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Stereoselective Synthesis and Biological Activities of *O*-(*E*)-1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethyleneamino-*O*-ethyl-*O*-arylphosphorothioates

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ABSTRACT: *To find novel lead compounds having high insecticidal activity, a series of phosphorothioate derivatives containing 1,2,3-triazole and pyridine rings were synthesized by the reaction of 1-{1-[(6-chloropyridin-3-yl)methyl]-5-methyl-1H-1,2,3-triazol-4-yl}ethanone oxime with phosphorochloridothioates. Their structures were confirmed by IR, ^{1}H NMR, ^{31}P NMR, mass spectrometry, and elemental analyses. The structure of* **6c** *was determined by single crystal X-ray diffraction, which is thermodynamically stable* E *isomer. The results of preliminary bioassay indicate that some title compounds possess insecticidal activity to some extent.* © 2008 Wiley Periodicals, Inc. Heteroatom Chem 19:15–20, 2008; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20367

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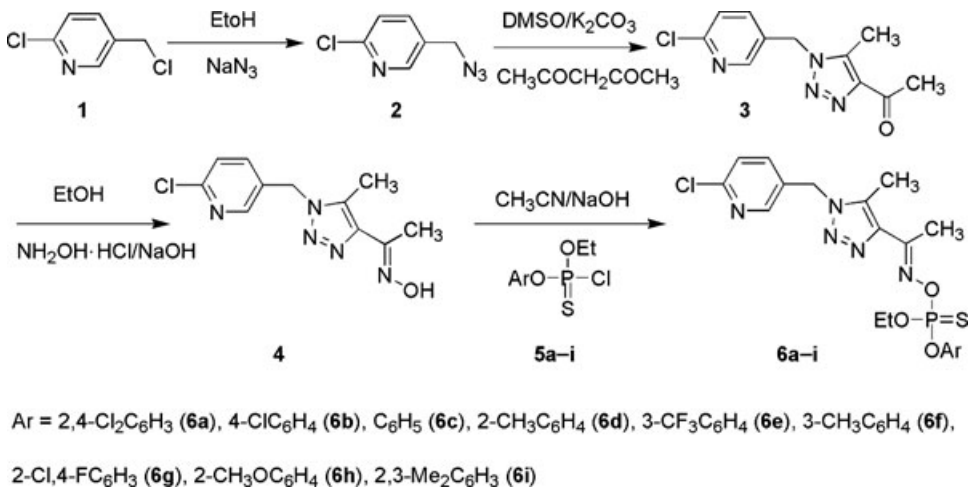
INTRODUCTION

Neonicotinoid insecticides as nicotinic acetylcholine receptor inhibitors have attracted increasing attention because of their safety, low toxicity and high activities [1,2]. Seven neonicotinoids, which have a pyridine-like moiety, have been commercialized. Many structure–activity relationships for these neonicotinoids have been reported [3,4]. It was found that most biologically active nicotinic compounds contain an 3-(aminomethyl)pyridine moiety [5]. The 1,2,3-triazole ring has been known for more than one hundred years. However, it was till recent decades that the 1,2,3-triazole chemistry developed very fast due to the discovery of the diverse biologically active triazole derivatives. Many of them have been used as insecticides, nematocides, acaricides, and plant growth regulators [6–12]. Thiophosphorate oxime ethers and their derivatives play an important role in pesticide science [13,14]. As a continuation of our research work in an attempt to find novel lead compounds having low toxicity and high insecticidal activity [15], we designed and synthesized a type of novel phosphorothioates containing pyridine and triazole rings. Structures of the products were characterized by IR, ^{1}H NMR, ^{31}P NMR, mass spectrometry, and elemental analyses. Results



SCHEME 1 Synthetic routes of the title compounds **6**

of the preliminary bioassay indicate that the title compounds possess potential insecticidal activities to some extent.

RESULTS AND DISCUSSION

Synthesis and Structure of the Products

We synthesized a series of novel phosphorothioate derivatives (**6a–6i**) containing 1,2,3-triazole and pyridine rings as shown in Scheme 1.

