Bulgarian Chemical Communications, Volume 41, Number 3 (pp. 248–252) 2009

Synthesis of phosphorus, nitrogen, oxygen and sulphur macrocycles

M. Kasthuraiah, M. V. N. Reddy, A. U. R. Sankar, B. S. Kumar, C. S. Reddy*

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

Received August 10, 2008; Revised January 19, 2009

Phosphorus macromolecules containing oxygen, nitrogen and sulphur were synthesised by the addition of phosphoric acid diallyl esters to 1,2-ethandithiol or various amines in dry dichloromethane. All compounds were characterised by IR, NMR (^{1}H, ^{13}C and ^{31}P) and mass spectral studies and elemental analysis. Their antimicrobial activity has also been evaluated.

Key words: allylic alchol, macrocycles, phosphorodichloridates, antimicrobial activity.

INTRODUCTION

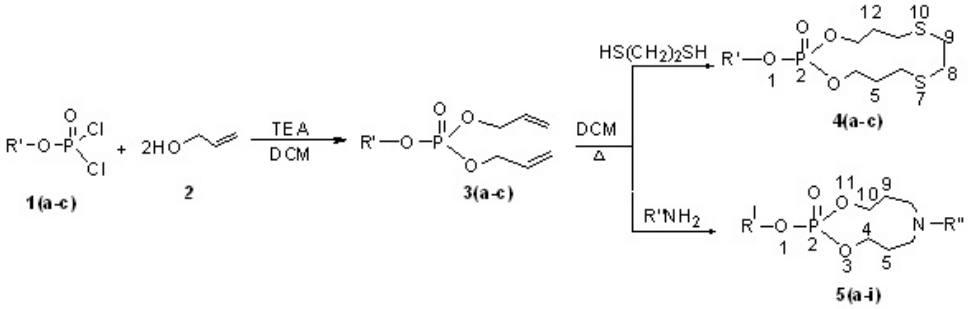
Phosphorus-containing macrocycles are interesting molecules with potential application in supramolecular and synthetic organic chemistry [1]. They have been synthesised as phosphine oxides, phosphines, phosphonium salts, phosphates, phosphonates and phosphoranes [2]. The importance of these molecules, as phosphorous analogues of crown ethers, is their potential catalytic activity and ion-carrier properties. The design and synthesis of host molecules capable of binding neutral organic molecules as guests is an area of rapidly expanding interest [3]. Cram [4], Lehn [5], Vogtle [6], Diederich [7] and others have made significant advances in the field of host-guest complexation [8]. Some of our past and present research has led to the construction of large preorganised macrocyclic cavities bearing concave functionalities [9]. They are also expected to function as good 'Hosts' in the 'Host-guest chemistry'. This particular property enables them to carry the drug molecule to the required cite in the living system, thus foreseeing great future for them in pharmaceutical industry. More recently Pietrusiewicz *et al.* have presented the synthesis of macrocyclic systems containing phosphorus and sulphur-based on a double conjugate addition of dithiolates to vinyl phosphane oxides and sulphides as Michael acceptors [10]. Nitrogen and oxygen mixed donor macrocycles can form stable complexes with alkali and transition metal ions. Therefore, mixed donor macrocycles have received much attention as receptors for a range of metal ions and other cations [11−14]. This particular property enables their use as efficient reagents to trap heavy toxic metals in polluted water. In view of their possible applications and novelty in the chemistry of Host-guest molecular ensembles, it is thought worth to synthesise and study the properties of several phosphorus macro-cycles with nitrogen, oxygen and sulphur as donor atoms.

RESULTS AND DISCUSSION

The synthesis Scheme 1 involves the condensation of allylic alchol (**2**) with various aryl phosphorodichloridates (**1a**–**c**) at 0°C under inert, anhydrous conditions in dry dichloromethane to afford the corresponding phosphoric acid diallyl esters (**3a**–**c**). Their subsequent addition to 1,2-ethanedithiol or various amines in dry dichloromethane at refluxed conditions leads to the final products, which were purified by column chromatography using hexane: ethylacetate step gradient mixtures as eluents.

