Heteroatom Chemistry
Volume 5, Number 5/6, 1994

Synthesis and Spectral Analysis of 6-Substituted-1,2,4,8,10,11-Hexachloro-12H-dibenzo[d,g][1,3,2]dioxaphosphocin 6-Oxides

B. Sankara Reddy, C. Devendranath Reddy,* and P. Mallikarjuna Reddy

Department of Chemistry, Sri Venkateswara University, Tirupati 517 502, India

Received 18 July 1994; revised 19 August 1994

ABSTRACT

Synthesis of several 6-substituted-1,2,4,8,10,11-hexachloro-12H-dibenzo[d,g][1,3,2]dioxaphosphocin 6-oxides and their IR, ^1H, ^{13}C, and ^{31}P NMR and mass spectral analyses are described. Observation of geminal coupling [$^2J_{(H,H)}$ = 16.0 Hz] between bridged methylene protons (12-CH_2) suggested their nonequivalence and is consistent with a boat-chair form which may be in conformational equilibrium with either a boat-boat or a distorted and extended boatlike conformation.

INTRODUCTION

Organophosphorus compounds have multifaceted applications [1,2]. The chemistry of dibenzo dioxaphosphocins has gained considerable importance recently because of the latter's use as antioxidants [3] and as superior ligands in transition-metal-mediated hydroformation reactions [4]. As part of our continuing investigations [5], syntheses of 6-aryloxy/arylthio-1,2,4,8,10,11-hexachloro-12H-dibenzo[d,g][1,3,2]dioxaphosphocin 6-oxides (**3a–3o**) and analyses of their IR, NMR, and mass spectra were accomplished.

RESULTS AND DISCUSSION

The reactions of 2,2'-methylenebis(3,4,6-trichlorophenol) (**1**) with aryl phosphorodichloridates (**2**) in the presence of triethylamine in dry toluene at 50–60°C afforded compounds **3a–3k** (Scheme 1).

On the other hand, compounds **3l–3o** were prepared by an alternative method (Scheme 1) involving the preparation of 6-chloro-1,2,4,8,10,11-hexachloro-12H-dibenzo[d,g][1,3,2]dioxaphosphocin 6-oxide (**4**) as an intermediate. Cyclization of **1** with phosphorus oxychloride in the presence of triethylamine in dry toluene at 45–55°C for 3 hours produced **4**, which, on subsequent condensation in situ with members of **5l–5o** in the same reaction vessel under the same conditions, led to the formation of corresponding products (**3l–3o**).

	R		R
3a	C_6H_5O	**3i**	2'-ClC_6H_4O
3b	2'-$H_3CC_6H_4O$	**3j**	4'-ClC_6H_4O
3c	3'-$H_3CC_6H_4O$	**3k**	4'-$O_2NC_6H_4O$
3d	4'-$H_3CC_6H_4O$	**3l**	4'-$(H_3C)_3CC_6H_4O$
3e	2',3'-$(H_3C)_2C_6H_3O$	**3m**	C_6H_5S
3f	2',4'-$(H_3C)_2C_6H_3O$	**3n**	4'-$H_3CC_6H_4S$
3g	2',6'-$(H_3C)_2C_6H_3O$	**3o**	4'-ClC_6H_4S
3h	3',4'-$(H_3C)_2C_6H_3O$		

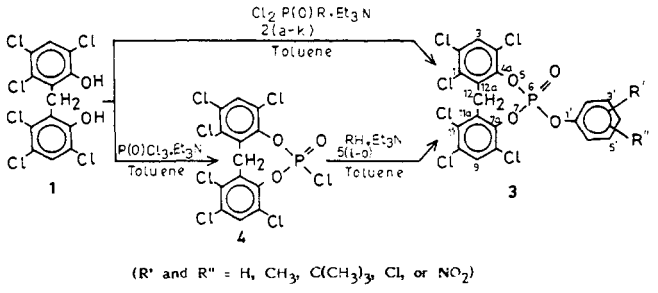
Physical properties, characteristic IR frequencies [6–9] and ^{31}P NMR data of compounds **3** are given in Table 1.