2-Chloro-5-(chloromethyl)pyridine **1** was treated with sodium azide in dry ethanol under reflux to obtain 5-azidomethyl-2-chloropyridine **2**, which was cyclized to compound **3** with acetylacetone in DMSO in the presence of anhydrous potassium carbonate. Treatment of compound **3** with hydroxylamine hydrochloride and sodium hydroxide afforded oxime compound **4**, which reacted with various phosphorochloridothioates **5** in anhydrous acetonitrile to give the title compounds **6**.

To optimize the reaction conditions, we attempted to react 1-{1-[(6-chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethanone oxime **4** with phosphorochloridothioates **5a–5i** in various base-solvent systems (e.g., Et_3N/CH_2Cl_2, Et_3N/CH_3CN, Et_3N/toluene, $NaOH/CH_3CN$, NaOH/DMF, and NaOH/toluene). Finally, we found that the reaction can take place smoothly in the presence of sodium hydroxide in acetonitrile at room temperature to give the target products **6** in good yield (77%–89%). Compounds **6a–6i** were characterized by IR, ^{1}H NMR, ^{31}P NMR, EI-MS, and elemental analyses, which are listed in the experimental part.

Because of the C=N bond, it probably existed in *E*- and *Z* isomers in compound **4** and target molecules **6a–6i**. In ^{1}H NMR spectra, the CH_3(1) group protons (Fig. 1) of the *E* isomer is shifted downfield relative to that of the *Z* isomer, owing to its shorter distance between the oxygen atom in C=N—O— moiety and stronger deshielding effect [13]. In this article, both compounds **4** and **6** exist as

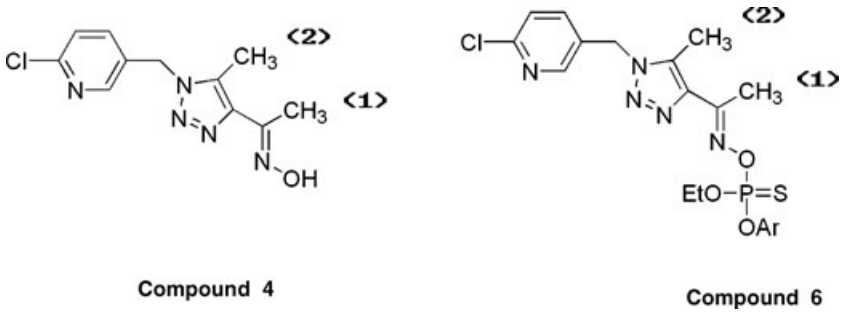


FIGURE 1 Stereo-configurations of compound **4** and **6**.