All the compounds (**4a**–**c**, **5a**–**i**) exhibited IR stretching frequencies for P=O, P–O–($C_{(aromatic)}$), (P)–O–C_{arom} in the region of 1260−1291, 926−939 and 1202−1228 cm^{-1}, respectively [15−21] (Table 1). Their ^{1}H NMR spectra gave signals (Table 2) for all aromatic protons at δ 6.96−7.69 as complex multiplets [22−24]. The methylene groups (H-4 and 13) directly attached to oxygen in **4a**–**c** resonated as triplets at δ 4.24−4.26 (J = 5.0−6.0 Hz). Another two triplets in the region of 2.82−2.85 ppm and 2.62−2.63 ppm are attributed to H-6 and 11 and H-5 and 9, respectively. Multiplets in the region of 1.93−1.96 ppm are assigned to H-5 and 12. Similarly, the endocyclic six methylene protons of dioxaphosphocin system in **5a**–**i** exhibited two triplets for H-4 and 10 and multiplets for H-5 and 9 in the expected regions. All carbons in the compounds (**4a**–**b**, **5a**, **5d**, **5h** and **5i**) exhibited signals at their expected values (Table 3). Carbons 8 and 9 in compounds **4a** and **4b**, exhibited one singlet.

* To whom all correspondence should be sent:
E-mail: csrsvu@gmail.com



Com-pound	R'	Com-pound	R'	R"	Com-pound	R'	R"	Com-pound	R'	R"
1a & **4a**	C_6H_5	**5a**	C_6H_5	$CH_2CH_2CH_3$	**5d**	2-$CH_3C_6H_4$	$CH_2CH_2CH_3$	**5g**	4-$CH_3C_6H_4$	$CH_2CH_2CH_3$
1b & **4b**	2-$CH_3C_6H_5$	**5b**	C_6H_5	$CH_2(CH_2)_2CH_3$	**5e**	2-$CH_3C_6H_4$	$CH_2(CH_2)_2CH_3$	**5h**	4-$CH_3C_6H_4$	$CH_2(CH_2)_2CH_3$
1c & **4c**	4-$CH_3C_6H_5$	**5c**	C_6H_5	$CH_2C_6H_5$	**5f**	2-$CH_3C_6H_4$	$CH_2C_6H_5$	**5i**	4-$CH_3C_6H_4$	$CH_2C_6H_5$

Scheme 1.

Table 1. Analytical and spectral data of **4a**–**c** and **5a**–**i**.

Compound	M.p., °C	Yield, %	Molecular formula	Elemental analysis, % Calculated (Found)		P–O–C, cm^{-1}			^{31}P NMR (85% H_3PO_4)
				C	H	P=O	P–O	O–C	
4a	77−79	86	$C_{14}H_{21}O_4S_2P$	48.13 (48.26)	6.01 (6.07)	1270	928	1212	−3.29
4b	99−101	89	$C_{15}H_{23}O_4S_2P$	49.60 (49.71)	6.21 (6.39)	1260	926	1210	−4.73
4c	85−87	87	$C_{15}H_{23}O_4S_2P$	49.56 (49.71)	6.26 (6.39)	1291	939	1202	−2.2
5a	48−50	80	$C_{16}H_{26}O_4NP$	58.58 (58.70)	7.91 (8.00)	1282	930	1216	−2.2
5b	69−71	79	$C_{17}H_{28}O_4NP$	59.70 (59.81)	8.11 (8.26)	1273	931	1228	−4.5
5c	110−111	80	$C_{20}H_{26}O_4NP$	63.79 (63.99)	6.81 (6.98)	1289	929	1213	−3.3
5d	63−65	83	$C_{16}H_{26}O_4NP$	58.60 (58.70)	8.00 (8.005)	1261	931	1221	−3.7
5e	69−71	79	$C_{17}H_{28}O_4NP$	59.70 (59.81)	8.11 (8.26)	1276	928	1228	−4.5
5f	77−78	78	$C_{20}H_{26}O_4NP$	63.80 (63.99)	6.968 (6.979)	1273	931	1215	−4.3
5g	72−74	76	$C_{16}H_{26}O_4NP$	58.58 (58.70)	8.09 (8.01)	1269	936	1219	−3.8
5h	95−96	86	$C_{17}H_{28}O_4NP$	59.75 (59.81)	8.16 (8.26)	1278	924	1227	−3.2
5i	110−112	88	$C_{20}H_{26}O_4NP$	63.79 (63.94)	6.81 (6.98)	1289	929	1213	−3.3

