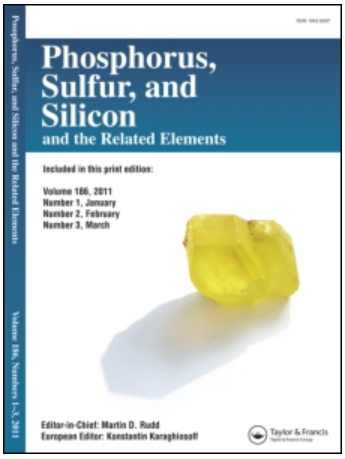

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

Phosphorylation and Thiophosphorylation of 2-Substituted Benzoxazoles and Their Biological Activities

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To cite this Article Soni, K. , Samota, M. K. , Jhajharia, P. and Seth, Gita(2008) 'Phosphorylation and Thiophosphorylation of 2-Substituted Benzoxazoles and Their Biological Activities', Phosphorus, Sulfur, and Silicon and the Related Elements, 183: 11, 2845 — 2853
To link to this Article: DOI: 10.1080/10426500802016968
URL: http://dx.doi.org/10.1080/10426500802016968

Phosphorus, Sulfur, and Silicon, 183:2845–2853, 2008

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426500802016968

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Phosphorylation and Thiophosphorylation of 2-Substituted Benzoxazoles and Their Biological Activities

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Novel organophosphorus compounds have been conveniently prepared using 2-(2′-hydroxynapthyl)benzoxazole and 2-(2′-aminophenyl)benzoxazole as starting materials. Phosphorylation and thiophosphorylation was performed using different molar ratio (1:1, 1:2 and 1:3) of phosphorus oxychloride/thiophosphoryl chloride and substituted benzoxazole. The toxicity of newly synthesized phosphorylated/thiophosphorylated compounds was tested on Aspergillus niger and Fusarium oxysporium. All were found antifungal agents. Plausible structures have been proposed on the basis of IR, ^{1}H NMR, ^{31}P NMR spectral studies.

Keywords 2-(2′-hydroxynapthyl)benzoxazole; 2-(2′-aminophenyl) benzoxazole; phosphorus oxychloride; phosphorus thiochloride; fungicidal activity

INTRODUCTION

The reported biological activity of benzoxazoles[1–3] and organophosphorus compounds[4–5] stimulated our interest to synthesize several phosphorylated/thiophosphorylated derivatives of 2-substituted benzoxazole. Phosphorylated heterocycles have gained importance in a variety of fields due to their antibacterial, antifungal, antiviral, antitumor activities and also as insecticides, herbicides, anthelaminties, histamine antagonists, and fluorescent agents.[6–12] The presence of heterocyclic substitutents in the phosphorylating substances, increases their protonation at the site of action of pesticides and other biocides and facilitates the phosphorylation of acetylcholinesterase enzyme (AchE). The importance of the phosphoryl group has been reported in the literature as it regulates important biological functions.[13–17]

Received 7 January 2008; accepted 27 February 2008.

We are grateful to the head of the Department of Chemistry, University of Rajasthan, Jaipur for providing necessary facilities. Mahesh Kumar Samota is grateful to the council of scientific and Industrial Research (CSIR), New Delhi, India, for financial support in the form of direct senior Research Fellow vide ground. No. 9/149(325)/2K3-EMR-I.

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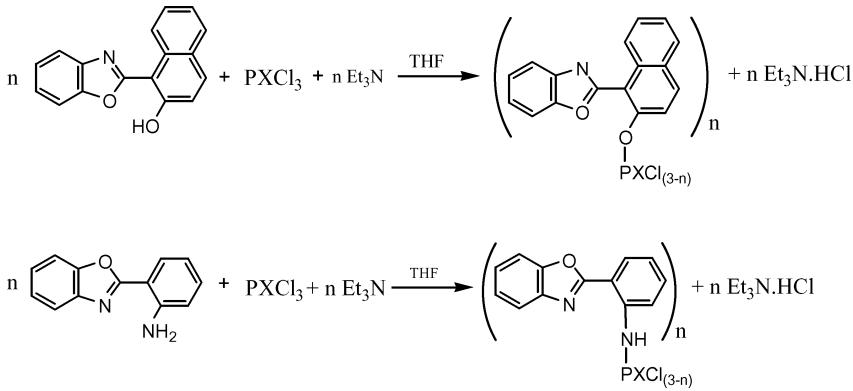
RESULTS AND DISCUSSION