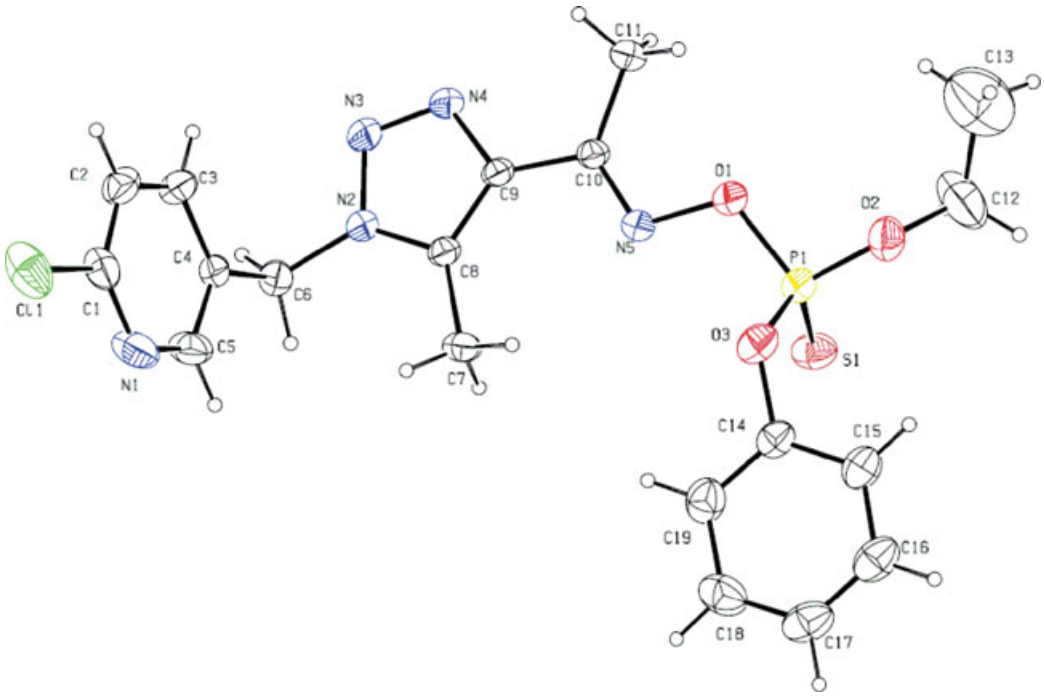


FIGURE 2 Molecular structure of **6c** (50% probability ellipsoids, arbitrary atom numbering).

E isomers, which were confirmed by ^{1}H NMR and single crystal X-ray diffraction studies.

To confirm its molecular configuration and investigate its stereochemistry, a single crystal of **6c** was obtained from ethyl acetate and petroleum ether (1:3 v/v) solvent system. X-ray diffraction analysis indicated that the single crystal of **6c** is triclinic, space group *P*-1, cell parameters $a = 8.549(1)$ Å, $b = 10.746(1)$ Å, $c = 13.805(2)$ Å, $\alpha = 89.373(2)^\circ$, $\beta = 76.631(2)^\circ$, $\gamma = 68.565(2)^\circ$, $V = 1144.7(3)$ Å^3, $Z = 2$, $D_c = 1.352$ g/cm^3, $F(0\ 0\ 0) = 484$, $\mu = 0.36$ mm^{-1}, and final $R = 0.060$, $wR = 0.174$ for 3111 reflections ($I > 2\sigma(I)$). Figures 2 and 3 show the molecular structure of compound **6c** and packing of the molecules in the unit cell, respectively. The selected bond distances and angles are listed in Table 1. The S1—P1—O1, S1—P1—O2, and S1—P1—C3 angles are larger than the O1—P1—O2, O1—P1—C3,

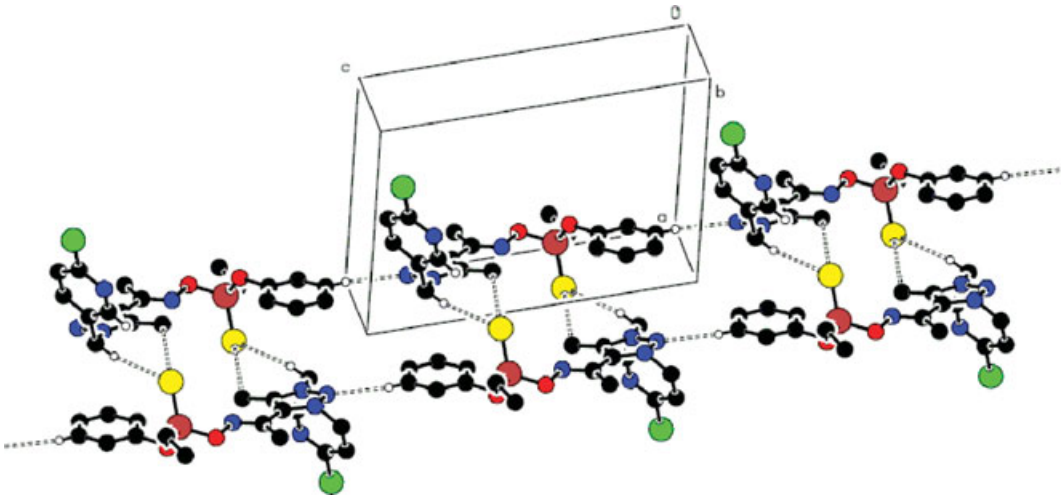


FIGURE 3 Packing of the molecules in the unit cell, showing the formation of C—H···O and C—H···S hydrogen bonds (dashed lines).

