemicals by adding 3ml of each concentration of each compound. All the Petri dishes were treated with all concentrations of each compound and it was distributed on the upper surface of the poured diet using volume syringe by moving the dish gently in circles as well as untreated check and the Petri-dish treated was left to dryness under room conditions. Each concentration was replicated three times. Twenty newly hatched larvae (0-6 hr old) were transferred over treated artificial diet in the Petri-dish using a camel hair brush and it was incubated under mentioned above conditions.

***S. littoralis*:** Disks (9 cm. diameter) of castor bean leaves were dipped in the tested concentrations of each compound for about 10 seconds then left to dry under room condition and offered to feed larvae, which starved for 4-6 hours before treatment Merdan (1968). Larvae were placed into glass jars (5 pounds), each treatment was replicated 5 times (10 larvae per each) as well as untreated disks were dipped in distilled water only. The dead larvae were counted after 48 hours for all the tested compounds and the untreated check. The percent larval mortality was estimated for both insects tested. The LC<sub>50</sub>, LC<sub>90</sub> and slope values of each tested compound against *P. gossypiella* and the 2<sup>nd</sup>, 4<sup>th</sup> instar larvae of *S. littoralis* were calculated according to the method described by Finney (1971).

Toxicity index (T.I.) was determined by using the sun's equation (1950)

Relative potency (R.P.) values were measured according to the method described by Zidan and Abdel-Megeed (1988).

#### **Latent Effect of The Tested Chemical Compounds Against 1<sup>st</sup> Instar Larvae of *P. gossypiella* and 2<sup>nd</sup> and 4<sup>th</sup> Instar Larvae of *S. littoralis*:**

**1. *P. gossypiella*:** To study the latent effects of the tested chemicals compounds on certain biological aspects of *P. gossypiella* 1<sup>st</sup> instar larvae. The concentrations used were as follows: (175.07 ppm); (271.27 ppm) and (229.08 ppm) for (A), (B) and (C) compounds, respectively. The alive larvae after 48 hours from treatment with LC<sub>25</sub> for each tested chemical compounds and the control was transferred individually into the glass tube (2×7.5 cm), containing about five grams of untreated semi-artificial diet. The tubes were covered with a piece of absorbent cotton and held under the same conditions as mentioned before, the weight of the 4<sup>th</sup> instar larvae from these records, some biological aspects such as larval and pupal duration as well as the weight of 4<sup>th</sup> instar larvae and pupae, and pupation percentages. The *P. gossypiella* was sexed in the fourth larval stage according to (Raslan 1994). After the insect pupation, pupae were transferred individually to clean vials covered with cotton stopper and incubated at mentioned above till moth emergence. The adult emergence percentage and sex ratio of adult moths were calculated. The emerged moths from each treatment and control were caged in one pair (male and female) in the glass jar (7.00 cm in diameter) under the previously mentioned rearing conditions and covered with muslin cloth serving as oviposition site. Moths fed on 10% sucrose solution, used soaked cotton wool changed daily by new once. Each replicate has 5 pairs of newly emerged moths and it was inspected daily to record the number of the deposited eggs/female, longevity of male and female adults. The hatchability percentages of all deposited eggs per female were also determined as well as untreated moths.

**2. *S. littoralis*:** To study the latent effects of the tested chemical compounds on certain biological aspects of *S. littoralis*, The concentrations used were as follows: (373.89 ppm); (493.01 ppm) and (568.54 ppm) for (A), (B) and (C) compounds,

respectively for 2<sup>nd</sup> instar larvae .While in 4<sup>th</sup> instar larvae the concentrations of tested chemical compounds used were as follows: (453.19 ppm); (550.93 ppm) and (650.91 ppm), respectively. The alive larvae after 48 hours from treatment with LC<sub>50</sub> value for each tested chemical compounds and the control was transferred individually into clean glass containers and fed on fresh castor leaves until the pupation and five replicates in each treatment were prepared. After pupation, pupae were collected and placed in wide clean jars until adult emergence. Then, the emerged adults were supplied with a piece of cotton wetted with 10% sugar solution and branches of tafla (*Nerium oleanderd*) as a suitable site for oviposition. Newly laid egg masses were collected daily and transferred into the rearing jars (Gaaboub *et al.*, 2012).The biological parameters of the foregoing three compounds were evaluated as larval and pupal period, the pupation and adult emergence percentages, larval and pupal weight, fecundity, the adult longevity of male and female, the sex ratio of adult stage and hatchability percentages.

#### Statistical Analysis:

The obtained results of larval mortality for each treatment and biological measurements were subjected to analysis of variance to clear the significance of the toxic and latent effects of the different tested compounds against *P. gossypiella* and *S. littoralis* larvae. The proper "F" and LSD value was calculated as described by Fisher (1950) and Snedecor (1970).

