Oriental Journal of Chemistry Vol. **24**(3), 1127-1129 (2008)

Synthesis and characterization of new organophosphorus pesticides: phosphorylated Schiff bases of salicylaldehyde

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(Received: July 07, 2008; Accepted: October 16, 2008)

ABSTRACT

Phosphorylated Schiff bases of salicylaldehyde ie diethyl 2-((phenylimino) methyl) phenyl phosphate and 2-((4-chlorophenylimino) methyl) phenyl diethyl phosphate have been synthesized and characterized using I.R. and ^{1}H NMR spectra.

Key words: Phosphorylated Schiff base, organophosphorus compounds, Schiff base of salicylaldehyde.

INTRODUCTION

Organophosphorus compounds are one of the most important group of modern pesticides[1-8], due to their high insecticidal and acaricidal activity, the broad spectrum and rapidity of action on pests. Various types of organophosphorus compounds have been synthesized and their biological activities have been tested[1-8]. However, literature survey has indicated that phosphorylated Schiff bases of salicylaldehyde have not been synthesized and their biological activities have not been tested. This short communication reports the synthesis and spectral characterization of diethyl 2-((phenylimino) methyl) phenyl phosphate and 2-((4-chlorophenylimino) methyl) phenyl diethyl phosphate.

MATERIAL

O,O-diethyl chlorophosphate was from Across Chemicals, New Delhi, Pyridine was from Qualigens Fine Chemicals, Mumbai.

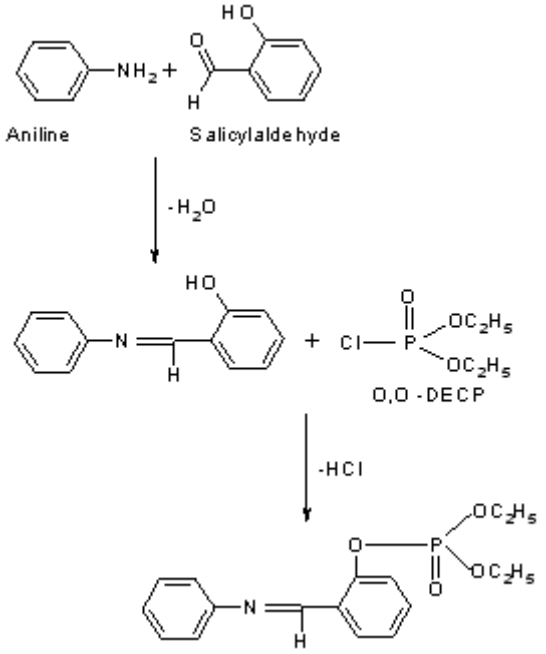
All other chemicals were from Rankem Chemicals, Bangalore and were used without further purification.

METHODS

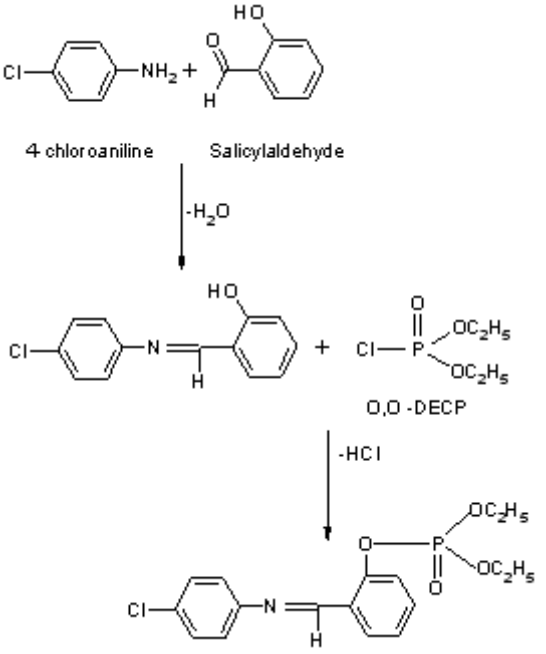
Preparation of 2-((phenylimino) methyl) phenyl diethyl phosphate

Diethyl 2-((phenylimino) methyl) phenyl phosphate was synthesized by the reaction of O-hydroxy benzylidene aniline (0.01 mole) with O,O-diethyl chlorophosphate (0.01 mole) in 30 cm^3 methanol as the solvent and 1 cm^3 pyridine as the catalyst.