TABLE 1 Selected Geometric Parameters

	Bond length (Å)		
O1—P1	1.588(2)	O3—P1	1.577(2)
O2—P1	1.555(3)	P1—S1	1.903(1)
	Bond angle (°)		
O2—P1—O3	103.2(1)	O2—P1—S1	117.6(1)
O2—P1—O1	99.7(1)	O3—P1—S1	115.9(1)
O3—P1—O1	100.9(1)	O1—P1—S1	116.8(1)

and O2—P1—C3 angles, indicating a distorted configuration of the P atom.

In the crystal, weak intermolecular C—H···N hydrogen bonds link molecules into rows along the *c* axis, while weak, inversion related C—H···S hydrogen bonds join parallel rows forming a network (Fig. 3, Table 2). In addition, short intermolecular distances between the centroids of the C8—C9/N2—N4 (Cg1) and the C1—C5/N1 (Cg2) rings of adjacent molecules indicate the presence of π–π stacking interactions in the crystal [16]. [Cg1–Cg2^{i} = 3.894(2) Å; symmetry code: (i) $1-x, 3-y, 2-z$] (Fig. 3). The X-ray structure of **6c** shows that the compound exist as *E* isomer (Fig. 2), which is in accord with the result of ^{1}H NMR analysis.

Insecticidal Activity

Compounds **6a–6i** were tested for insecticidal activity against aphides at a concentration of 0.25 g/L. The results of the preliminary bioassay indicate that some title compounds possess insecticidal activity to some extent (Table 3).

EXPERIMENTAL

Melting points were determined with a WRS-1B digital melting point apparatus, and the thermometer was uncorrected. ^{1}H NMR and ^{31}P NMR spectra were recorded on a VARIAN MERCURY-PLUS400 spectrometer, with TMS and 85% H_3PO_4 as the internal and external references, respectively, and $CDCl_3$ as the solvent, while mass spectra were obtained on a Finnigan TRACEMS2000 spectrometer using the EI method. IR spectra were measured by a NICOLET NEXUS470 spectrometer. Elemental analyses were performed on an ELEMENTAR Vario ELIIICHNSO elementary analyzer. X-ray diffraction analysis was carried out with a BRUKER SMART APEX CCD X-ray diffraction instrument. All of the solvents and materials were reagent grade and purified as required. 5-Azidomethyl-2-chloropyridine **2** and phosphorochloridothioates **5** were prepared according to the literature procedure [17,18].

TABLE 2 Hydrogen Bond Geometry

D—*H*···*A*	*D*—*H* (Å)	*H*···*A* (Å)	*D*···*A* (Å)	*D*—*H*···*A* (°)
C16—H16···N3^{i}	0.93	2.65	3.413(5)	140
C5—H5···S1^{ii}	0.93	2.98	3.877(4)	162
C6—H6A···S1^{ii}	0.97	2.97	3.914(3)	166

Symmetry codes: (i) $x, y, z-1$; (ii) $-x+2, -y+2, -z+1$.

TABLE 3 Insecticidal Activity of Compounds **6a–6i** Against Aphides (0.25 g/L, Inhibitory Rate %)

6a	6b	6c	6d	6e	6f	6g	6h	6i
14.0	2.0	13.0	14.0	18.0	25.0	12.0	8.0	1.0

*Synthesis of 1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethanone* **3**

5-Azidomethyl-2-chloropyridine **2** (8.4 g, 50 mmol) and acetylacetone (5.0 g, 50 mmol) were added to a suspension of milled potassium carbonate (20.7 g, 150 mmol) in DMSO (50 mL). The mixture was stirred at room temperature for 7 h (monitored by TLC), and then it was poured into water (500 mL). The solid was collected by filtration, washed with water and diethyl ether, and finally dried to give 6.8 g of **3** (yield: 81%, m.p. 394–395 K).