## RESULTS AND DISCUSSION

### I- Acute toxicity of the three tested chemical compounds against 1<sup>st</sup> instar larvae of *P. gossypiella* and 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis*:

#### 1. *P. gossypiella*:

**1.1. LC<sub>50</sub> and LC<sub>90</sub> Values:** Data presented in Table (1) showed that at LC<sub>50</sub> level the toxicity effect of the three tested chemical compounds against *P. gossypiella* can be arranged in descending order as follows: A, B and C compounds, where their values were, 330.08, 492.82 and 521.88 ppm., respectively. While at the LC<sub>90</sub> level, data in the same Table showed that chemical A was the most toxicant one, followed by B and C chemicals, which recorded 1101.38, 1532.37 and 2494.66 ppm., respectively.

Generally, from LC<sub>50</sub> and LC<sub>90</sub> values, it could be noticed that chemical A was the most toxicant one, and it was the most promising compound for controlling *P. gossypiella*. While, chemical C was the least toxic effect against 1<sup>st</sup> instar larvae of *P. gossypiella*.

**1.2. Toxicity Lines, Slope Values and LC<sub>90</sub>/LC<sub>50</sub> Ratio:** According to the results obtained in Table (1), the toxicity effect of the three tested chemical compound against 1<sup>st</sup> instar larvae of *P. gossypiella*, it could be proved that chemical B, has the steepest toxicity line, where it has the highest slope value 2.60 and lowest LC<sub>90</sub>/LC<sub>50</sub> ratio was 3.11, while chemical C, has the flattest one, where it has the lowest slope value was 1.89 and highest LC<sub>90</sub>/LC<sub>50</sub> ratio was 4.78 while the compound A, was in intermediate between B and C chemicals it recorded slope value 2.44. while LC<sub>90</sub>/LC<sub>50</sub> ratio was 3.34.

**1.3. Toxicity Index:** According to Sun's equation (1950) in Table (1) the relative toxicity of the three tested chemical compounds against the *P. gossypiella* 1<sup>st</sup> larvae. At the LC<sub>50</sub> and LC<sub>90</sub> levels, chemical compound A, taken as the standard compound (which resulted in the least LC<sub>50</sub> of all tested chemical compounds) and given the arbitrary value of 100 units, the toxicity index of the

other two chemicals at LC<sub>50</sub> were 66.98 and 63.25 at LC<sub>90</sub> level were 71.87 and 44.15 for B and C compounds, respectively.

**1.4. Relative Potency:** The potency levels of the tested chemical compounds are expressed as the number of folds at the required toxicity level, compared with the least effective chemical in the three tested chemical compounds against *P. gossypiella*. Hence the number of folds representing the relative potency level in Table (1) was obtained by dividing the LC<sub>50</sub> and LC<sub>90</sub> of chemical C (the standard chemical). At the relative potency levels were 1.58 and 1.06 times as toxic as C compound, respectively. At the LC<sub>90</sub> level, the for A and B compounds were 2.27 and 1.63 times as compared with 1.00 fold for C compound, respectively.

Table (1): Toxicity of three pyrazoles compounds against 1<sup>st</sup> instar larvae of *P. gossypiella* laboratory strain after 48 hrs from treatment .

Chemical compounds	LC <sub>50</sub> (ppm)	LC <sub>90</sub> (ppm)	Toxicity index at		Relative potency at		Slope values	Ratio LC <sub>90</sub> /LC <sub>50</sub>
			LC <sub>50</sub>	LC <sub>90</sub>	LC <sub>50</sub>	LC <sub>90</sub>		
A	330.08	1101.38	100.00	100.00	1.58	2.27	2.44	3.336
B	492.82	1532.37	66.98	71.87	1.06	1.63	2.60	3.109
C	521.88	2494.66	63.25	44.15	1.00	1.00	1.89	4.780

**Where:** A= 3-amino-5-hydrazino-4- phenyl azo -1*H*-pyrazole B= 3-hydrazino-4- phenyl azo -5-hydroxy 1*H*-pyrazole C= 1-(5-hydroxy-4- phenyl azo -1*H*-pyrazole-3-yl ) 3, 5-dimethylpyrazole

## 2. *S. littoralis*:

**2.1. LC<sub>50</sub> and LC<sub>90</sub> Values:** Data in Table (2) indicated that at LC<sub>50</sub> level the toxicity effect of the three tested chemicals against 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S.littoralis* can be arranged in descending order as follows: A, B and C, where as follows: 373.89, 493.01 and 568.54 ppm., respectively. But, at the data of LC<sub>90</sub> values, showed that chemical A was the most toxicant one, followed by B and C chemicals, which recorded 1472.99, 1842.49 and 2484.91 ppm., respectively. While against 4<sup>th</sup> instar larvae at LC<sub>50</sub> level the toxicity effect of the three tested chemical compounds can be arranged in descending order as follows: A, B and C, where their values were, 453.19 550.93 and 650.91 ppm., respectively. Meanwhile the at the LC<sub>90</sub> values data showed that chemical A was the most toxicant one, followed by B and C chemicals, which recorded 1722.28, 2516.11 and 3412.61 ppm., respectively.