^1H NMR spectra of **3** (Table 2) showed signals at δ 6.98–8.36 for the aromatic protons of the dibenzodioxaphosphocin and 6-aryloxy/arylthio moieties. The singlet at δ 7.52–7.55 was assigned to H(3) and H(9) on the basis of comparison with the chemical shift of the corresponding protons in compound **1**. The bridged methylene protons (12-CH_2) showed two distinct doublets in the regions δ 4.39–4.61 and 4.81–4.90 due to geminal coupling [5,10].

*To whom correspondence should be addressed.

© 1994 VCH Publishers, Inc.

1042-7163/94/$5.00 + .25



SCHEME 1

The expected long-range coupling [$^5J_{(H,P)}$] between phosphorus and one of the bridged methylene protons (12-CH_2) was not observed. The bridged methylene protons (12-CH_2) in certain dibenzodioxaphosphocins, which are largely in the boat-chair conformation, exhibit only geminal coupling [$^2J_{(H,H)}$] and do not show long-range coupling with phosphorus [$^5J_{(H,P)}$] [11]. The $^5J_{(H,P)}$ coupling would not be expected for molecules in a boat-chair conformation with the phosphoryl oxygen in a pseudoequatorial position, based upon the throughspace mechanism for transmission of coupling information involving the lone pair of electrons of the endocyclic oxygen atoms [12]. X-ray crystallographic studies of dibenzodioxaphosphocins showed clearly that they exist in the boat-chair conformation [13].

On the same basis, it may be construed that the title compounds **3** have the boat-chair (BC) conformation, which may be in rapid conformational equilibrium with either a boat-boat (BB) or a distorted and extended boatlike form (Figure 1) [5,11–13].

^{13}C NMR chemical shifts of the dibenzodioxaphosphocin moieties of **3** (Table 3) were interpreted on the basis of a comparison with the ^{13}C

TABLE 1 Physical Data of 6-Substituted-1,2,4,8,10,11-hexachloro-12H-dibenzo[d,g][1,3,2]dioxaphosphocin 6-Oxides (**3**)

				IR (cm^{-1})					
					P–O–C(Ar)				
Compound	*Mp*[a] (°C)	*Yield*[b] (%)	*Molecular Formula*	*P=O*	*O–C*	*P–O*	*^{31}P NMR*[c] (*$CDCl_3$*)	*Found* **C**	*(Required)* (%) **H**
3a	165–166	66	$C_{19}H_9Cl_6O_4P$	1305	1240	995	−16.65	41.82 (41.88)	1.78 (1.67)
3b	120–121	65	$C_{20}H_{11}Cl_6O_4P$	1305	1255	990	—	42.03 (42.97)	1.93 (1.98)
3c	153–154	63	$C_{20}H_{11}Cl_6O_4P$	1300	1250	995	−16.53	42.82 (42.97)	2.12 (1.98)
3d	167–168	67	$C_{20}H_{11}Cl_6O_4P$	1300	1240	990	−16.42	43.07 (42.97)	2.21 (1.98)
3e	179–180	58	$C_{21}H_{13}Cl_6O_4P$	1320	1240	995	−16.04	43.96 (44.02)	2.31 (2.29)
3f	175–176	62	$C_{21}H_{13}Cl_6O_4P$	1275	1250	985	−16.20	43.96 (44.02)	2.31 (2.29)
3g	146–147	65	$C_{21}H_{13}Cl_6O_4P$	1280	1250	990	—	43.95 (44.02)	2.26 (2.29)
3h	123–124	57	$C_{21}H_{13}Cl_6O_4P$	1275	1240	975	−16.42	44.15 (44.02)	2.35 (2.29)
3i	160–161	61	$C_{19}H_8Cl_7O_4P$	1300	1240	970	−16.78	39.31 (39.39)	1.43 (1.39)
3j	154–155	64	$C_{19}H_8Cl_7O_4P$	1275	1240	990	−16.72	39.47 (39.39)	1.46 (1.39)
3k	178–179	62	$C_{19}H_8Cl_6NO_6P$	1295	1255	990	−17.83	38.59 (38.68)	1.42 (1.37)
3l	169–170	48	$C_{23}H_{17}Cl_6O_4P$	1280	1250	995	−16.76	45.82 (45.96)	2.73 (2.85)
3m	168–169	41	$C_{19}H_9Cl_6O_3PS$	1280	1230	970	—	40.57 (40.68)	1.58 (1.62)
3n	147–148	42	$C_{20}H_{11}Cl_6O_3PS$	1275	1250	990	−18.37	41.71 (41.77)	1.87 (1.93)
3o	150–151	46	$C_{19}H_8Cl_7O_3PS$	1280	1235	985	—	38.36 (38.32)	1.38 (1.35)