The O-[naphthylbenzoxazolyl-2]phosphorodichloridate/phosphorodichloridothioate, (**1**, **2**), O,O,-bis(napthyl benzoxazolyl-2)phosphorochloridate/phosphorochloridothioate (**3**, **4**) and O,O,O-tris(napthylbenzoxazolyl-2)phosphate/phosphorothioate (**5**, **6**) and NH-(phenylbenzoxazolyl-2) phosphorodichloridoamidate/phosphorodichloridoamidothioate (**7**, **8**), NH,NH-bis (phenylbenzoxazolyl-2) phosphorodiamidochloridate/phosphorochloridodiamidothioate (**9**, **10**) and NH,NH,NH-tri(phenylbenzoxazolyl-2) phosphorotriamidate/phosphorotriamidothioate (**11**, **12**) were synthesized by the dropwise addition of $POCl_3/PSCl_3$ (0.001 mol) in a fast stirring solution of 2-(2′-hydroxynapthyl)benzoxazole and 2-(2′-aminophenyl)benzoxazole (0.001 mol, 0.002 mol, 0.003 mol, respectively) in the presence of stoichiometric amount of triethylamine in THF (Scheme 1). Physical and elemental analysis data of all the compounds are listed in Table I.



Where n = 1, 2, 3
X = O/S

SCHEME 1 Synthesis and structures of compounds **1–12**.

IR Spectra

IR spectra of all these compounds were characterized by ν(P=O), ν(P=S), ν(P–Cl), ν(P–O–C), and ν(P–N–C) bands. P=O and P=S stretching[18–20] and were found in the range of 1260–1295 cm^{-1}. ν(P–O–C) (aryl) were found at 1090–1135 cm^{-1} and 960–975 cm^{-1} and ν(P–N–C)[22] were found at 670–685 cm^{-1} and 1080–1120 cm^{-1} (Table II).[21–22]

TABLE I Physical Properties, and Analytical Data of Phosphorylated and Thiophosphorylated Benzoxazole Derivatives

Compound		Reactants A	Reactants B	Molar ratio of A:B	Melting point (°C)	Yield (%)	Analytical % Cal. (found) C	H	N	P	S	Cl	Molecular weight
(**1**)	($C_{17}H_{10}NO_2$) $P(O)Cl_2$ (Violet)	$C_{17}H_{11}NO_2$	$POCl_3$	1:1	215	53	54 (53.86)	2.66 (2.48)	3.70 (3.56)	8.19 (8.06)	—	18.75 (18.64)	378.15 (376)
(**2**)	($C_{17}H_{10}NO_2$) $P(S)Cl_2$ (Purple)	$C_{17}H_{11}NO_2$	$PSCl_3$	1:1	218	57	51.80 (51.66)	2.56 (2.48)	3.55 (3.42)	7.86 (7.74)	8.13 (8.02)	17.99 (17.86)	394.21 (392.02)
(**3**)	($C_{17}H_{10}NO_2$) $P(O)Cl_2$ (Violet)	$C_{17}H_{11}NO_2$	$POCl_3$	2:1	230	51	67.73 (67.58)	3.34 (3.22)	4.64 (4.48)	5.14 (5.02)	-	5.88 (5.76)	602.97 (600.14)
(**4**)	($C_{17}H_{10}NO_2$) $P(S)Cl_2$ (Dark pink)	$C_{17}H_{11}NO_2$	$PSCl_3$	2:1	Viscous	47	65.97 (65.78)	3.26 (3.18)	4.52 (4.38)	5.00 (4.86)	5.18 (5.06)	5.73 (5.58)	619.03 (618.06)
(**5**)	($C_{17}H_{10}NO_2$) $P(O)Cl_2$ (Violet)	$C_{17}H_{11}NO_2$	$POCl_3$	3:1	240	52	74 (73.86)	3.65 (3.56)	5.08 (4.94)	3.74 (3.48)	—	—	827.79 (827.00)
(**6**)	($C_{17}H_{10}NO_2$) $P(S)Cl_2$ (Violet)	$C_{17}H_{11}NO_2$	$PSCl_3$	3:1	243	48	72.59 (72.45)	3.58 (3.46)	4.98 (4.86)	3.67 (3.46)	3.80 (3.68)	—	843.85 (841.99)
(**7**)	$C_{13}H_9N_2OP(O)Cl_2$ (Yellowish green)	$C_{13}H_{10}N_2O$	$POCl_3$	1:1	204	54	47.73 (47.53)	2.77 (2.60)	8.56 (8.47)	9.47 (9.38)	—	21.68 (21.60)	327.08 (325.00)
(**8**)	$C_{13}H_9N_2OP(S)Cl_2$ (Light yellow)	$C_{13}H_{10}N_2O$	$PSCl_3$	1:1	208	58	45.50 (45.27)	2.64 (2.70)	8.164 (8.22)	9.07 (9.15)	9.34 (9.20)	20.66 (20.61)	343.14 (341.12)
(**9**)	$(C_{13}H_9N_2O)_2P(O)Cl$ (Brownish green)	$C_{13}H_{10}N_2O$	$POCl_3$	2:1	213	52	62.35 (62.31)	3.62 (3.71)	11.18 (11.08)	6.18 (6.15)	—	7.08 (7.00)	500.88 (501.82)
(**10**)	$(C_{13}H_9N_2O)_2P(S)Cl$ (Brown)	$C_{13}H_{10}N_2O$	$PSCl_3$	2:1	219	57	60.41 (60.38)	3.51 (3.48)	10.84 (10.79)	6.00 (5.95)	6.20 (6.28)	6.86 (6.99)	516.95 (518.90)
(**11**)	$(C_{13}H_9N_2O)_3P(O)$ (Brown)	$C_{13}H_{10}N_2O$	$POCl_3$	3:1	222	53	69.43 (69.38)	4.03 (4.00)	12.46 (12.32)	4.59 (4.42)	—	—	674.66 (671.56)
(**12**)	$(C_{13}H_9N_2O)_3P(S)$ (Brown)	$C_{13}H_{10}N_2O$	$PSCl_3$	3:1	230	56	67.82 (67.72)	3.94 (3.89)	12.17 (12.20)	4.48 (4.54)	4.64 (4.70)	—	690.72 (692.64)

