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Note

Synthesis and spectral characterization of cyclotriphosphazene based 18-membered macrocycles

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ABSTRACT

New 18-membered cyclotriphosphazene-containing macrocycles **7-10** were obtained by 1 + 1 condensation reaction of [*dispiro*-$N_3P_3(C_{12}H_8O_2)_2((N(Me)N{=}CH)_2\ N_4C_{20}H_{26})$] (**2**) with *N*,*N*′-dimethyl-ethylenediamine-1,4-diyldimethylenebis(4-methyl-2-formylphenol) (**3**), *N*,*N*′-dimethyl-ethylenediamine-1,4-diyldimethylenebis(4,5-dimethyl-2-formylphenol) (**4**), *N*,*N*′-dimethyl-ethylenediamine-1,4-diyldimethylenebis(5-chloro-2-formylphenol) (**5**) and *N*,*N*′-dimethyl-ethylenediamine-1,4-diyldimethylenebis(5-bromo-2-formylphenol) (**6**), respectively.

Cyclophosphazenes are an important family of inorganic heterocyclic rings with large variation of ring size [1]. The cyclotriphosphazene $N_3P_3Cl_6$ has been extensively investigated particularly in terms of (i) nucleophilic substitution reactions at phosphorus [1–5], (ii) ring-opening polymerization [1–5], and (iii) utility as a support for building multisite coordination ligands [1–5]. Several groups have also shown that in addition to the traditional themes of investigation as outlined above [1–5]. Cyclophosphazenes can also be utilized for supporting chiral ligands, multi-electroactive, photoactive molecules and for studying supramolecular interactions [5,6]. Very interesting work was published by Elias et al. and others in recent years [6c]. On the other hand the utility of the cyclophosphazene ring as a support for constructing macrocyclic ligands is only slowly being realized [7a,8]. An important step in this direction is the assembly of several new crown ethers supported on the phosphazene skeleton. It has been observed that the reaction of $N_3P_3Cl_6$ with long chain diols such as tetraethylene glycol or pentaethylene glycol affords a mixture of products where the substitution of the glycol has occurred at the same phosphorus (spirocyclic) or at two different phosphorus atoms (ansa) within the cyclophosphazene ring. A decade ago Majoral and co-workers have demonstrated that the reaction of acyclic phosphodihydrazides with various dialdehydes led to a great variety of macrocycles arising from [1 + 1], [2 + 2], [3 + 3], or even [4 + 4] cyclocondensations [7a,9]. Recently A similar strategy was suggested by Chandrasekhar and co-workers [10]. However, the studies carried out on cyclophosphazene based macrocycles (apart from cyclophosphazene appended crown ethers) till now have been sporadic with limited success. In this letter we report details of the condensation reactions of dialdehyde and hydrozene-derived cyclotriphosphazene and spectral characterization of the new 18-membered cyclophosphazene-containing macrocycles are described.

The parent cyclotriphosphazene ($N_3P_3Cl_6$) contains six reactive P–Cl bonds and is not possible for controlled macrocyclic synthesis [10]. Consequently, compound **1** ($N_3P_3Cl_2(O_2C_{12}H_8)_2$ was synthesized from $N_3P_3Cl_6$ by adopting a known synthetic procedure [8,10]. The reaction of **1** with MeNH–NH_2 in chloroform afforded **2** [5a]. This reaction is regiospecific; the *N*-methyl end of the bifunctional reagent reacts with **1** leading to product containing reactive terminal –NH_2 groups. In this reaction MeNH–NH_2 was also used to scavenge the liberated hydrogen chloride, obviating the need for an additional base. Compound **2** possesses two reactive NH_2 groups which could be readily elaborated under mild reaction conditions. Condensation of the hydrazide **2** with stoichiometric amount *N*,*N*′-ethylenediamine-1,4-diyldimethylenebis(4-methyl-2-formylphenol) (Scheme 1) occurs at room temperature to afford the corresponding hydrazones **7** in quantitative yield.