**2.2. Toxicity Lines, Slope and LC<sub>90</sub>/LC<sub>50</sub> Ratio:** According to the results obtained in Table (2) the toxicity effect of the three tested chemicals against 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S.littoralis*, it could be proved that chemical B, has the steepest toxicity line, where it has the highest slope value was 2.24 and 1.94. The lowest LC<sub>90</sub>/LC<sub>50</sub> ratio was 3.74 and 4.57, while chemical C, has the flattest one, where it has the lowest slope value and highest LC<sub>90</sub>/LC<sub>50</sub> ratio were 4.37 and 5.24 compound A, was in intermediate between B and C chemicals which recorded 3.93 and 3.80.

**2.3. Toxicity Index:** According to Sun's equation (1950) in Table (2) the relative toxicity of the three tested chemical compounds against the 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis* were evaluated. At the LC<sub>50</sub> and LC<sub>90</sub> levels, chemical compound A, taken as the standard compound LC<sub>50</sub> given the arbitrary value of 100 units, the toxicity indices of the other two chemicals at LC<sub>50</sub> were 75.84 and 65.76 at LC<sub>90</sub>

level were 79.95 and 59.28 for B and C compounds, respectively against 2<sup>nd</sup> instar larvae, while in case of 4<sup>th</sup> instar larvae the toxicity index of the tested compounds at the LC<sub>50</sub> and LC<sub>90</sub> levels were 82.26 and 69.62 at LC<sub>90</sub> level were 68.45 and 50.47 for B and C compounds, respectively.

**2.4. Relative Potency:** The potency levels of the tested chemical compounds are expressed as the number of folds at the required toxicity level, compared with the least effective chemical in the three chemical compounds tested against the 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis*. The number of folds representing the relative potency level was obtained by dividing the LC<sub>50</sub> and LC<sub>90</sub> of chemical C (the standard chemical). The relative potency levels were 1.52 and 1.153 times as toxic recorded 1.00 fold as C compound, respectively. While, at the LC<sub>90</sub> level, the relative potency levels for A and B compounds were 1.69 and 1.35 times as compared with 1.00 fold for C compound, respectively. While in case of 4<sup>th</sup> instar larvae the LC<sub>50</sub> and LC<sub>90</sub> of A and B chemical compounds were 1.44 and 1.18 times as toxic as C, respectively. But, at LC<sub>90</sub> level for A and B compounds were 1.98 and 1.36 times as compared with 1.00 fold for C compound, respectively.

**II- Latent Effect:** The main objective of these experiments is to study the latent effect of phenyl pyrazoles on some biological aspects of the survived larvae and subsequent all development stages of *P. gossypiella* and 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis*. The results could be discussed as the follows:-

Table (2): Toxicity of three pyrazoles compounds against the 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis* laboratory strain.

Chemical compound s	Larval instars	LC <sub>50</sub> (ppm)	LC <sub>90</sub> (ppm)	Toxicity index at		Relative potency (folds) at		Slope values	Ratio LC <sub>90</sub> / LC <sub>50</sub>
				LC <sub>50</sub>	LC <sub>90</sub>	LC <sub>50</sub>	LC <sub>90</sub>		
A	2 <sup>th</sup>	373.89	1472.99	100.00	100.00	1.52	1.69	2.15	3.94
	4 <sup>th</sup>	453.19	1722.28	100.00	100.00	1.44	1.98	2.21	3.80
B	2 <sup>th</sup>	493.01	1842.49	75.84	79.946	1.153	1.348	2.24	3.74
	4 <sup>th</sup>	550.93	2516.11	82.26	68.45	1.18	1.36	1.94	4.57
C	2 <sup>th</sup>	568.54	2484.91	65.76	59.277	1.00	1.00	2.00	4.37
	4 <sup>th</sup>	650.91	3412.61	69.63	50.47	1.00	1.00	1.78	5.24

**Where:** A= 3-amino-5-hydrazino-4- phenyl azo -1*H*-pyrazole B= 3-hydrazino-4- phenyl azo -5-hydroxy-1*H*-pyrazole C= 1-(5-hydroxy-4- phenyl azo -1*H*-pyrazole-3-yl ) 3, 5-dimethylpyrazole

### 1. *P. gossypiella*:

**2.1.1. Accumulative Larval Mortality:** Data presented in Table (3) indicated that all compounds caused significant increases on larval mortality percentage of *P. gossypiella* compared with control. The highest average percentage of larval mortality (24.33%) was obtained with compound A compared with the control which was 5.00% larval mortality.

**1.2. Larval Weight:** Statistical analysis of presented data in Table (3) showed that all tested chemicals caused the highly significant decrease in larval weight of *P. gossypiella* than control.

**1.3. Larval Duration:** Data in Table (3) indicated that pyrazoles caused prolongation in the duration of the larval stage of *P. gossypiella*, which was a significant effect between the tested compounds compared with untreated larvae.

The mean larval durations were 17.75, 16.50 and 17.53 days for A, B and C compounds, respectively compared with 16.41 days for control.

**1.4. Pupal Weight:** The result concerning the effect of the tested compounds on the pupal weight of *P. gossypiella* indicated a significant effect (Table 3). Between the tested compounds as well as control.

**1.5. Pupal Duration:** Data presented in Table (3) indicated that all the tested compounds caused insignificant decreases on pupal durations of *P. gossypiella* compared with control. The lowest pupal periods was 8.00days for B compound. While, it was 9.12 days for untreated control.