TABLE II Assignment of Main IR bands (cm^{-1}) of Phosphorylated and Thiophosphorylated Benzoxazole Derivatives

Compound		IR Bands (cm^{-1})					
		υ(P—O—C)	υ(P═O)	υ(P═S)	υ(P—Cl)	ν(P—N—C)	ν(P—NH)
(**1**)	$(C_{17}H_{10}NO_2)P(O)Cl_2$	1095 965	1282	—	605 (Asym) 525 (Sym)	—	—
(**2**)	$(C_{17}H_{10}NO_2)P(S)Cl_2$	1092 962	—	812 (I) 710 (II)	602 (Asym) 520 (Sym)	—	—
(**3**)	$(C_{17}H_{10}NO_2)_2P(O)Cl$	1122 965	1288	—	525	—	—
(**4**)	$(C_{17}H_{10}NO_2)_2P(S)Cl$	1090 960	—	822 (I) 718 (II)	522	—	—
(**5**)	$(C_{17}H_{10}NO_2)_3P(O)$	1135 975	1295	—	—	—	—
(**6**)	$(C_{17}H_{10}NO_2)_3P(S)$	1225 970	—	795 (I) 718 (II)	—	—	—
(**7**)	$(C_{13}H_9N_2O)P(O)Cl_2$	—	1265	—	600 (Asym) 618 (Sym)	1080 670	3100 2910
(**8**)	$(C_{13}H_9N_2O)P(S)Cl_2$	—	–	825 (I) 700 (II)	608 (Asym) 520 (Sym)	1085 675	3125 2927
(**9**)	$(C_{13}H_9N_2O)_2P(O)Cl$	—	1268	—	605 (Asym) 515 (Sym)	1100 675	3108 2916
(**10**)	$(C_{13}H_9N_2O)_2P(S)Cl$	—	—	830 (I) 715 (II)	610 (Asym) 518 (Sym)	1080 680	3127 2922
(**11**)	$(C_{13}H_9N_2O)_3P(O)$	—	1268	—	—	1120 678	3114 2920
(**12**)	$(C_{13}H_9N_2O)_3P(S)$	—	—	840 (I) 712 (II)	—	1100 685	3135 2927

TABLE III ^{1}H NMR and ^{31}P NMR Spectral Data of Phosphorylated and Thiophosphorylated Benzoxazole Derivatives