[a]Recrystallized from ethylacetate-hexane.
[b]Yields were reported after one crystallization.
[c]^{31}P Chemical shifts were expressed in δ, 85% H_3PO_4 being used as external standard.

TABLE 2 ^{1}H NMR Data of 6-Substituted-1,2,4,8,10,11-hexachloro-12H-dibenzo[d,g][1,3,2]dioxaphosphocin 6-Oxides (**3**) from TMS (δ)

Compound[a]	*H(3/9)*	*H(12)*[b]	*R-H*	
3a	7.54	4.43 (16.1), 4.89 (16.1)	7.25–7.40 (m, 5H)	—
3b	7.53	4.45 (15.9), 4.87 (16.2)	7.18–7.40 (m, 4H)	2.34 (s, 3H, CH_3)
3c	7.54	4.45 (15.9), 4.89 (15.9)	7.19–7.35 (m, 4H)	2.37 (s, 3H, CH_3)
3d	7.54	4.43 (16.0), 4.86 (15.9)	7.16–7.27 (m, 4H)	2.35 (s, 3H, CH_3)
3e	7.52	4.44 (16.1), 4.89 (16.0)	7.05–7.37 (m, 3H)	2.23 (s, 3H, CH_3) 2.30 (s, 3H, CH_3)
3f	7.52	4.46 (16.1), 4.90 (16.1)	6.98–7.37 (m, 3H)	2.27 (s, 3H, CH_3) 2.30 (s, 3H, CH_3)
3g	7.53	4.47 (16.1), 4.85 (16.2)	7.06–7.09 (m, 3H)	2.25 (s, 6H, CH_3)
3h	7.53	4.52 (16.0), 4.81 (15.7)	7.11–7.17 (m, 3H)	2.25 (s, 3H, CH_3) 2.27 (s, 3H, CH_3)
3i	7.54	4.54 (15.9), 4.86 (15.9)	7.21–7.46 (m, 4H)	—
3j	7.55	4.41 (16.1), 4.88 (16.0)	7.31–7.41 (m, 4H)	—
3k	7.54	4.43 (16.2), 4.90 (16.1)	7.17 (d, 2H); 8.34 (d, 2H)	—
3l	7.52	4.61 (16.0), 4.89 (16.0)	7.16–7.31 (m, 4H)	1.34 (s, 9H, CH_3)
3m	7.52	4.37 (15.9), 4.85 (15.9)	7.17–7.38 (m, 5H)	—
3n	7.55	4.34 (15.9), 4.83 (16.1)	7.17–7.28 (m, 4H)	2.37 (s. 3H, CH_3)
3o	7.54	4.36 (16.0), 4.82 (15.8)	7.27–7.42 (m, 4H)	—

[a]Recorded in $DCCl_3$.
[b]Values in parentheses are coupling constants J in hertz.