**1.6. Pupation Percentage:** The effect of the three tested compounds on the pupation percentage of *P. gossypiella* was shown in Table (3) statistical analysis of data indicated that the effect of the tested compounds on pupation percentage was significant. The highest average of pupation percentage was 79.16% for C compound. While, the lowest one was 60.83% for B compound compared with 93.33% for untreated check. Generally, all tested chemical compounds caused decreased pupation percentages than control.

Table (3): Latent effect of three pyrazoles compounds on immature stages of *P.gossypiella* 1<sup>st</sup> instar larvae laboratory strain at LC<sub>25</sub> values.

Treatments	Larval mortality %	Larval duration (days)	Larval weight (gram)	Pupal weight (gram)	Pupal duration (days)	% Pupation
A	24.33 <sup>a</sup>	17.75 <sup>a</sup>	0.02772 <sup>ab</sup>	0.01756 <sup>ab</sup>	8.66	70.83 <sup>b</sup>
B	21.66 <sup>a</sup>	16.50 <sup>b</sup>	0.02134 <sup>c</sup>	0.0139 <sup>4c</sup>	8.00	60.83 <sup>b</sup>
C	23.00 <sup>a</sup>	17.53 <sup>ab</sup>	0.02406 <sup>b</sup>	0.01630 <sup>bc</sup>	8.33	79.16 <sup>ab</sup>
Control	5.00 <sup>b</sup>	16.41 <sup>a</sup>	0.03042 <sup>a</sup>	0.02013 <sup>a</sup>	9.12	93.33 <sup>a</sup>
P- value	**0.0006	*0.0355	*0.0073	*0.01280	N.S.0.5267	*0.0346
LSD <sub>0.05</sub>	6.83207	1.04	8.4455	6.9496	-	20.56

**Where:** A= 3-amino-5-hydrazino-4- phenyl azo -1*H*-pyrazole B= 3-hydrazino-4- phenyl azo -5-hydroxy-1*H*-pyrazole C= 1-(5-hydroxy-4- phenyl azo -1*H*-pyrazole-3-yl ) 3, 5-dimethylpyrazole

\* = significant \*\* = highly significant P= Probability

Within the same column and source data followed by the same letter are not significantly different (P > 0.05; LSD means separately.)

**1.7. Adult Emergence:** Statistical analysis of adult emergence, the present data in Table (4) indicated that the lowest adults emergence was 56.60% for compound B compared with 100.00% for control. Generally, it was noticed that pyrazole compounds caused lower adult emergence than control.

**1.8. Sex Ratio:** Date in Table (4) showed that the effect of the tested compounds on the calculated sex ratio of *P. gossypiella* adults was insignificantly affected as compared with control.

**1.9. Adult Longevity:** Data in Table (4) indicated that the three tested compounds had insignificant and significant effect on the *P. gossypiella* adult longevity. The lowest mean periods were 11.66 and 14.33 days for A compound as compared with 15.00 and 15.33 days for both male and female adults for control.

**1.10. Fecundity:** The number of laid eggs of *P. gossypiella* was significantly affected by treatment the three tested compounds compared with control. The lowest mean number of laid eggs averaged 103.83 eggs /female for B compound compared with 287.25 eggs /female at control. Generally, the fecundity of female produced from newly hatched larvae treated with pyrazole compounds was greatly reduced than that obtained from control Table (4).



**1.11. Hatchability Percentage:** Data presented in Tables (4) showed that compound A was the highest compound affect hatchability percentage. Also, the tested compounds caused significant reduction in the viability of laid eggs by *P. gossypiella*. Generally, the pyrazole compounds caused higher reduction than that of control on the hatchability rate.

Table (4): Latent effect of three pyrazoles compounds mature stages against 1<sup>st</sup> instar larvae of *p.gossypiella* laboratory strain at LC<sub>25</sub> values

Treatments	% Adult emergence	Sex ratio / ♀	Adult longevity/ (days)		Fecundity /female	% Hatchability
			♂	♀		
A	71.66 <sup>bc</sup>	57.50	11.66	14.33c	163.33b	61.48c
B	56.60 <sup>c</sup>	59.04	12.00	17.33a	103.83c	65.39bc
C	88.57 <sup>ab</sup>	42.22	14.63	17.06ab	147.00bc	79.45ab
Control	100.00 <sup>a</sup>	64.81	15.00	15.33bc	287.25a	88.00a
P-value	*0.0043	N.S.0.3250	N.S.0.0833	*0.0182	**0.0001	*0.0140
LSD <sub>0.05</sub>	10.05	-	-	6.1162	31.1412	6.7276

**Where:** A= 3-amino-5-hydrazino-4- phenyl azo -1H-pyrazole B= 3-hydrazino-4- phenyl azo -5-hydroxy-1H-pyrazole C= 1-(5-hydroxy-4- phenyl azo -1H-pyrazole-3-yl ) 3, 5-dimethylpyrazole  
\* = significant \*\* = highly significant P= Probability

## 2. *S. littoralis*:

**2.1. Larval mortality:** Data presented in Table (5) indicated that all the tested compounds caused a significant increase in larval mortality percentage of 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis*. The highest average larval mortality percentage was 35.33 and 20.33% was obtained with compound B compared with the control which was 3.00 and 3.66 % larval mortality.