The notable feature of this reaction is the complete absence of 2+2 condensed product (Scheme 2) and the isolation of the product **7** (1 + 1 condensed) in excellent yield. Additionally, **7** is also structurally interesting because it is a small macrocycle with more number of strong donors like phenolate oxygen and nitrogens which can hold the incoming metal atoms very strongly. All the compounds were examined by multinuclear NMR spectroscopy. The $^{31}P\{^1H\}$ NMR spectrum of compound **2** is of the AX2 type.

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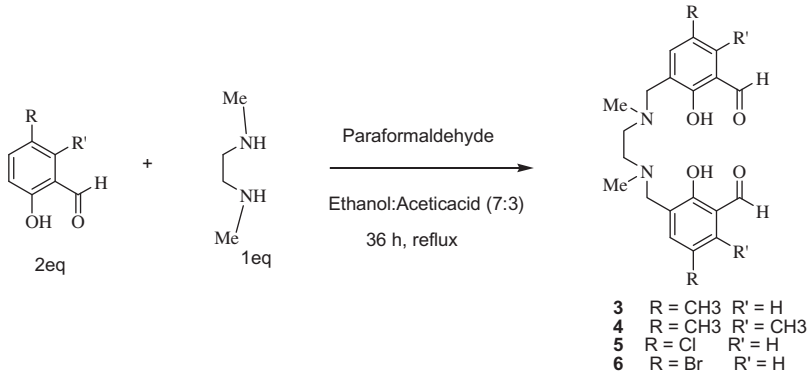
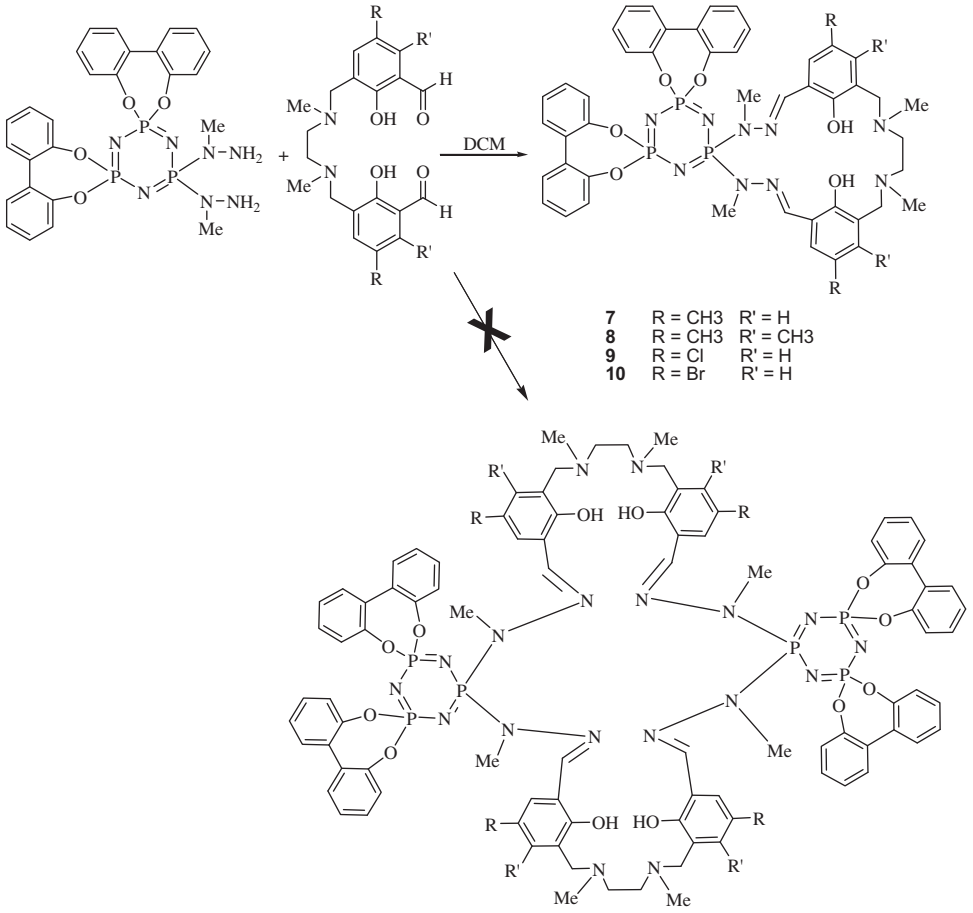


Scheme 1. Synthesis of compounds **3**–**6**.