^{1}H NMR ($CDCl_3$): δ 2.55 (s, 3H, CH_3), 2.69 (s, 3H, CH_3), 5.52 (s, 2H, CH_2), 7.36 (d, 1H, Py-H, $J = 8.4$ Hz), 7.52 (d, 1H, Py-H, $J = 11.2$ Hz), 8.36 (s, 1H, Py-H)

*Synthesis of 1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethanone Oxime* **4**

Aqueous sodium hydroxide (30% mass concentration, 4 g, 100 mmol) was added dropwise to a stirred solution of hydroxylamine hydrochloride (2.2 g, 32 mmol) and 1-{1-[(6-chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethanone **3** (5.01 g, 20.0 mmol) in 30 mL ethanol. After the addition was complete, the solution was stirred under reflux for 4 to 5 h, the solid was filtered off, and the filtrate was concentrated under vacuum. The residue was purified by column chromatography on a silica gel using petroleum ether and ethyl acetate (1:2 v/v) as the eluent, giving a white solid 2.53 g (yield: 50.6%, m.p. 445–448 K).

IR (KBr, cm^{-1}): ν 3421 (OH), 1419, 1385 (CH_3); ^{1}H NMR ($CDCl_3$): δ 1.54 (s, 1H, OH), 2.42 (s, 3H, CH_3), 2.43 (s, 3H, CH_3), 5.49 (s, 2H, CH_2), 7.34 (d, 1H, Py-H, $J = 8.0$ Hz), 7.50 (d, 1H, Py-H, $J = 10.8$ Hz),

8.35 (s, 1H, Py-H); MS *m/z* (%): 265 (M^+, 10.51), 236 (37.07), 126 (100), 90 (22.27), 77 (8.34), 73 (32.86), 63 (15.77).

General Procedure for the Synthesis of O-*(*E*)-1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethyleneamino*-O-*ethyl*-O-*arylphosphorothioates* **6a–6i**

A solution of 1-{1-[(6-chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethanone oxime (2 mmol) in anhydrous CH_3CN (10 mL) and NaOH powder (2 mmol) was placed in a three-necked flask. After vigorously stirring for 5 min, a solution of the phosphorochloridothioates **5** (2 mmol) in anhydrous CH_3CN (5 mL) was added dropwise. After the addition was complete, the mixture was stirred at room temperature until the reaction was complete (monitored by TLC). The resulting solid was filtered off and the filtrate was concentrated under vacuum. The residue was purified by column chromatography on a silica gel using petroleum ether and ethyl acetate (1:1 v/v) as the eluent, giving the corresponding phosphorothioates **6a–6i** in 77% to 89% yield.

O-*(*E*)-1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethyleneamino*-O-*2,4-dichlorophenyl*-O-*ethylphosphorothioate (***6a***)*. Yellow oil, yield: 88.7%; ^{1}H NMR ($CDCl_3$): δ 1.26 (t, 3H, CH_3), 2.44 (s, 3H, CH_3), 2.57 (s, 3H, CH_3), 4.39 (q, 2H, OCH_2), 5.50 (s, 2H, CH_2), 7.20–7.52 (m, 5H, Ar-H, Py-H), 8.35 (s, 1H, Py-H); MS *m/z* (%): 248 (12.4), 161 (94.6), 126 (100), 90 (28.91), 73 (23.86), 63 (48.24); Anal. calcd. for $C_{19}H_{19}Cl_3N_5O_3PS$: C, 42.67%; H, 3.58%; N, 13.10%; found: C, 42.88%; H, 3.46%; N, 13.19%.

O-*(*E*)-1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethyleneamino*-O-*4-chlorophenyl*-O-*ethylphosphorothioate (***6b***)*. Yellow solid, m.p. 367–368 K, yield: 87.2%; ^{1}H NMR ($CDCl_3$): δ 1.40 (t, 3H, CH_3), 2.46 (s, 3H, CH_3), 2.55 (s, 3H, CH_3), 4.34 (q, 2H, OCH_2), 5.50 (s, 2H, CH_2), 7.14–7.53 (m, 6H, Ar-H, Py-H), 8.35 (s, 1H, Py-H); MS *m/z* (%): 248 (9.63), 128 (100), 126 (95.10), 90 (26.18), 73 (5.41), 63 (6.11); Anal. calcd. for $C_{19}H_{20}Cl_2N_5O_3PS$: C, 45.61%; H, 4.03%; N, 14.00%; found: C, 45.72%; H, 3.97%; N, 13.93%.