**2.2. Larval Duration:** Data in Table (5) indicated that pyrazoles caused prolongation in the duration of the larval stage of 2<sup>nd</sup> instar larva of *S. littoralis* which was significant effect between the treated and untreated larvae for all compounds. The mean larval durations were 21.33, 20.56 and 21.76 days for A, B and C compounds, respectively, compared with 16.86 days for control. While in case of the effect of pyrazoles compounds against the 4<sup>th</sup> instar larvae prolongation in the duration of *S. littoralis* was noticed and significant differences found between the tested compounds only with compound B and C as compared with untreated larvae. The mean larval durations were 19.43, 17.96 and 17.00 days for A, B and C compounds, respectively, compared with 13.83 days for control.

**2.3. Larval Weight:** Statistical analysis of presented data in Table (5) showed that all tested chemicals caused the highly significant decrease in larval weight of 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis* than control .

**2.4. Pupal Duration:** Data presented in Table (5) indicated that all tested compounds caused insignificant decreases on pupal durations of 2<sup>nd</sup> and 4<sup>th</sup> instars larval of *S. littoralis* compared with control. The lowest pupal periods were 12.60, 10.50 days for B compound as compared with 8.07 and 8.10 days for control, respectively.

**2.5. Pupal Weight:** The result concerning the effect of the tested compounds on the pupal weight of 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis* indicated a significant effect between the tested compounds and control treatment. (Table 5).

**2.6. Pupation Percentage:** The effect of the three tested compounds on the pupation percentage of 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis* was shown in Table (5). Statistical analysis of data indicated that the effect of the tested compounds on pupation percentage was significant. The highest average of pupation percentage was 62.00 and 73.33% for A compound. While, the lowest one was 51.10 and 58.20 % for C compound compared with 99.66 and 99.33 % for untreated check. Generally, all tested chemicals resulted in pupation percentages less than control

Table (5): Latent effect of the three pyrazole compounds against immature of 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis* treated with  $lc_{50}$  values.

Treatments	Larval instars	Larval mortality	Larval duration (days)	Larval weight (gram)	Pupal weight (grams)	Pupal duration (days)	% Pupation
A	2 <sup>nd</sup>	26.33 <sup>a</sup>	21.33 <sup>ab</sup>	0.02957 <sup>c</sup>	0.02476 <sup>b</sup>	12.83 <sup>b</sup>	62.00 <sup>b</sup>
	4 <sup>th</sup>	17.33 <sup>a</sup>	19.43 <sup>a</sup>	0.0288 <sup>b</sup>	0.02615 <sup>b</sup>	13.10 <sup>a</sup>	73.33 <sup>b</sup>
B	2 <sup>nd</sup>	35.33 <sup>a</sup>	20.56 <sup>b</sup>	0.03054 <sup>b</sup>	0.02750 <sup>a</sup>	12.60 <sup>b</sup>	56.53 <sup>b</sup>
	4 <sup>th</sup>	20.33 <sup>a</sup>	17.96 <sup>ab</sup>	0.0279 <sup>b</sup>	0.02493 <sup>b</sup>	10.50 <sup>b</sup>	67.03 <sup>bc</sup>
C	2 <sup>nd</sup>	27.33 <sup>a</sup>	21.76 <sup>a</sup>	0.03167 <sup>ab</sup>	0.02743 <sup>a</sup>	14.40 <sup>a</sup>	51.10 <sup>b</sup>
	4 <sup>th</sup>	16.33 <sup>a</sup>	17.00 <sup>b</sup>	0.0239 <sup>c</sup>	0.02469 <sup>b</sup>	12.40 <sup>a</sup>	58.20 <sup>c</sup>
Control	2 <sup>nd</sup>	3.00 <sup>b</sup>	16.86 <sup>c</sup>	0.03723 <sup>a</sup>	0.03102 <sup>a</sup>	8.07 <sup>c</sup>	99.66 <sup>a</sup>
	4 <sup>th</sup>	3.66 <sup>b</sup>	13.83 <sup>c</sup>	0.0380 <sup>a</sup>	0.0337 <sup>a</sup>	8.10 <sup>c</sup>	99.33 <sup>a</sup>
P-value	2 <sup>nd</sup>	*0.0003	**0.000	**0.0264	**0.000	**0.000	**0.0002
	4 <sup>th</sup>	*0.0123	**0.0000	**0.000	*0.001	**0.0000	**0.0000
LSD <sub>0.05</sub>	2 <sup>nd</sup>	9.73814	0.8645	0.06031	0.0709	0.7913	14.3894
	4 <sup>th</sup>	9.04	1.57	0.0387	0.0536	1.58	12.47

**Where:** A= 3-amino-5-hydrazino-4- phenyl azo -1H-pyrazole B= 3-hydrazino-4- phenyl azo -5-hydroxy-1H-pyrazole C= 1-(5-hydroxy-4- phenyl azo -1H-pyrazole-3-yl ) 3, 5-dimethylpyrazole

\* = significant \*\* = highly significant P= Probability

With in the same column and source data followed by the same letter are not significantly different (P > 0.05; LSD mean separately.)