Scheme 2. Synthesis of macrocycles **7**–**10**.

The spirocyclic phosphorus atoms $P(O_2C_{12}H_8)$ resonate at 26.2 ppm (doublet) while $P(N(Me)-)_2$ was seen at 29 ppm (triplet). The ^{31}P NMR spectrum of **7** revealed the presence of two distinct signals: δ 24.4 (d, $-P(-O_2C_{12}H_8)_2$), 17.7 (t, $-P$ (O_4 $N_4C_{20}H_{26}(CH{=})_2$)), ^2J(P–N–P) = 98.6 Hz). It is of interest to note that the chemical shift of the latter is upfield with respect to the free hydrazide **2**. Compound **7** also is characterized by the presence of a $[M+2]^+$ ion at 943 in its FAB mass spectrum (Fig. 1). Compounds **8**–**10** were prepared following a procedure similar to that used for **7**. Compounds **8**–**10** were characterized by multinuclear NMR (^1H, ^{13}C and ^{31}P), and elemental analysis. ^1H NMR integration, ^{31}P resonance and molecular ion peak in ESI Mass spectrum confirms the formation of **8**–**10** and are comparable with **7**.

In conclusion we report a facile procedure for the assembly of large macrocycles containing cyclophosphazene rings. The phenol oxygen and imino and amino nitrogen atoms present in the macrocyclic cavity can be utilized for coordination to metal ions. This aspect is being investigated.

1. Experimental

All preparations were carried out under nitrogen atmosphere by means of standard Schlenk techniques. Dispiro-$N_3P_3(C_{12}H_8O_2)_2$(N-(Me)$NH_2)_2$ (**2**) was prepared by literature method reported by us previously [5a]. Solvents and other general reagents used in this

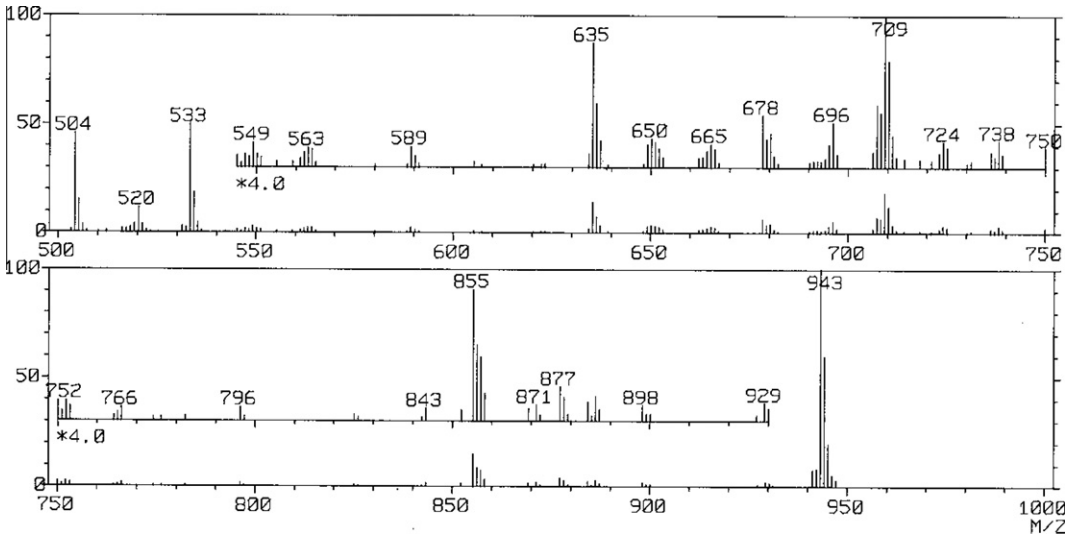


Fig. 1. Mass spectrum of compound **7**.