The signals for C-4 and 10, C-6 and 8, and C-5 and 9 in **5a**–**b** appeared as doublets at their corresponding values. The oxygen bearing C-4 and 13, C-4 and 10 in **4a**–**b** and **5a**, **5d**, **5h**, **5i** experienced coupling with phosphorous and exhibited doublets. All the compounds except **4a**, **4b** exhibited two ^{31}P NMR chemical shift values because of their presence as two conformers in solution state.

EXPERIMENTAL

Melting points were determined in open capillary tubes on a Mel-Temp. apparatus and were not corrected. IR spectra (ν_{max} in cm^{-1}) were recorded in KBr pellets on a Perkin-Elmer 1000 unit. The ^{1}H, ^{13}C and ^{31}P NMR spectra were recorded on various Gemini 300 and Varian AMX 400 MHz NMR spectrometers operating at 300 or 400 MHz for ^{1}H, 75.46 or 100.57 MHz for ^{13}C and 121.7 MHz for ^{31}P. All the compounds were dissolved in $CDCl_3$ and chemical shifts was referred to those of TMS (^{1}H and ^{13}C) and 85% H_3PO_4 (^{31}P). Microanalytical data were obtained from the Central Drugs Research Institute, Lucknow, India.

Table 2. ^{1}H NMR spectral data of **4a–c** and **5a–i.**

Compound	Chemical shifts, ppm
4a	6.98−7.26 (m, 5H, Ar-H), 4.26 (t, *J* = 5.1 Hz, 4 H, H-4 & 13), 2.85, (t, *J* = 10.8 Hz, H-6 & 11), 2.63 (t, *J* = 10.8 Hz, H-8 & 9), 1.96 (m, 4H, H-5 & 12)
4b	7.07−7.29 (m, 4H, Ar-H), 4.25 (t, *J* = 5.0 Hz, 4H, H-4 & 13), 2.82 (t, *J* = 10.6 Hz, 4H, H-6 & 11), 2.63 (t, *J* = 10.3 Hz, 4H, H-8 & 9), 1.95 (m, 4H, H-5 & 12), 2.31 (s, 3H, H-7')
4c	7.06−7.28 (m, 4H, Ar-H), 4.24 (t, *J* = 6.0 Hz, 4H, H-4 & 13), 2.84 (t, *J* = 10.8 Hz, H-6 & 11), 2.62 (t, *J* = 9.6 Hz, 4H, H-8 & 9), 1.93 (m, 4H, H-5 & 12), 2.30 (s, 3H, H-7')
5a	7.06−7.28 (m, 5H, Ar-H), 4.23 (t, *J* = 5.3 Hz, 4H, H-4 & 10), 2.81−2.86 (t, *J* = 8.9 Hz, 4H, H-6 & 8), 2.69−2.74 (m, 2H, H-1″), 1.68−1.73 (m, 4H, H-5 & 9), 1.26−1.31 (t, 2H, H-2″), 0.88−0.92 (m, 3H, H-3″)