**2.7. Adult Emergence:** Statistical analysis of data in Table (6) indicated that the tested compounds significantly decreased adult emergence for 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis*, but the percent of adult's emergence of 4<sup>th</sup> instar larvae of *S. littoralis*. The lowest mean of adult's emergence was averaged 63.66 and 66.86 % for C compound compared with 86.66 and 95.86 % for control. Generally, it was noticed that pyrazole compounds caused lower adult emergence than control

**2.8. Sex Ratio/Female:** Data in Table (6) showed that the effect of the three tested compounds 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis*. The sex ratio was significantly affected by the three tested compounds compared with control. The percent sex ratio of female has differed slightly from compound to another.

**2.9. Adult Longevity:** Data in Table (6) indicated that the three tested compounds had insignificant effect on the *S. littoralis* male and female longevity treated as 2<sup>nd</sup> and 4<sup>th</sup> instar larvae. The lowest mean of adult longevity were 9.00, 8.33 and 4.83, 5.66 days for A compound as compared with 9.33, 7.93 and 8.13, 7.70 days for control, respectively.

**2.10. Fecundity:** The number of laid eggs of *S. littoralis* treated as 2<sup>nd</sup> and 4<sup>th</sup> instar larvae were significantly affected by treatment with A and C compounds compared with control, Table (6).

The highest compound noticed were A and C recorded 123.33 and 121.33 eggs /female comparing with 503.33 and 663.33 eggs /female at control. Generally, the fecundity of female produced from newly hatched larvae treated with pyrazole compounds was greatly reduced than that obtained from control.

Table (6): Latent effect of the three pyrazole compounds against mature of 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis* treated with  $LC_{50}$  values.

Chemicals	Larval instars	% Adult emergence	Sex ratio %/ ♀	Adult longevity (days)		Fecundity	% Hatchability
				♂	♀		
A	2 <sup>nd</sup>	65.10 <sup>b</sup>	64.03 <sup>a</sup>	9.00	8.33	123.33 <sup>b</sup>	73.11 <sup>b</sup>
	4 <sup>th</sup>	80.00 <sup>b</sup>	71.03 <sup>a</sup>	4.83 <sup>b</sup>	5.66 <sup>b</sup>	196.83 <sup>b</sup>	79.66 <sup>b</sup>
B	2 <sup>nd</sup>	66.00 <sup>b</sup>	66.63 <sup>a</sup>	9.83	8.66	162.33 <sup>b</sup>	55.57 <sup>c</sup>
	4 <sup>th</sup>	76.25 <sup>bc</sup>	54.86 <sup>ab</sup>	5.00 <sup>b</sup>	7.00 <sup>a</sup>	130.00 <sup>b</sup>	68.21 <sup>b</sup>
C	2 <sup>nd</sup>	63.66 <sup>b</sup>	55.11 <sup>b</sup>	10.33	9.00	210.00 <sup>b</sup>	60.00 <sup>bc</sup>
	4 <sup>th</sup>	66.86 <sup>c</sup>	46.66 <sup>b</sup>	5.33 <sup>b</sup>	6.50 <sup>ab</sup>	121.33 <sup>b</sup>	69.75 <sup>b</sup>
Control	2 <sup>nd</sup>	86.66 <sup>a</sup>	62.00 <sup>ab</sup>	9.33	7.93	503.33 <sup>a</sup>	93.00 <sup>a</sup>
	4 <sup>th</sup>	95.86 <sup>a</sup>	65.33 <sup>a</sup>	8.13 <sup>a</sup>	7.70 <sup>a</sup>	663.33 <sup>a</sup>	97.47 <sup>a</sup>
P-value	2 <sup>nd</sup>	*0.0346	*0.0411	N.S.0.6761	N.S.0.7963	**0.0001	**0.0018
	4 <sup>th</sup>	**0.0000	*0.002	**0.0042	*0.005	**0.0000	**0.0000
LSD <sub>0.05</sub>	2 <sup>nd</sup>	16.3215	7.6461	2.6208	2.5488	94.9577	15.0707
	4 <sup>th</sup>	10.82	16.53	1.32	1.21	93.64	12.96

Where:

A= 3-amino-5-hydrazino-4- phenyl azo -1*H*-pyrazole B= 3-hydrazino-4- phenyl azo -5-hydroxy-1*H*-pyrazole

C= 1-(5-hydroxy-4- phenyl azo -1*H*-pyrazole-3-yl ) 3, 5-dimethylpyrazole

\* = significant \*\* = highly significant P= Probability

With in the same column and source data followed by the same letter are not significantly different (P > 0.05; LSD mean separately.)

**2.11. Hatchability Percentage:** Present results proved that significant differences found between the three tested compounds and untreated control. The compound B was the highest affect hatchability percentages of laid egg masses recorded 55.57 and 68.21% for both 2<sup>nd</sup> and 4<sup>th</sup> instar larvae of *S. littoralis* compared with 93.00 and 97.47% for control.