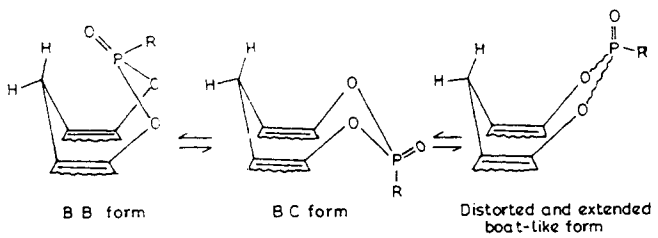


FIGURE 1

NMR data of **1**, additivity rules, C-P couplings, and intensity of signals. The low intensity doublets in the region δ 146.0–146.5 [$^2J_{POC(4a)}$ and $^2J_{POC(7a)}$ = 8.1–8.5 Hz] were assigned to the oxygen-bearing carbons C(4a) and C(7a) [14]. Chemical shifts in the region δ 125.1–125.6 [$^3J_{POCC(4)}$ and $^3J_{POCC(8)}$ = 4.4–6.1 Hz] were attributed to C(4) and C(8) [5,15]. C(11a) and C(12a) appeared as a doublet at δ 129.6–129.8 [$^3J_{POCC(11a,12a)}$ = 3.5–3.7 Hz] [5,15]. The other chlorine-bearing carbons C(1,11) and C(2,10) also showed signals at δ 132.4–132.7 and 131.1–131.6, respectively [14]. The signal of the unsubstituted carbons C(3) and C(9) appeared with high intensity in the region δ 129.8–130.9. The bridged methylene carbon C(12) resonated at δ 30.9 [5].

The carbon chemical shifts of 6-aryloxy (**3a–3k**) and arylthio (**3l–3o**) groups (Table 4) were assigned on comparison with those of related systems [5,14,16]. The doublet at δ 146.6–151.2 [$^2J_{POC(1')}$ = 7.2–8.6 Hz] was assigned to C(1'), while C(2') and C(6') signals occurred in the region δ 117.1–130.9 [$^3J_{POCC(2',6')}$ = 3.6–6.3 Hz]. Chemical shifts for the C(3'), C(4'), and C(5') were observed at δ 125.7–139.3, 125.9–147.7, and 126.1–133.8, respectively, depending on the nature of substituents at various positions. The observed upfield shift of about 4 ppm for the methyl group attached to C(2') (**3b**, **3f**, and **3g**) was attributed to its γ interaction with the exocyclic oxygen [5a,14]. Interestingly, the C(2') methyl group in **3e** experienced further shielding due to its nonbonded interaction with the exocyclic oxygen atom and the C(3') methyl group [5a,14].

^{31}P NMR signals of **3** (Table 1) were observed in the range from δ −16.01 to −18.37 [5].

Electron impact mass spectra of **3** (Table 5) exhibited M^+, $(M\text{-}Cl)^+$, $(M\text{-}Cl_2)^+$, $(M\text{-}OR)^+$, $(M\text{-}RO_2P)^+$, and $(M\text{-}C_{13}H_4O_3Cl_6P)^+$ as characteristic ions, supporting the proposed structures. Occurrence of these ions obviously indicates the reasonable stability of the dioxaphosphocin ring under electron impact.

EXPERIMENTAL

All melting points were determined in open capillary tubes on a Mel-Temp apparatus and are uncorrected. Elemental analyses and mass spectra were obtained from the Central Drug Research Institute, Lucknow, India. IR spectra (γ_{max} in cm^{-1}) were recorded as KBr pellets on a Perkin-Elmer 683 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a Varian XLAA-300 spectrometer operating at 300 MHz for ^{1}H and 75 MHz for ^{13}C, while ^{31}P NMR spectra were taken on a Varian XLAA-400 spectrometer operating at 162 MHz on solutions of the compounds in $DCCl_3$. All chemical shifts

TABLE 3 ^{13}C Chemical Shifts[a] of Dibenzodioxaphosphocin Moieties of **3** (δ)