5b	7.03−7.24 (m, 5H, Ar-H), 4.21−4.27 (t, *J* = 5.1 Hz, 4H, H-4 & 10), 2.85−2.92 (t, *J* = 8.8 Hz, 4H, H-6 & 8), 2.66−2.72 (m, 2H, H-1″), 1.70−1.74 (m, 4H, H-5 & 9), 1.42−1.46 (m, 2H, H-2″), 1.25−1.31 (m, 2H, H-3″), 0.87−0.94 (m, 3H, H-4″)
5c	6.96−7.33 (m, 10H, Ar-H), 4.19−4.25 (t, *J* = 4.9 Hz, 4H, H-4 & 10), 3.82 (d, *J* = 16.2 Hz, 2H, H-1″), 2.73−2.76 (t, *J* = 9.2 Hz, 4H, H-6 & 8), 1.76−1.81 (m, 4H, H-5 & 9)
5d	7.11−7.42 (m, 4H, Ar-H), 4.41−4.44 (t, *J* = 5.4 Hz, 4H, H-4 & 10), 2.84−2.90 (t, *J* = 9.1 Hz, 4H, C-6 & 8), 2.65−2.71 (m, 2H, H-1″), 2.3 (s, 3H, Ar-CH_3), 1.77−1.82 (m, 4H, H-5 & 9), 1.36−1.39 (m, 2H, H-2″), 0.81−0.87 (m, 3H, H-3″)
5e	7.09−7.52 (m, 4H, Ar-H), 4.28−4.34 (t, *J* = 4.84 Hz, H, H-4 & 10), 2.82−2.87 (t, *J* = 9.4 Hz, 4H, H-6 & 8), 2.59−2.63 (m, 2H, H-1″), 2.36 (s, 3H, Ar-CH_3), 1.72−1.79 (m, 4H, H-5 & 9), 1.42−1.47 (m, 2H, H-2″), 1.21−1.26 (m, 2H, H-3″), 0.81−0.85 (m, 2H, H-4″)
5f	7.12−7.69 (m, 9H, Ar-H), 4.42−4.49 (t, *J* = 5.1 Hz, 4H, H-4 & 10), 3.74 (d, *J* = 16.7 Hz, 2H, H-1″), 2.71−2.78 (m, 4H, H-6 & 8), 2.29 (s, 3H, Ar-CH_3), 1.69−1.72 (m, 4H, H-5 & 9)
5g	6.92−7.34 (m, 4H, Ar-H), 4.11−4.16 (t, *J* = 5.0 Hz, 4H, H-4 & 10), 2.76−2.81 (m, 4H, H-6 & 8), 2.63−2.68 (m, 2H, H-1″), 2.27 (s, 3H, Ar-CH_3), 1.74−1.79 (m, 4H, H-5 & 9), 1.21−1.26 (m, 2H, H-2″), 0.91−0.96 (m, 3H, H-3″)
5h	6.97−7.28 (m, 4H, Ar-H), 4.21−4.27 (t, *J* = 4.9 Hz, 4H, H-4 & 10), 2.91−2.94 (m, 4H, H-6 & 8), 2.72−2.76 (m, 2H, H-1″), 2.32 (s, 3H, Ar-CH_3), 1.69−1.74 (m, 4H, H-5 & 9), 1.41−1.45 (m, 2H, H-2″), 1.11−1.16 (m, 2H, H-3″), 0.90−0.94 (m, 3H, H-4″)
5i	7.04−7.36 (m, 9H, Ar-H), 4.280−4.33 (t, *J* = 5.1 Hz, 4H, H-4 & 10), 3.76 (d, *J* = 16.9 Hz, 2H, H-1″), 2.67−2.71 (t, *J* = 9.3 Hz, 4H, H-6 & 8), 2.29 (s, 3H, Ar-CH_3), 1.68−1.71 (m, 4H, H-5 & 9)

[a] Chemical shifts in ppm from TMS and coupling constants *J* (Hz) given in parenthesis; [b] Recorded in deuterochloroform.