The present results are in accordance with those obtained by Giles *et al.*, (1984) studied the effectiveness of benzophenone hydrazones against leaf-feeding insects; they act as stomach poisons with only poor contact activity. Although, it caused insect death affected insects frequently feeding shortly after ingestion and death occur ranged between 48–72h. Holan and smith (1986) found that the aryl hydrazone and nitro phenyl hydrazones were active as the insecticide against *Lucilia cupring* and mosquito larvae. Ester *et al.*, (1997) mentioned that seeds of winter leeks (*Allium porrum*) coatings with fipronil gave a good control against *Thrips tabaci*. Krushelnycky and Reimer (1998) showed that phenyl pyrazoles were active against many species of ants. (Eric *et al.*, 2001) found that phenylpyrazole insecticide fipronil was very toxic to neonate European corn borer larvae in feeding bio assays of treated diet ( $LC_{50} = 3.34$  ng a.i./cm<sup>2</sup>) and to 5<sup>th</sup> instars in topical bio assays ( $LD_{50} = 18.78$  ng /insect). Arthur (2002) working on stored grain insects; adult red flour beetles and maize weevils treated with 10 ppm Ethiprole (pyrazole compound) alone or in combination treatments. The mortalities of maize weevils ranged from 77.9 to 100%, and mortalities of red flour beetles were from 46.2 to 94.2%. Recently Chandler *et al.*, (2004) found that phenyl pyrazoles have been highly toxic to *Esturaine copepod* after 96-h at  $LC_{50}$  of 6.8 Mg /L . Also, Mohamed *et al.* (2007) who stated that phenyl pyrazole has an extended (latent)effect on the reproduction

capacity of the adults. Fikry *et al.*, (2017) stated that pyrazoles highly toxicity and effective on both of mature or immature stages of spiny bollworms, *Earias insulana*.

## REFERENCES

- Abd El-Hafez, A.; A. G. Metwalley and M. R. A. Saleh (1982): Rearing of the pink bollworm, *Pectinophera gossypiella* (Saunders) on kidney bean diet (Lepidoptera: Gelechiidae). *J. Econ. Entomol.*, 49: 559-560.
- Arthur, F. H. (2002): Efficacy of ethiprole applied alone and in combination with conventional insecticides for protection of stored wheat and stored corn. *Prot. Sci.*, 11(1): 134–148.
- Chandler, G. T.; T. L. Cary.; D. C. Volz.; S. S. Walse.; J. L. Ferry and S. L. Klosterhaus (2004): Fipronil effects on estuarine copepod (*Amphiascus tenuiremis*) development, fertility, and reproduction; a rapid life-cycle assay in 96 – well micro plate format. *Environ. Toxicol. Chem.*, 23(1): 117-124.
- Cole, L. M.; R. A. Nicholson and J. E. Casida (1993): Action of phenyl pyrazole insecticides at the GABA gated chloride channel. *Pest. Biochem. Physio.*, 46:47-54.
- El-Defrawi, M. E.; A. Topozada, N. Mansour and M. Zeid. (1964): Toxicological studies on the Egyptian cotton leaf worm, *Prodenia litura* F. susceptibility of different larval instars of prodenia to insecticides. *J. Econ. Entomol.*, 57: 591-593.
- Elnagdi, M. H. and S. O. Abdalla (1973): *J. Prakt. Chem.*, 315, 1009.
- Eric, W. D.; M. E. Scharf. and B. D. Siegfried (2001): Toxicity and neurophysiological effects of fipronil and its oxidative sulfone metabolite on European corn borer larvae (Lepidoptera- Crambidae). *Pest. Biochem. and Physio.*, 71: 97-106.
- Ester, A.; R. devogel and E. Bouma (1997): Controlling *Thrips tabaci* (Lind.) in leek by film-coating seeds with insecticides. *Crop Prot.*, 16: 673 – 677.
- Fikry, R. M.; S. A. Raslan; N. A. Ismail and H. S. El-Tahawe (2017): Acute toxicity and biological effect of some synthetic organic compounds on spiny bollworms. *Egypt. J. Agri. Res.*, 95(2), 2017.
- Finney. D. J. (1971): Probit analysis 3<sup>rd</sup> ed., Cambridge Univ. press, London UK.
- Fisher, A. R. (1950): Statistical methods for research workers. II. Rev. ed., Oliver and Boyd, London.
- Gaaboub, I. A.; H. A. El-kady; E. F. Khayat and A. M. Shawy (2012): Biochemical and histological effect of some plant extracts insecticide (methomyl) and bio insecticide (protecto) against cotton leafworm, *Spodopteralittoralis* (Boisd.). 1<sup>st</sup> internet. Conf. on Biotech. Applications in Agric. Benha Univ. Moshtohor and Hurghada, 18-22, feb.Egypt.
- Giles, D.P.; L.G. Copping and R. J. Willis (1984): Benzophenone hydrazones, a group of novel insecticides. Croydon, United Kingdom, British Crop Prot. Coun., 2: 405 –412.
- Holan, G. and D. R. J. Smith (1986): A new selective insecticidal uncoupler of oxidative phosphorylation. CSIRO. Div. Applied Organic Chem. Gpo Box 4331. Melbourne Vic. 3001. Australia, 42(5): 558 – 560.
- Krushelnycky, P. D. and N. J. Reimer. (1998): Bait preference by the Argentine ant (Hymenoptera : formicidae) in Haleakala National park, Hawaii in Haleakala. National. Park, Maui, Hawaii. *Environ. Entomol.*, 27 (6): 1473 –1481.
- Merdan, A. H. (1968): Studies on the effect of certain chemical entomopathogens on some cotton worms in Egypt. M. Sc. Thesis, Fac. Sci., Cairo Univ., Egypt.