Compound[b]	C(1/11)	C(2/10)	C(3/9)	C(4/8)	C(4a/7a)	C(11a/12a)	C(12)
3a	132.6 (2.1)	131.3 (2.2)	129.9	125.2 (6.1)	146.0 (8.2)	129.7 (3.6)	30.9
3b	132.6 (2.0)	131.3 (2.1)	129.9	125.3 (6.2)	146.1 (8.4)	129.8 (3.4)	30.8
3c	132.5 (2.1)	131.2 (2.0)	129.9	125.3 (6.1)	146.0 (8.2)	129.7 (3.5)	31.0
3d	132.5 (2.1)	131.2 (2.3)	130.0	125.3 (6.0)	146.0 (8.5)	129.7 (3.5)	30.9
3e	132.6 (2.2)	131.2 (2.2)	130.1	125.2 (6.0)	146.2 (8.2)	129.8 (3.6)	30.9
3f	132.5 (2.2)	131.1 (2.3)	129.9	125.3 (6.0)	146.1 (8.2)	129.7 (3.5)	30.9
3g	132.7 (2.1)	131.3 (2.2)	129.9	125.2 (6.2)	146.2 (8.4)	129.7 (3.4)	30.9
3h	132.6 (2.3)	131.2 (2.3)	130.1	125.4 (6.0)	146.1 (8.4)	129.8 (3.6)	30.7
3i	132.5 (2.4)	131.6 (2.2)	130.0	125.1 (6.3)	146.2 (8.3)	129.7 (3.4)	30.9
3j	132.7	131.5 (2.3)	129.9	125.2 (6.1)	146.1 (8.5)	129.6 (3.7)	30.8
3k	132.7	131.3	129.8	125.5 (4.4)	146.4 (8.4)	129.7 (3.5)	31.1
3l	132.6 (2.2)	131.2 (2.2)	130.0	125.3 (6.1)	146.2 (8.2)	129.7 (3.6)	30.9
3m	132.5 (2.2)	131.4 (2.3)	130.0	125.5 (6.2)	146.0 (8.4)	129.6 (2.3)	31.0
3n	132.4	131.5	130.9	125.6	146.4 (8.1)	129.7 (3.6)	30.9

[a]Values in parentheses are coupling constants J_{PC} in hertz.
[b]Recorded in $DCCl_3$.

TABLE 4 ^{13}C NMR Chemical Shifts[a] of 6-Aryloxy/arylthio Groups of **3** (δ)

Compound	C(1′)	C(2′)	C(3′)	C(4′)	C(5′)	C(6′)	C′(CH_3)
3a	149.8 (8.2)	120.3 (5.0)	130.1	126.2	130.1	120.3	—
3b	148.8 (8.3)	129.5 (4.7)	131.7	125.9	127.3	119.3	16.3
3c	152.1 (7.2)	117.1 (4.5)	139.3	126.7	131.5	120.7	21.5
3d	147.8 (7.5)	120.0 (4.9)	130.3	135.9	130.3	120.0 (4.8)	20.8
3e	148.8	128.2	139.1	126.4	127.6	117.7	12.5, 20.1
3f	148.5 (7.8)	129.1	132.0	135.7	127.5	119.7	16.1, 20.6
3g	148.7 (7.5)	129.1 (4.3)	130.2	125.6	130.2	129.1 (4.3)	17.4
3h	147.9 (7.2)	121.2 (5.1)	138.4	134.5	130.6	119.3	19.2, 19.9
3i	146.7 (7.2)	126.6	130.9	128.2	133.6	121.5	—
3j	148.3	121.7 (5.2)	130.1	131.9	130.1	121.7 (5.2)	—
3k	149.7	119.7 (4.4)	130.2	144.9	130.2	119.7 (4.4)	—
3l	149.3 (8.2)	119.5 (5.0)	127.1	147.7	127.1	119.5 (5.0)	35.4, 31.4
3m	130.8 (5.6)	129.6 (2.8)	129.9	126.5	129.9	129.6 (2.8)	—
3n	127.8	128.5 (3.0)	128.2	134.5	128.2	128.5 (3.0)	21.4

[a]Values in parentheses are coupling constants J_{CP} in hertz.