work were purified according to standard procedures. 2,2′-Dihydroxy biphenyl (Fluka) was used as obtained. $N_3P_3Cl_6$ (Aldrich) was recrystallized from *n*-hexane before use. *N*-Methylhydrazine was obtained as a gift from Prof. V. Chandrasekhar, Department of Chemistry, IIT-kanpur, India, and used as such. Melting points were measured using a JSGW melting point apparatus and are uncorrected. Elemental analyses were carried out by using a Thermoquest CE instruments model EA/110 CHNS-O elemental analyzer. ^{1}H and ^{31}P NMR spectra were recorded in $CDCl_3$ solution on a JEOL spectrometer operating at 400.0 and 161.7 MHz, respectively. Chemical shifts are reported with respect to internal tetramethylsilane (1H) or external 85% H_3PO_4 (^{31}P).

1.1. Synthesis

1.1.1. Synthesis of **2**

Compound **2** was prepared by adopting known synthetic procedure [5a]. The quantities involved and the spectral characterization are given below. $N_3P_3Cl_2(O_2C_{12}H_8)_2$ (3.00 g, 5.24 mmol) and *N*-methylhydrazine (1.20 g, 26 mmol). (125 mL). Yield (3 g, 88.5%). Mp: 278 °C. ^{1}H NMR: 2.96 (d, 6H, –N(C*H*$_3$); ^3J(1H-^{31}P)) 11.0 Hz), 3.08 (s, 4H, –N*H*$_2$), 7.24–7.50 (m, 16H, aromatic). ^{31}P NMR: 29.0 (t, P(N(Me)–)$_2$), 26.2 (d, P($O_2C_{12}H_8$)$_2$), ^2J(PN–P)) 57.95 Hz. *Anal.* Calc. for $C_{26}H_{26}N_7O_4P_3$: C, 52.62; H, 4.42; N, 16.52. Found: C, 52.29; H, 4.40; N, 16.41.

1.1.2. Synthesis of **3**

2-Hydroxy-5-methyl-benzaldehyde (1 g, 7.3 mmol), *N*,*N*′-dimethyl-ethane-1,2-diamine (0.50 mL, 4.86 mmol) and paraformaldehyde (0.2 g) in ethanol and acetic acid mixture (7:3) (100 mL) was heated under reflux for 36 h. The reaction mixture was filtered and evaporated to afford **2** as a pale yellow solid, which were purified subsequently by recrystallization in chloroform. Yield: 85%; ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): 2.26 (s, 3H), 2.28 (s, 3H), 2.67 (s, 3H), 2.68 (s, 3H), 3.0 (s, 4H), 3.65 (s, 4H) 7.22 (s, 2H), 7.38 (s, 2H), 10.21 (s, 1H); *Anal.* Calc. for. $C_{22}H_{28}N_2O_4$ (%): C, 68.73; H, 7.34; N, 7.29. Found: C, 68.11; H, 6.98; N, 7.15.

1.1.3. Synthesis of **4**

Compound **4** was prepared by adopting above synthetic procedure. The quantities involved and the spectral characterization are given below. 2-Hydroxy-4,5-di-methyl-benzaldehyde (1 g, 6.66 mmol), *N*,*N*′-dimethyl-ethane-1,2-diamine (0.5 mL, 4.46 mmol) and paraformaldehyde (0.30 g) .Yield: 80%; ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): 2.26 (s, 3H), 2.28 (s, 3H), 2.67 (s, 3H), 2.68 (s, 3H), 3.0 (s, 4H), 3.65 (s, 4H) 7.25 (s, 2H), 10.20 (s, 1H); *Anal.* Calc. for. $C_{24}H_{32}N_2O_4$ (%): C, 69.86; H, 7.82; N, 6.79. Found: C, 69.20; H, 6.15; N, 6.45.