Synthesis of 2-o-tolyloxy-1,3-dioxa-7,10-dithio-2-phosphacyclotridecane-2-oxide (**3b**)

A solution of 2-methylphenylphosphorodichloridate (**1b**, 2.25 g, 0.01 mole) dissolved in 20 ml of dry dichloromethane (DCM) was added over a period of 20 minutes at 0°C to a stirred solution of allyl alcohol (1.24 g, 0.02 mole) and triethylamine (2.02 g, 0.021 mole) in 30 ml of dry DCM. After completion of addition, the temperature of the reaction mixture was raised to 45−50°C and kept for two hours with stirring. Progress of the reaction was monitored by TLC analysis, the precipitated triethylamine hydrochloride was separated by filtration and the filtrate was vacuum evaporated. The crude product obtained was dissolved in DCM and 1,2-ethanedithiol (1.4 g, 0.015 mole) in 25 ml of dry DCM was added dropwise with stirring. The mixture was refluxed over a period of two hours to ensure the completion of the reaction. The resulting syrupy liquid was purified by column chromatography (ethyl acetate-hexane 0:100 to 25:75) to afford 1.25 g (89%) of **3b** as a semi-solid. Analogous were prepared by adopting the above procedure.

Table 3. ^{13}C NMR spectral data of **4a-b**, **5a**, **5d**, **5h** and **5i**.

Compound	Chemical shifts, ppm
4a	67.9 (d, *J* = 5.2 Hz, 1C, C-4), 66.9 (s, 1C, C-13), 38.6 (s, 1C, C-6), 36.9 (s, 1C, C-11), 31.3 (s, 1C, C-5), 30.8 (s, 1C, C-12), 25.6 (s, 2C, C-8 & 9), 148.4 (s, 1C,C-1'), 120.0 (s, 2C, C-2' & 6'), 132.8 (s, 2C, C-3' & 5'), 123.9 (s, 1C, C-4')
4b	68.66 (d, *J* = 5.1 Hz, 1C, C-4), 66.69 (s, 1C, C-13), 38.2 (s, 1C, C-6), 37.4 (s, 1C, C-11), 31.3 (s, 1C, C-5), 30.1 (s, 1C, C-12), 24.5 (s, 2C, C-8 & 9), 148.9 (s, 1C, C-1'), 129.1 (s, 1C, C-2'), 131.2 (s, 1C, C-3'), 126.9 (s, 1C, C-4'), 127.3 (s, 1C, C-5'), 119.6 (s, 1C, C-6'), 16.3 (s, 1C, C-2', $\underline{C}H_3$)
5a	149.1 (s, 1C, C-1′), 134.3 (s, 1C, C-4′), 132.2 (s, 2C, C-3′ & 5′), 119.5 (d, *J* = 6.0 Hz, 2C, C-2′ & 6′), 66.61 (d, *J* = 5.8 Hz, 2C, C-4 & 10), 46.8 (d, *J* = 4, 2C, C-6 and 8), 46.1 (d, *J* = 21.7 Hz, C-1′′), 31.2 (s, 2C, C-5 & 9), 36.8 (s, 1C, C-2″), 13.9 (s, 1C, C-3″)
5d	66.63 (d, *J* = 5.8 Hz, 2C, C-4 & 10), 46.2 (d, *J* = 4.1, Hz 2C, C-6 & 8), 46.1 (d, *J* = 21.5 Hz, C-1′′), 37.1 (s, 1C, C-2″), 13.02 (m, 1C, C-3″), 149.8 (s, 1C, C-1′), 133.9 (s, 1C, C-4′), 132.1 (s, 2C, C-3′ & 5′), 118.9 (d, *J* = 6.1 Hz, 2C, C-2′ & 6′), 20.7 (s, 1C, C-2′, $\underline{C}H_3$)
5h	66.72 (d, *J* = 6.1 Hz, 2C, C-4 & 10), 46.1 (d, *J* = 4.1, 2C, C-6 & 8), 32.8 (s, 2C, C-5 & 9), 47.2 (d, *J* = 27.8 Hz, 1C, C-1′′), 37.9 (s, 1C, C-2″), 21.2−21.7 (m, 1C, C-3′′), 13.6−13.9 (m, 1C, C-4′′), 150.9 (d, *J* = 7.3 Hz, 1C, C-1′), 133.8 (s, 1C, C-4′), 131.9 (s, 2C, C-3′ & 5′), 119.7 (d, *J* = 4.6 Hz, 2C, C-2′ & 6′), 22.9 (s, 1C, C-4′)
5i	66.63 (d, *J* = 6.9 Hz, 2C, C-4 & 10), 45.7 (d, *J* = 5.9 Hz, 2C, C-6 & 8), 31.3 (s, 2C, C-5 & 9), 47.2 (d, *J* = 17.8 Hz, 1C, C-1′′), 148.7 (s, 1C, C-1′), 143.7 (s, 1C, C-2′′), 135.7 (s, 1C, C-4′), 133.1 (s, 1C, C-3′ & 5′), 128.7 (s, 2C, C-4′′ & 6′′), 126.9 (s, 2C, C-3′′ & 7′′), 126.1 (s, 1C, C-5′′), 22.9 (s, 1C, C-4′, CH_3)