Compounds	^{31}P NMR δ (ppm)	^{1}H NMR (δ, ppm)
(**1**) $(C_{17}H_{10}NO_2)P(O)Cl_2$	76.1	7.4–8.0 (m, 10H, Ar-H)
(**2**) $(C_{17}H_{10}NO_2)P(S)Cl_2$	61.3	7.3–8.1 (m, 10H, Ar-H)
(**3**) $(C_{17}H_{10}NO_2)_2P(O)Cl$	72.3	6.9–8.0 (m, 10H, Ar-H)
(**4**) $(C_{17}H_{10}NO_2)_2P(S)Cl$	70.5	7.2–7.9 (m, 10H, Ar-H)
(**5**) $(C_{17}H_{10}NO_2)_3P(O)$	68.5	7.3–8.0 (m, 10H, Ar-H)
(**6**) $(C_{17}H_{10}NO_2)_3P(S)$	70.5	7.4–8.2 (m, 10H, Ar-H)
(**7**) $(C_{13}H_9N_2O)P(O)Cl_2$	75.3	7.5–8.2 (m, 8H, Ar-H) 5.7 (d, 1H, P—NH)
(**8**) $(C_{13}H_9N_2O)P(S)Cl_2$	76.1	7.75–8.2 (m, 8H, Ar-H) 5.6 (d, 1H, P—NH)
(**9**) $(C_{13}H_9N_2O)_2P(O)Cl$	71.1	7.2–8.2 (m, 16H, Ar-H) 5.8 (d, 1H, P—NH)
(**10**) $(C_{13}H_9N_2O)_2P(S)Cl$	72.4	7.3–8.1 (m, 16H, Ar-H) 5.6 (d, 1H, P—NH)
(**11**) $(C_{13}H_9N_2O)_3P(O)$	68.6	7.5–8.2 (m, 24H, Ar-H) 6.0 (d, 1H, P—NH)
(**12**) $(C_{13}H_9N_2O)_3P(S)$	71.3	7.1–8.0 (m, 24H, Ar-H) 5.7 (d, 1H, P—NH)

^{1}H NMR Spectra

Aromatic protons showed their signals at 5.6–8.2 ppm. In phosphorylated/thiophosphorylated derivatives of 2-(2′-hydroxynaphthyl) benzoxazole (O—H) proton peak was found to be absent due to the removal of the H atom by Cl of $POCl_3$/$PSCl_3$. The peak due to P—NH[23] appeared at δ 5.8-6 ppm in different derivatives of phosphorylated / thiophosphorylated compounds (Table III).

^{31}P NMR Spectra

In ^{31}P NMR[24] spectra, only one ^{31}P resonance signal has been observed at δ 61.3–76.2 ppm.

Fungicidal Activity

Bioactivity of all the phosphorylated and thiophosphorylated benzoxazole compounds have been screened against *Aspergillus niger* and *Fusarium oxysporium* at concentrations 50, 100, and 200 ppm by Radial growth method. Radial growth method is a food poison technique in which potato dextrose agar medium was prepared and sterilized.[25] To this medium was added requisite amount of the phosphorylated/thiophosphorylated derivatives of benzoxazoles dissolved in dimethyl formamide. A culture of test fungus is then grown on this culture media. The results thus obtained for fungicidal screening of the phosphorylated and thiophosphorylated derivatives with

TABLE IV Fungitoxic Screening Data of Phosphorylated and Thiophosphorylated Benzoxazole Derivatives

		Percent mycelial inhibition					
		Aspergillus niger compound dose (ppm)			*Fusarium oxysporium* compound dose (ppm)		
Compound		50	100	200	50	100	200
(1)	$(C_{17}H_{10}NO_2)P(O)Cl_2$	17.5	55.4	71.6	19.8	44.1	64.3
(2)	$(C_{17}H_{10}NO_2)P(S)Cl_2$	36.2	61.6	75.8	40.5	55.6	73.3
(3)	$(C_{17}H_{10}NO_2)_2P(O)Cl$	46.4	70.2	84.7	49.4	66.4	77.8
(4)	$(C_{17}H_{10}NO_2)_2P(S)Cl$	22.1	59.7	77.1	35.2	56.2	68.6
(5)	$(C_{17}H_{10}NO_2)_3P(O)$	50.3	74.0	80.7	44.7	75.9	81.7
(6)	$(C_{17}H_{10}NO_2)_3P(S)$	68.2	81.9	91.2	70.6	84.7	93.9
(7)	$C_{13}H_9N_2OP(O)Cl_2$	37.8	47.1	70.3	38.0	49.2	72.5
(8)	$C_{13}H_9N_2OP(S)Cl_2$	39.2	55.1	73.9	37.2	57.1	78.2
(9)	$(C_{13}H_9N_2O)_2P(O)Cl$	43.4	59.1	78.8	45.2	59.5	81.6
(10)	$(C_{13}H_9N_2O)_2P(S)Cl$	50.4	64.5	82.1	50.8	65.7	85.3
(11)	$(C_{13}H_9N_2O)_3P(O)$	62.4	70.1	85.6	66.7	74.5	88.2
(12)	$(C_{13}H_9N_2O)_3P(S)$	70.6	82.3	92.5	72.5	86.1	93.6
Dithane M-45		75	90	100	73	92	100

standard Dithane M-45 are furnished in Table IV. Results show, that these derivatives are more toxic than the preliminary ligand 2-(2′-hydroxynapthyl)benzoxazole and 2-(2′-aminophenyl) benzoxazole and less toxic as compared with standard Diethane M-45.