1.1.4. Synthesis of **5**

Compound **5** was prepared by adopting the similar synthetic procedure used for **3**. The quantities involved and the spectral characterization are given below. 5-Chloro-2-hydroxy-benzaldehyde (760 mg, 4.86 mmol), *N*,*N*′-dimethyl-ethane-1,2-diamine (0.35 mL, 3.24 mmol) and paraformaldehyde (0.32 g) .Yield: 82%; ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): 2.3 (s, 3H), 2.32 (s, 3H), 3.20 (s, 4H), 3.70 (s, 4H) 7.32 (s, 4H), 10.35 (s, 1H); *Anal.* Calc. for. $C_{20}H_{22}Cl_2N_2O_4$ (%): C, 56.48; H, 5.21; N, 6.59. Found: C, 55.92; H, 4.89; N, 5.96.

1.1.5. Synthesis of **6**

Compound **6** was prepared by adopting the similar synthetic procedure used for **3**. The quantities involved and the spectral characterization are given below. 5-Bromo-2-hydroxy-benzaldehyde (1 g, 2.43 mmol), *N*,*N*′-dimethyl-ethane-1,2-diamine (0.20 mL, 1.8 mmol) and paraformaldehyde (0.28 g). Yield: 80%; ^{1}H NMR ($CDCl_3$, 400 MHz, ppm): 2.25 (s, 3H), 2.29 (s, 3H), 3.21 (s, 4H), 3.72 (s, 4H) 7.31 (s, 4H), 10.32 (s, 1H); *Anal.* Calc. for. $C_{20}H_{22}Br_2N_2O_4$ (%): C, 46.72; H, 4.31; N, 5.45. Found: C, 45.98; H, 3.96; N, 4.89.

1.1.6. Synthesis of **7**

A solution of {(C_6H_5)P(O)[N(Me)NH_2]$_2$} (**2**) (0.42 g, 2.0 mmol) in methanol (100 mL) and **3** (0.77 g, 2.0 mmol) in chloroform (100 mL) were added simultaneously drop wise (1 h) to a round bottom flask containing methanol (100 ml) maintained at 0 °C under constant stirring. The reaction mixture was stirred at 25 °C for about 5 h and the solvent evaporated *in vacuo* to afford **5** as a solid product. This was purified by column chromatography using chloroform/hexane (1:9) as eluting solvent. Yield: 0.89 g (75%); Mp. 228 °C decomposes); ^{31}P NMR ($CDCl_3$, 161.7 MHz): δ 24.4 (d, –*P*(–$O_2C_{12}H_8$)$_2$), 17.7 (t, –*P* (O_4 $N_4C_{20}H_{26}$(CH=)$_2$)), ^2J(P–N–P) = 98.6 Hz); ^{1}H NMR ($CDCl_3$, 400 MHz): 2.28 (s, 3H), 2.30 (s, 3H), 2.71 (s, 3H), 2.74 (s, 3H), 3.13 (s, 6H), 3.25 (s, 4H), 3.68 (s, 4H) 7.24 (s, 2H), 7.41–7.83 (m, 20H); MS(FAB): 943 $(M+1)^+$. *Anal.* Calc. for. $C_{48}H_{50}N_9O_6P_3$: C, 61.21; H, 5.35; N, 13.38. Found: C, 60.92; H, 4.98; N, 12.86.

*1.1.7. Synthesis of **8***

Compound **6** was prepared by adopting above synthetic procedure. The quantities involved and the spectral characterization are given below. $\{(C_6H_5)P(O)[N(Me)NH_2]_2\}$ (**2**) (0.42 g, 2.0 mmol), **4** (0.77 g, 2.0 mmol). Yield: 0.89 g (75%); Mp. 242 °C decomposes); ^{31}P NMR ($CDCl_3$, 161.7 MHz): δ 25.0 (d, $-P(-O_2C_{12}H_8)_2$), 18.8 (t, $-P$ (O_4 $N_4C_{20}H_{26}(CH{=})_2$)), ^2J(P–N–P) = 99.2 Hz); ^1H NMR ($CDCl_3$, 400 MHz): 2.26 (s, 3H), 2.31 (s, 3H), 2.73 (s, 3H), 2.75 (s, 3H), 3.20 (s, 6H), 3.72 (s, 4H) 7.30–7.75 (m, 18H); MS(FAB): 965 $(M-4)^+$. *Anal.* Calc. for. $C_{50}H_{54}N_9O_6P_3$: C, 61.91; H, 5.61; N, 13.0. Found: C, 61.45; H, 5.25; N, 12.78.

