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NOTE

Synthesis of Some new Imidoposphoranes with Possible Insecticidal Activity

ROOPALI MEHRA, ANURAG MOHAN and S.C. MEHRA*
Department of Chemistry, Bareilly College, Bareilly-243 005, India

Various Imidiphosphoranes were synthesized by condensing 2-amino-5-aryl-oxazole with dibromo triphenyl phosphorane in dry benzene in presence of triethyl amine and screened for their insecticidal activities.

Key Words: Synthesis, Imidoposphorane, Insecticidal activity.

Imidophosphoranes in which nitrogen atoms as attached to heterocyclic ring are rare. Due to the increasing biological[1-6] and industrial[7] uses of 1,3,4-oxodiazole derivatives. It was thought worthwhile to synthesize some new imidophosphoranes. The synthetic route was found to be most convenient for desired heterocyclic imidophosphorances, employed the method of Hormen and Oediger[8]. Thus, on condensation of 2-amino-5-aryl-oxazole with dibromo triphenyl phosphorane in dry benzene in the presence of triethyl amine at room temperature to yield the corresponding imidophosphoranes.

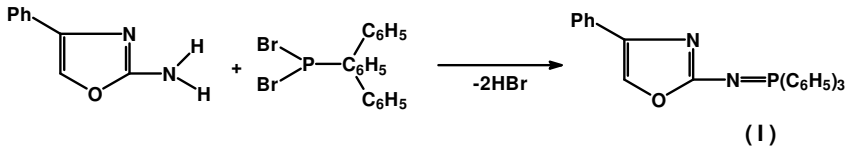
Infrared spectra were determined in KBr pelletes on Bechmann IR-5A unit. All melting points are uncorrected.

The 2-amino-5-aryl-oxazoles were obtained by the reaction of urea and acetophenone in presence of iodine[9].

Preparation of N-(4-subtituted aryl-1,3-oxazol-2-yl)triphenyl imidophosphorane (I):

A well stirred suspension of dibromotriphenyl phosphorane (1 mmol) is 30 mL dry benzene was treated slowly (0.5 h) with a solution of 2-amino-4-phenyl oxazole (1 mmol) and triethyl amine (1 mmol) in 30 mL dry benzene. The mixture was stirred at room temperature for 1 h and then refluxed for 4 h under nitrogen. The precipitated triethylamine hydrochloride was removed by filteration. The analytical data of the other imidophosphoranes are given in Table-1.

The solvent was removed under vacuum and the residue crystallized from benzene. IR (KBr, ν_{max}, cm^{-1}), 1300 due to N=P stretching vibration, 1440 P-C (aromatic) stretching vibration, 1680-1630 due to C=N, stretching vibration. PMR = δ 7.1-7.8 (20 H, m, 4 × Ar-H).



(I)

TABLE-1
ANALYTICAL DATA OF N-(4-SUBSTITUTED ARYL-1,3-OXOZOLYL-2-YL) TRIPHENYL IMIDOPHOSPHORANE

Compd. no.	R	m.f.	Yield (%)	m.p. (ºC)	Elemental analysis (%)	
					Calcd. N	Found N
1	H	$C_{27}H_{21}N_2OP$	45	172	6.66	6.42
2	*p*-Nitro	$C_{27}H_{20}N_3O_3P$	40	196	9.03	8.96
3	*p*-Bromo	$C_{27}H_{20}N_2OPBr$	48	202	5.61	5.38
4	*p*-Methyl	$C_{28}H_{23}N_2OP$	36	203	6.45	6.28
5	*p*-Hydroxy	$C_{27}H_{21}N_2O_2P$	42	162	6.42	6.32
6	*p*-Methoxy	$C_{28}H_{23}N_2O_2P$	44	180	6.22	6.08
7	*p*-Chloro	$C_{27}H_{22}N_2OPCl$	40	194	6.16	5.96
8	*p*-Fluoro	$C_{27}H_{20}N_2OPF$	45	206	6.39	6.18

Hydrolysis of N-(5-aryl-1,3-oxazole-2-yl)triphenylimiophosphoranes: Imidophosphoranes (**I**) (1 g) was refluxed for three hours with dilute HCl (30 mL), then cooled and extracted with benzene. The benzene solution was washed with water and dried over sodium sulfate. Upon evaporation of benzene, triphenyl-phosphine oxide was obtained (70 %), identified by mixed m.p. and comparison of the infrared with an authentic. The parent aminooxazoles were obtained from the acidic solution by neutralization with sodium hydroxide.

Insecticidal activity

Synthesized imidophosphoranes have been screened for insecticidal activities[9]. In the present investigation, the topical method[10] of application by macrometer syringe was employed to test the insect toxicity of compounds on male and female cockroaches (*Pericplanata americema*).

The compounds were dissolved in acetone at two concentrations (0.5 and 0.1 %) and applied topically to individual male and female cockroaches on the dorsal thoracic region between the 4th and 5th segents abdominal) at a dose of 0.02 mL per insect. After treatment, the cockroaches were kept under cover separately and mortality rate counted ranging from 48 h. During the period no food was given and with every sample ten replications were performed and average results were recorded. Parathion was used as standard insecticides in control experiments. The results are given in Table-2.

TABLE-2
INSECTICIDAL ACTIVITY OF SOME IMIDOPHOSPHORANES

Compd.	Mean	KD time (h)
1	5.9	8.6
3	12.0	13.5
4	9.5	10.3
5	13.0	14.0
6	6.5	7.5
7	7.5	9.0
Parathion	4.5	5.5
Acetone	40.0	40.0

Compounds **1**, **6** showed significant mortality. The remaining compounds showed moderate and less activity against cockroaches. All the compounds were less active in comparison to an organophosphorous pesticide (parathion), which was taken as standard.

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