TABLE 5 Mass Spectral Data of Certain Members of **3**[a]

Compound	M^+	$(M\text{-}Cl)^+$	$(M\text{-}Cl_2)^+$	$(M\text{-}OR)^+$	$(M\text{-}RO_2P)^+$	$(M\text{-}C_{13}H_4O_3Cl_6P)^+$
3a	542 (2.4)	507 (17.3)	472 (14.5)	449 (8.0)	402 (6.3)	94 (100)
3c	556 (1.7)	521 (37.4)	486 (24.6)	449 (25.3)	402 (7.2)	108 (100)
3d	556 (5.3)	521 (33.7)	486 (15.2)	449 (37.8)	402 (4.3)	108 (100)
3e	570 (22.0)	535 (48.2)	500 (27.0)	449 (3.0)	402 (7.3)	122 (100)
3f	570 (1.2)	535 (5.1)	500 (2.0)	449 (4.5)	402 (1.0)	122 (100)
3h	570 (37.0)	535 (9.5)	500 (2.7)	449 (4.8)	402 (1.3)	122 (100)
3i	576 (41.0)	541 (1.1)	506 (10.2)	449 (2.0)	402 (30.0)	128 (100)
3j	576 (2.8)	541 (24.0)	506 (2.0)	449 (22.0)	402 (32.0)	128 (100)
3k	—	552 (2.3)	—	449 (2.5)	402 (7.0)	139 (42.5)

[a]Values in parentheses are relative intensities.

were recorded in δ values relative to TMS (^1H and ^{13}C) or 85% H_3PO_4 (^{31}P).

1,2,4,8,10,11-Hexachloro-6-(2',3'-dimethylphenoxy)-12H-dibenzo[d,g][1,3,2]-dioxaphosphocin 6-Oxide (**3e**)

A solution of 2,3-dimethylphenyl phosphorodichloridate (**2e**, 1.20 g, 0.005 mol) in dry toluene (15 mL) was added dropwise over a period of 15 minutes to a stirred solution of 2,2'-methylenebis(3,4,6-trichlorophenol) (**1**, 2.03 g, 0.005 mol) and triethylamine (1.01 g, 0.01 mol) in dry toluene (30 mL). After the completion of the addition, the mixture was heated to 50–60°C. TLC analysis of the reaction mixture on silica gel indicated the completion of the reaction after stirring for 6 hours. Removal of triethylamine hydrochloride by filtration and evaporation of the solvent from the filtrate produced a residue which, on washing with water and recrystallization from ethylacetate-hexane, yielded 1.9 g (66%) of **3e** as white crystals, mp 180°C. Anal. calcd for $C_{21}H_{13}O_4Cl_6P$ (573.023): C, 44.02; H, 2.29. Found: C, 43.96; H, 2.21.

This procedure was also used for the preparation of **3a–3k**.

1,2,4,8,10,11-Hexachloro-6-(4'-methylthiophenoxy)-12H-dibenzo[d,g]-[1,3,2]dioxaphosphocin 6-oxide (**3n**)

Phosphorus oxychloride (0.76 g, 0.005 mol) in dry toluene (20 mL) was added over a period of 10 minutes to a stirred solution of 2,2'-methylenebis(3,4,6-trichlorophenol) (**1**, 2.03 g, 0.005 mol) and triethylamine (1.01 g, 0.01 mol) in dry toluene (30 mL) at 0–5°C. After the addition, the temperature was slowly raised to 45–55°C with stirring being continued for 3 hours. When the monochloride intermediate (**4**) formation was completed, as indicated by TLC analysis on silica gel, the mixture was cooled to room temperature and a solution of 4-methylthiophenol (**5n**, 0.062 g, 0.005 mol) and triethylamine (0.51 g, 0.005 mol) in dry toluene (10 mL) was added to the same vessel. The total contents were stirred at 50–60°C for an additional 3 hours. Triethylamine hydrochloride was separated by filtration and the solvent was removed from the filtrate in a rotoevaporator under reduced pressure. The residue was washed with water and recrystallized from ethyl acetate-hexane mixture (1:2) to yield 1.2 g (41%) of **3n** as a white powder, mp 147–148°C. Anal. calcd for $C_{20}H_{11}O_3Cl_6PS$ (570.057): C, 41.77; H, 1.93. Found: C, 41.71; H, 1.87.