[a] Chemical shifts in ppm from TMS and coupling constants *J* (Hz) given in parenthesis; [b] Recorded in deuterochloroform.

Table 4. Mass spectral data of **4b**, **5a** and **5i**.

Com-pound	m/z, %
4b	363[M^+1] (24), 267 (31), 186 (24), 162 (19), 150 (26)
5a	313[M^+] (30), 289 (60), 270 (56), 188 (24), 141 (20)
5i	375[M^+] (60), 285 (32), 281 (40), 236 (80), 190 (28), 186 (29)

ANTIMICROBIAL ACTIVITY

Compounds **4a–c** and **5a–i** were screened for their antimicrobial activity against the growth of bacteria *Staphylococcus aureus* (gram +Ve) and *Escherichia coli* (gram −Ve) and fungi *Aspergillus niger* and *Helminthosporium oryzae* at concentrations [25, 26] 20 μg/disc and 400. μg/disc. They have exhibited moderate antibacterial and moderate antifungal activity when compared to the standard reference compounds (Table 5, 6).

Table 5. Antibacterial activity of **4a–c** and **5a–i**.

Compound	Zone of inhibition, mm			
	Staphylococcus aureus		*Escherichia coli*	
	200 [a] μg/disc	400 [a] μg/disc	200 [a] μg/disc	400 [a] μg/disc
4a	14	19	13	20
4b	13	18	11	15
4c	18	16	15	15
5a	16	14	16	12
5b	17	12	18	14
5c	17	15	17	13
5d	12	18	14	13
5e	13	19	18	12
5f	16	17	16	11
5g	13	18	14	17
5h	15	17	16	18
5i	14	20	17	18
Penicillin [b]	22		21	

[a] Concentration in ppm; [b] Standard reference compound.

Table 6. Antifungal activity of **4a–c** and **5a–i.**

Compound	Zone of inhibition, mm			
	Aspergillus niger		*Helminthosporium oryzae*	
	200 [a] μg/disc	400 [a] μg/disc	200 [a] μg/disc	400 [a] μg/disc
4a	16	22	18	23
4b	14	23	16	22
4c	13	21	14	20
5a	18	24	17	23
5b	17	21	16	20
5c	18	23	17	21
5d	11	19	12	21
5e	13	22	13	21
5f	12	20	11	19
5g	18	23	15	22
5h	17	22	16	24
5i	18	25	16	23
Griseofulvin [b]	28		28	

[a] Concentration in ppm; [b] Standard reference compound.

Acknowledgements: *The authors express their thanks to Prof. C. Devendranath Reddy for his helpful guidance and discussions and the Director of CDRI, Lucknow and SIF, IISC, Bangalore for the analytical and spectral data.*