O-*(*E*)-1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethyleneamino*-O-*ethyl*-O-*phenylphosphorothioate (***6c***)*. Yellow solid, m.p. 365–367 K, yield: 81.0%; IR (KBr, cm^{-1}) ν 1588 (CH=NO), 1563, 1487, 1458, 1390, 1342 (Ar-H, Py-H), 1035 (P—O—C), 689 (P=S); ^{1}H NMR ($CDCl_3$): δ 1.40 (t, 3H, CH_3), 2.48 (s, 3H, CH_3), 2.57 (s, 3H, CH_3), 4.35 (q, 2H, OCH_2), 5.50 (s, 2H, CH_2), 7.18–7.52 (m, 7H, Ar-H, Py-H), 8.35 (s, 1H, Py-H); ^{31}P NMR ($CDCl_3$): 62.83; MS *m/z* (%): 248 (18.61), 218 (47.60), 126 (87.91), 93 (100), 90 (39.75), 73 (23.18), 63 (46.15); Anal. calcd. for $C_{19}H_{21}ClN_5O_3PS$: C, 48.98%; H, 4.54%; N, 15.03%; found: C, 49.17%; H, 4.65%; N, 15.26%.

O-*(*E*)-1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethyleneamino*-O-*ethyl-O-2-tolylphosphorothioate (***6d***)*. Yellow solid, m.p. 379–381 K, yield: 81.3%; ^{1}H NMR ($CDCl_3$): δ 1.41 (t, 3H, CH_3), 2.31 (s, 3H, CH_3), 2.45 (s, 3H, CH_3), 2.56 (s, 3H, CH_3), 4.34 (q, 2H, OCH_2), 5.50 (s, 2H, CH_2), 7.08–7.51 (m, 6H, Ar-H, Py-H), 8.35 (s, 1H, Py-H); MS *m/z* (%): 479 (M^+, 1.15), 248 (36.78), 232 (59.54), 126 (100), 107 (70.79), 90 (55.18), 73 (20.79), 63 (44.16); Anal. calcd. for $C_{20}H_{23}ClN_5O_3PS$: C, 50.05%; H, 4.83%; N, 14.59%; found: C, 50.18%; H, 4.80%; N, 14.64%.

O-*(*E*)-1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethyleneamino*-O-*ethyl-O-3-(trifluoromethyl)phenylphosphorothioate (***6e***)*. Yellow oil, yield: 85.9%; IR (KBr, cm^{-1}): ν 1586 (CH=NO), 1565, 1484, 1460, 1392, 1334 (Ar-H, Py-H), 1033 (P—O—C), 687 (P=S); ^{1}H NMR ($CDCl_3$): δ 1.40 (t, 3H, CH_3), 2.45 (s, 3H, CH_3), 2.55 (s, 3H, CH_3), 4.36 (q, 2H, OCH_2), 5.50 (s, 2H, CH_2), 7.35–7.53 (m, 6H, Ar-H, Py-H), 8.35 (s, 1H, Py-H); MS *m/z* (%): 286 (8.11), 248 (2.41), 161 (100), 126 (79.93), 90 (22.41), 73 (16.15), 63 (13.45); Anal. calcd. for $C_{20}H_{20}ClF_3N_5O_3PS$: C, 44.99%; H, 3.78%; N, 13.12%; found: C, 44.76%; H, 3.95%; N, 13.06%.

O-*(*E*)-1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethyleneamino*-O-*ethyl-O-3-tolylphosphorothioate (***6f***)*. Yellow solid, m.p. 344–345 K, yield: 85.4%; IR (KBr, cm^{-1}): ν 1592 (CH=NO), 1567, 1491, 1448, 1392, 1327 (Ar-H, Py-H), 1039 (P—O—C), 697 (P=S); ^{1}H NMR ($CDCl_3$): δ 1.40 (t, 3H, CH_3), 2.32 (s, 3H, CH_3), 2.47 (s, 3H, CH_3), 2.55 (s, 3H, CH_3), 4.40 (q, 2H, OCH_2), 5.50 (s, 2H, CH_2), 7.34–7.52 (m, 6H, Ar-H, Py-H), 8.35 (s, 1H, Py-H); ^{31}P NMR ($CDCl_3$): 62.68; MS *m/z* (%): 248 (7.99), 232 (29.74), 126 (100), 107 (34.87), 90 (29.14), 73 (5.31), 63 (5.65); Anal. calcd. for $C_{20}H_{23}ClN_5O_3PS$: C, 50.05%; H, 4.83%; N, 14.59%; found: C, 50.23%; H, 4.58%; N, 14.85%.