The preceding procedure was also used for the preparation of 3l-3o.

ACKNOWLEDGMENTS

BSR gratefully acknowledges the financial assistance (JRF) from the University Grants Commission, New Delhi, India. Prof. K. D. Berlin, Oklahoma State University, is gratefully acknowledged for recording the NMR spectra.

REFERENCES

[1] C. Fest, K.-J. Schmidt: *The Chemistry of Organophosphorus Pesticides*, Springer-Verlag, Berlin, 1982.
[2] D. L. Hill: *A Review of Cyclophosphamide*, Charless C. Thomas, Springfield, IL, 1975.
[3] J. D. Spivack: Brit. Patent 2,087,399 (1982), *Chem. Abstr., 97*, 198374 (1982).
[4] E. Billig, A. G. Abatjoglou, D. R. Bryant, R. E. Murray, J. M. Mather: U.S. Patent 4,599,206 (1986), *Chem. Abstr., 105*, 81142 (1986).
[5] (a) C. D. Reddy, R. S. Reddy, C. N. Raju, M. ElMasri, K. D. Berlin, S. Subramanian, *Magn. Reson. Chem., 29*, 1140 (1991); (b) C. D. Reddy, K. D. Berlin, R. S. Reddy, C. N. Raju, M. ElMasri, S. Subramanian, *Phosphorus, Sulfur Silicon, 81*, 61 (1993); (c) C. D. Reddy, R. S. Reddy, M. S. Reddy, M. Krishnaiah, K. D. Berlin, P. Sunthankar, *Phosphorus, Sulfur Silicon, 62*, 1 (1991).
[6] L. C. Thomas, R. A. Chittenden, *Chem. Soc. (London)*, 1913 (1961).
[7] L. C. Thomas, R. A. Chittenden, *Spectro. Chim. Acta, 20*, 489 (1964).
[8] R. A. Nyquist, *Spectro. Chim. Acta, 19*, 713 (1963).
[9] (a) R. A. McIvor, G. A. Grant, C. E. Hubley, *Can. J. Chem., 34*, 1611 (1956); (b) R. A. McIvor, C. E. Hubley, G. A. Grant, A. A. Grey, *Can. J. Chem., 36*, 820 (1958).
[10] (a) S. D. Pastor, J. D. Spivack, *J. Heterocyclic Chem., 20*, 1311 (1983); (b) S. D. Pastor, J. D. Spivack, L. P. Steinhuebel, C. Matzura, *Phosphorus Sulfur, 15*, 253 (1983); (c) P. A. Odorisio, S. D. Pastor, J. D. Spivack, L. Steinhuebel, R. K. Rodebaugh, *Phosphorus Sulfur 15*, 9 (1983); (d) P. A. Odorisio, S. D. Pastor, J. D. Spivack, *Phosphorus Sulfur, 19*, 285 (1984); (e) P. A. Odorisio, S. D. Pastor, J. D. Spivack, *Phosphorus Sulfur, 19*, 1 (1984); (f) S. D. Pastor, *Sulfur Lett., 9*, 39 (1989).
[11] S. D. Pastor, J. D. Spivack, *J. Heterocyclic Chem., 28*, 1561 (1991).
[12] J. D. Goddard, A. W. Payne, M. Cook, *J. Heterocyclic Chem., 25*, 575 (1988).
[13] R. P. Arshinova, *Phosphorus, Sulfur Silicon, 68*, 155 (1992).
[14] G. W. Buchanan, R. H. Whitman, M. Malaiyandi, *Org. Magn. Reson., 19*, 98 (1982).
[15] G. C. Levy, J. D. Cargioli, *J. Chem. Soc., Chem. Commun.*, 1663 (1970).
[16] R. M. Silverstein, G. C. Bassler, T. C. Morrill: *Spectrometric Identification of Organic Compounds*, 5th ed., John Wiley and Sons, New York, 1991.