The reaction solution was refluxed for 78 hrs, poured into ice-cold water and the resulting solid mass was recrystallized from acetone (mp 120°C, yield 72%). The Schiff base O-hydroxy benzylidene aniline was prepared by the reported method[9], (0.05 mole) each of aniline and salicylaldehyde in 30 cm^3 dimethylformamide was refluxed for 26 hrs, the reaction solution was poured in ice cold water and the resulting solid mass was dried and used in the above reaction.



Scheme 1: Diethyl 2-((phenylimino) methyl) phenyl phosphate



Scheme 2: 2-((4-chlorophenylimino) methyl)phenyl diethyl phosphate

Preparation of 2-((4-chlorophenylimino) methyl) phenyl diethyl phosphate

2-((4-chlorophenylimino) methyl) phenyl diethyl phosphate was synthesized by the reaction of O-hydroxy benzylidene (4- chloroaniline) (0.01 mole) with O,O-diethyl chlorophosphate (0.01 mole) in 30 cm^3 tetrahydrofuran (THF) as the solvent and 1 cm^3 pyridine as the catalyst. The reaction solution was refluxed for 74 hrs, poured into ice-cold water and the resulting solid mass was recrystallized from acetone (mp 101^{0}C, yield 78%). The Schiff base O-hydroxybenzylidene (4-chloroanilene) was prepared by the method used for o-hydroxy benzylidene aniline.

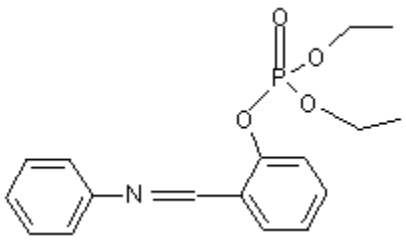
Recording of Spectra:

I.R. spectra in KBr were recorded on a Perkin Elmer 993 I.R. spectrophotometer. ^{1}H NMR spectra were recorded on a Brucker WM (400 MHz), FT spectrometer in DMSO-d6 using TMS as internal reference.

RESULTS AND DISCUSSION

Phosphorylated Schiff bases of salicylaldehyde were synthesized by the reaction of O,O-diethyl chlorophosphate with Schiff bases derived by the condensation of aniline and 4-chloroaniline respectively with salicylaldehyde as shown in scheme 1 and 2 respectively.

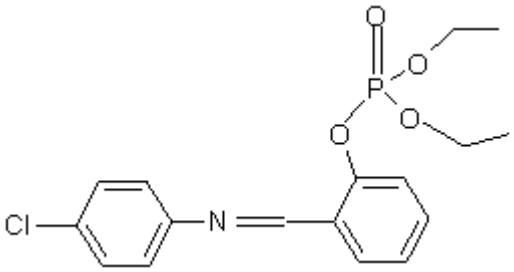
The spectral characterizations of the synthesized compound are as given below.



Diethyl 2-((phenylimino) methyl) phenyl phosphate

IR (KBr): υ 3116 (aromatic C-H strech), 1640 (N=C), 1541 (aromatic C=C strech), 1270 (P=O stretch), 1062, 960 (doublet POC ethyl strech), 902, 765 (mono-substituted benzene ring), 737, 685 cm^{-1}.

^1H NMR (400 Mz; DMSO-d_6): δ 1.29 (t, 6H, J=7.5 Hz, CH_3CH_2O), 4.20 (q, 4H, J=7.5 Hz, CH_3CH_2O), 6.60-7.58 (m, $9H_{arom}$), 8.14 (s, 1H, CH=N).



2-((4-chlorophenylimino) methyl)phenyl diethyl phosphate

IR (KBr): υ 3119 (aromatic C-H strech), 1616 (N=C), 1535 (aromatic C=C strech), 1272 (P=O stretch), 1060, 960 (doublet POC ethyl strech), 830 (p-di-substituted benzene ring), 798, 740,683 cm^{-1}.

^1H NMR (400 Mz; DMSO-d_6): δ 1.30 (t, 6H, J=7.1 Hz, $\mathbf{CH_3}CH_2O$), 4.17 (q, 4H, J=7.1 Hz, $CH_3\mathbf{CH_2}O$), 6.69-7.60 (m, $8H_{arom}$), 8.19 (s, 1H, CH=N).

Attempts to synthesize more new phosphorylated Schiff bases of salicylaldehyde and analysis of their biological activities are underway and results will be published in due course.

ACKNOWLEDGEMENTS

Authors acknowledge thankfully the help rendered by Sophisticated Analysis Instruments Facilities of CDRI, Lucknow in recording the I.R. and ^1H NMR spectra.

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