REFERENCES

1. A. M. Caminade, J. P. Majoral, *Chem. Rev*., **94**, 1183 (1994).
2. J. P. Dutasta, P. Simon, *Tetrahedron Lett.,* **28,** 3577 (1987).
3. For an excellent review see: F. Diederich, *Angew. Chem. Int. Ed., Eng*., **27**, 362 (1988).
4. A. J. Tucker, C. B. Knobler, K. N. Trueblood, J. D. Cram, *J. Am. Chem. Soc.,* **111***,* 3688 (1989)**.**
5. F. Fages, J. P. Desvergue, F. Kotzbahibert, J. M. Lehn, P. Marson, A. M. Albrechtgary, H. Bouaslarent, M. Aljoubloeh, *J. Am. Chem. Soc.,* **111**, 8672 (1989).
6. A. Wallar, J. Peter-Katinic, W. M. Werher, F. Vogtle, *Chem. Ber*., **123**, 375 (1990).
7. E. M. Seward, R. B. Hopkins, W. Sauerer, S. W. Tam, F. Diederich, *J. Am. Chem. Soc*., **112,** 1783 (1990).
8. R. Brelow, N. Greenspoon, T. Guo, R. Zarzycki, *J. Am. Chem. Soc*., **111**, 8296 (1989).
9. R. E. Sheridan, H. W. Whitlock,. *J. Am. Chem. Soc*., **110,** 407 (1988).
10. K. Zygo, W. Wieczorek, K. M. Pietrusiemicz, 14th Int. Conf. Phosphorous Chemistry, Cincinnati, 1998.
11. Y. Inous Gokel, G. W. Cation, Binding by Macrocycles*,* Marcel Dekker, New York, 1991.
12. J. S. Bradshaw, R. M. Lzatt, *Acc. Chem.* Res., **30**, 388 (1997).
13. B. Zhao, F. Q. Wang, L. J. Tian, *J. Heterocycl. Chem*., **38**, 781(2001).
14. S. Kumar, S.H. Mashnaqui, *Indian J. Chem*., **36B**, 656 (1997).
15. L. W. Daash, S. C. Smith, *Anal. Chem*., **23**, 853 (1961).
16. L. C. Thomas, R. A. Chittenden, *Spectrochim. Acta*, **20**, 489 (1964).
17. L. C. Thomas, Interpretation of the Infrared Spectra of Organophosphorous Compounds, Heydon, London, 1974.
18. D. E. C. Corbridge, *Top. Phosphorus Chem*., **6**, 235 (1969).
19. L. J. Bellamy, L. J. Beecher, *J. Chem. Soc*., 728. (1953).
20. R. A. Nyquist, *Appl. Spectrosc.*, **11,** 161 (1957).
21. M. M. Crutchfield, C. H. Dungan, J. H. Letcher, V. Mark, J. R. Van Wazer, ^{31}P Nuclear Magnetic Resonance, Interscience Publisher, New York, 1967.
22. R. M. Silverstein, G. C. Bassler, T. C. Morril, Spectrometric Identification of Organic Compounds, John Wiley & Sons, New York, 1991.
23. C. D. Reddy, R. .S. Reddy, C. N. Raju, M. Elmasri, K. D. Berlin, S. Subramanian, *Magn. Reson. Chem.,* **29**, 01 (1991).
24. E. Pretsch, J. Selible, W. Simon, T. Clerc, Tables of Spectral Data of Structure Determination of Organic Compounds, Springer-Verlag, Berlin, 1981.

25. J. C. Vincent, H. W. Vincent, *Proc. Soc. Exp. Biol., Med.*, **55**,162 (1944).
26. H. J. Benson, Microbiological Applications, 5th ed., W. C. Brown Publications, Boston, 1990.

СИНТЕЗА НА МАКРОЦИКЛИ СЪДЪРЖАЩИ ФОСФОР, АЗОТ, КИСЛОРОД И СЯРА

М. Кастурайа, М. В. Н. Реди, А. Ю. Р. Санкар, Б. С. Кумар, С. С. Реди*

Департамент по химия, Университет Сри Венкатесуара, Тирупати 517 502, Индия

Постъпила на 10 август 2008 г.; Преработена на 19 януари 2009 г.

(Резюме)

Синтезирани са макромолекули съдържащи фосфор, кислород, азот и сяра чрез реакция на диалилови естери на фосфорна киселина с 1,2-етандитиол или различни амини в сух дихлорометан. Всички съединения са охарактеризирани с ИЧС, ^{1}H, ^{13}C и ^{31}P ЯМР, масспектрални изследвания и елементен анализ. Оценена е също и тяхната антимикробна активност.