EXPERIMENTAL

IR spectra of all these compounds were recorded as KBr discs or Nujol Mulls on Nicolet Magna 550 FTIR spectrophotometer in the range 4000–200 cm^{-1}. ^1H NMR spectra were scanned on a JEOL FX 90Q/JEOL AL 300 MHz FTNMR spectrometer in deuterated dimethyl sulfoxide (DMSO-d_6) and deuterated chloroform ($CDCl_3$) using TMS as internal reference at room temperature. ^{31}P NMR spectra were scanned on a JEOL AL 300 MHz FTNMR spectrometer at 121.49 MHz in DMSO-d_6 or $CDCl_3$ using TMS and 85% H_3PO_4 as internal and external references respectively at room temperature.

Synthesis of O-(naphthylbenzoxazolyl-2) Phosphorodichloridate/phosphorodichloridothioate

2-(2′-hydroxynaphthyl) benzoxazole (0.001 mol) in dry THF (30 ml) and Et_3N (0.001 mol) were taken in a round bottom flask and stirred. A

solution of $POCl_3/PSCl_3$ (0.001 mol) in dry THF (30 ml) was added dropwise via dropping funnel. After mixing the reactants, stirring was continued for 4 h at 0°C. Further the reaction was removed from the ice bath and then refluxed further under nitrogen atmosphere for 14–16 h with continuous stirring. It was then cooled and filtered through a closed sintered funnel to separate triethylamine hydrochloride ($Et_3N.HCl$) formed during the reaction. The filtrate was concentrated and kept for crystallization in dessicator for 2 days. The product was recrystallized in ethanol and dried in vacuo.

Synthesis of O,O-bis(naphthylbenzoxazolyl-2) Phosphorochloridate/phosphorochloridothioate

2-(2′-hydroxynaphthyl)benzoxazole (0.002 mol) in dry THF and Et_3N (0.002 mol) were taken in a round bottom flask and stirred. A solution of $POCl_3/PSCl_3$ (0.001 mol) in dry THF (30 ml) was added dropwise by dropping funnel. Then the reaction was carried out in a manner similar as described above.

Synthesis of O,O,O-tris(naphthyl benzoxazolyl-2) phosphate/phosphorothioate

The solution of $POCl_3/PSCl_3$ (0.001 mol) in dry THF (30 ml) was added dropwise, in a fast stirring ice cold solution of 2-(2′-hydroxynaphthyl)benzoxazole (0.003) in dry THF (30 ml) and Et_3N (0.003 mol). The reaction was carried out in a manner similar as described above.

Synthesis of NH-(phenylbenzoxazolyl-2)phosphorodichloridoamidate/phosphorodichloridoamidothioate

To the solution of 2-(2′-aminophenyl)benzoxazole (0.001 mol) in dry THF (30 ml) and Et_3N (0.001 mol) in dry THF (20 ml) a solution of $POCl_3/PSCl_3$ (0.001 mol) in dry THF (30 ml) was added dropwise. Then, the reaction mixture was carried out in a manner similar as described above.

Synthesis of NH,NH-bis(phenylbenzoxazolyl-2) phosphorodiamidochloridate/phosphorochloridodiamidothioate

In a fast stirring solution of 2-(2′-aminophenyl)benzoxazole (0.002 mol) in dry THF (30 ml) and Et_3N (0.002 mol) in dry THF (30 ml), a solution of $POCl_3/PSCl_3$ (0.001 mol) in dry THF (30 ml) was added dropwise by

dropping funnel. Then the reaction was carried out in a manner similar to described above.

Synthesis of NH,NH,NH-tris(phenylbenzoxazolyl-2)phosphorotriamidate/phosphorotriamidothioate

The solution of $POCl_3/PSCl_3$ (0.001 mol) in dry THF (30 ml) was added dropwise in a fast stirring ice-cold solution of 2-(2′-aminophenyl)benzoxazole (0.003 mol) in dry THF (20 ml). Then the reaction was carried out in a manner similar to described above.

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