O-*(*E*)-1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethyleneamino*-O-*(2-chloro-4-fluorophenyl)*-O-*ethylphosphorothioate (***6g***)*. Yellow oil, yield: 82.9%; ^{1}H NMR ($CDCl_3$): δ 1.41 (t, 3H,

CH_3), 2.45 (s, 3H, CH_3), 2.57 (s, 3H, CH_3), 4.40 (q, 2H, OCH_2), 5.50 (s, 2H, CH_2), 6.95–7.52 (m, 5H, Ar-H, Py-H), 8.35 (s, 1H, Py-H); MS *m/z* (%): 272 (3.31), 248 (28.53), 146 (64.47), 126 (100), 90 (18.73), 73 (6.43), 63 (6.83); Anal. calcd. for $C_{19}H_{19}Cl_2FN_5O_3PS$: C, 44.03%; H, 3.69%; N, 13.51%; found: C, 44.32%; H, 3.51%; N, 13.66%.

O-*(*E*)-1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethyleneamino*-O-*ethyl*-O-*2-methoxyphenylphosphorothioate (***6h***)*. Yellow oil, yield: 82.9%; ^{1}H NMR ($CDCl_3$): δ 1.41 (t, 3H, CH_3), 2.45 (s, 3H, CH_3), 2.56 (s, 3H, CH_3), 4.39 (q, 2H, OCH_2), 3.81 (s, 3H, CH_3), 5.50 (s, 2H, CH_2), 6.88–7.51 (m, 6H, Ar-H, Py-H), 8.35 (s, 1H, Py-H); ^{31}P NMR ($CDCl_3$): 63.98; MS *m/z* (%): 248 (18.53), 126 (100), 123 (95.59), 90 (37.27), 73 (20.62), 63 (37.26); Anal. calcd. for $C_{20}H_{23}ClN_5O_4PS$: C, 48.44%; H, 4.67%; N, 14.12%; found: C, 48.69%; H, 4.47%; N, 14.03%.

O-*(*E*)-1-{1-[(6-Chloropyridin-3-yl)methyl]-5-methyl-1*H*-1,2,3-triazol-4-yl}ethyleneamino*-O-*2,3-dimethylphenyl*-O-*ethylphosphorothioate (***6i***)*. Yellow oil, yield: 77.3%; ^{1}H NMR ($CDCl_3$): δ 1.40 (t, 3H, CH_3), 2.20 (s, 3H, Ar-CH_3), 2.26 (s, 3H, Ar-CH_3), 2.45 (s, 3H, CH_3), 2.56 (s, 3H, CH_3), 4.34 (q, 2H, OCH_2), 5.50 (s, 2H, CH_2), 6.99–7.52 (m, 5H, Ar-H, Py-H), 8.35 (s, 1H, Py-H); MS *m/z* (%): 248 (1.50), 126 (74.35), 121 (56.34), 106 (100), 90 (31.42), 73 (10.90), 63 (19.45); Anal. calcd. for $C_{21}H_{25}ClN_5O_3PS$: C, 51.06%; H, 5.10%; N, 14.18%; found: C, 50.92%; H, 5.18%; N, 13.95%.

INSECTICIDAL BIOASSAY METHOD: AGAINST APHIS BY DIPPING

Two cucumber leaves *ca*. 8 cm in diameter were dipped into a water solution of the test compound for a few seconds until the leaf surface was wet. After drying, the leaves were place on soil in a pet cup. Ten second-instar larvae were released into the cup. The cup was covered with a lid and stored at 25°C°19]. The mortality was assessed 2 and 7 days after treatment. The test was run in triplicate, and the results were averaged and given as activity rate in Table 3.

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