- Metwally, A. G.; A. Abdel-Hafez.; A. Khalifa and M. F. El-Shaarawy (1980): Breeding pink bollworm on different host plants 1<sup>st</sup> Conf. P. Prot. Res. Ins. Cairo. Egypt.
- Mohamed, E. K.; A. A. Amer.; S. A. Raslan and H. S. El-Tahawe (2007): Acute and biological effects of some synthetic organic compounds on pink bollworm. Egypt. J. Agri. Res., 85(5).
- Raslan, S. A. A. (1994): Toxicological studies on certain cotton pests. Ph.D. Thesis, Fac. Agric., Zagazig Univ., 182 pp.
- Scott, W. P. and G. L. Sondgrass (2000): A review of chemical control of the tarnished plant bug in cotton. South West. Entomol., 23: 67-81.
- Snedecor, G. W. (1970): Statically method applied to experiments in agriculture and biology. Iowa State. Univ. Press. Ames., U.S.A.534pp.
- Sun, Y.P (1950): Toxicity index, an improved method of comparing the relative toxicity of insecticides. J. Econ. Entomol., 43: 45-53.
- Zidan, Z. H. and M. I. Abdel- Megeed (1988): New approaches in pesticides and insect control. Arabic Publishing House and Delivery, Cairo: 605 PP. (In Arabic Language ).

### ARABIC SUMMERY

#### التأثير السام والبيولوجي لبعض المركبات العضوية المخلفة على السلالات المعملية لدودة اللوز القرنفيلية و دودة ورق القطن

هند سعد الطحاوي، وفاء عبد المجيد شهاوى، محمد السيد محمد على حجاب  
معهد بحوث وقاية النباتات – مركز البحوث الزراعية – الدقى – جيزه – مصر.

أجريت التجارب المعملية في معهد بحوث وقاية النباتات – فرع الشرقيه – قسم بحوث ديدان اللوز لدراسة التأثير السام والبيولوجي لثلاثة مركبات عضويه مخلفه للبيرازول هي: (أ): ٣ – أمينو-٥-هيدرازينو-٤-أزو فينيل- ١ اتش – بيرازول. (ب): ٣- هيدرازينو -٤-أزو فينيل- ١ اتش – بيرازول. (ج): ١ – (٥-هيدروكسى-٤-أزو فينيل- ١ اتش – بيرازول-٣-يل) و٥-٣-داى ميثيل بيرازول ضد العمر اليرقى الأول لدودة اللوز القرنفيلية و العمر اليرقى الثانى و الرابع لدودة ورق القطن تحت ظروف معملية ثابتة من ٢٧ ± ١ درجة حرارة مئوية و ٧٠ ± ٥ % رطوبة نسبية.

أشارت النتائج أن المركبات المخلفه للبيرازول أعطت سمية مرتفعه ضد العمر اليرقى الأول لدودة اللوز القرنفيلية والعمر اليرقى الثانى والرابع لدودة ورق القطن . وعلى الجانب الآخر وجد أن التأثيرات المتأخره لمركبات البيرازول سببت إطاله فى فترة حياة اليرقات والعذارى لكلا الحشرتين . أيضا سببت خفض معنوى فى أوزان كل من طورى اليرقات والعذارى لكلا الحشرتين وكذلك سببت خفضاً فى فترة حياة الحشرات الكامله وأيضا خفضت المركبات المختبره كميته البيض الموضوع لكلا الحشرتين . كان أعلى خفض فى كميته البيض الموضوعه ١٠,٨٣ بيضه/انثى لدودة اللوز القرنفيلية يليها ١٢٣,٣٣ و ١٢١,٣٣ بيضه / أنثى للعمر اليرقى الثانى والرابع لدودة ورق القطن مقارنة ٢٨٧,٢٥ بيضه /أنثى لدودة اللوز القرنفيلية و ٥٠٣,٣٣ و ٦٦٣,٣٣ بيضه /أنثى فى حاله المقارنه لدودة ورق القطن . أيضا اظهرت النتائج أن المركبات المختبره خفضت نسبة فقس البيض لدودة اللوز القرنفيلية وكانت ٦١,٤٨ % للمركب (أ) & ٥٥,٥٧ و ٦٨,٢١ % خفض فى العمر اليرقى الثانى والرابع لدودة ورق القطن مقارنه ٨٨,٠٠ % فى دودة اللوز القرنفيلية و ٩٣,٠٠ و ٩٧,٤٧ % للعمر اليرقى الثانى والرابع لدودة ورق القطن على التوالي.