*1.1.8. Synthesis of **9***

Compound **6** was prepared by adopting above synthetic procedure. The quantities involved and the spectral characterization are given below. $\{(C_6H_5)P(O)[N(Me)NH_2]_2\}$ (**2**) (0.42 g, 2.0 mmol), **5** (0.85 g, 2.0 mmol). Yield: 78%; Mp. 250 °C decomposes); ^{31}P NMR ($CDCl_3$, 161.7 MHz): δ 25.2 (d, $-P(-O_2C_{12}H_8)_2$), 18.5 (t, $-P$ (O_4 $N_4C_{20}H_{26}(CH{=})_2$)), ^2J(P–N–P) = 99 Hz); ^1H NMR ($CDCl_3$, 400 MHz): ^1H NMR ($CDCl_3$, 400 MHz): 2.25–2.30 (s, 6H), 3.17 (s, 6H), 3.72 (s, 4H), 7.25–7.82 (m, 20H). *Anal.* Calc. for. $C_{46}H_{44}Cl_2N_9O_6P_3$: C, 56.21; H, 4.51; N, 12.83. Found: C, 55.52; H, 3.95; N, 11.92.

*1.1.9. Synthesis of **10***

Compound **10** was prepared by adopting similar synthetic procedure used for **7**. The quantities involved and the spectral characterization are given below. $\{(C_6H_5)P(O)[N(Me)NH_2]_2\}$ (**2**) (0.42 g, 2.0 mmol), **6** (1 g, 2.0 mmol). Yield: 72%; Mp. 235 °C decomposes); ^{31}P NMR ($CDCl_3$, 161.7 MHz): δ 25.3 (d, $-P(-O_2C_{12}H_8)_2$), 18.4 (t, $-P$ (O_4 $N_4C_{20}H_{26}(CH{=})_2$)), ^2J(P–N–P) = 99 Hz); ^1H NMR ($CDCl_3$, 400 MHz): 2.26–2.31 (s, 6H), 3.15 (s, 6H), 3.73 (s, 4H), 7.28–7.89 (m, 20H). *Anal.* Calc. for. $C_{46}H_{44}Br_2N_9O_6P_3$: C, 51.56; H, 4.14; N, 11.76. Found: C, 50.86; H, 3.83; N, 11.02.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.ica.2012.03.016.

References

[1] (a) Z. Xiao-fei, L. Yan-hong, Z. Di, W. Le, Y. Yong, Z. Yufen, Phosphorus, Sulfur, and Silicon 186 (2011) 281;
(b) B. Serap, F. Hanife, K. Adem, U. Ilker, Y. Fatma, Polyhedron 29 (2010) 3220;
(c) K. Keshav, N. Singh, A.J. Elias, Inorg. Chem. 49 (2010) 5753;
(d) A. Wang, C. Ornelas, D. Astruc, P. Hapiort, J. Am. Chem. Soc. 131 (2009) 6652;
(e) A.K. Diallo, J.C. Daran, F. Varret, J. Ruiz, D. Astruc, Angew. Chem., Int. Ed. 48 (2009) 314;
(f) V. Chandrasekhar, P. Thilagar, B. Murugesa Pandian, Coord. Chem. Rev. 251 (2007) 1045;
(g)M. Gleria, R. De Jaeger (Eds.), Applicative Aspects of Cyclophosphazenes, Nova Science, New York, 2004;
(h) Y. Mustafa, E. Digdem, U. Huseyin, I.O. Nazan, J. Mol. Struct. 753 (2005) 165;
(i) A.J. Elias, J.M. Shreeve, Adv. Inorg. Chem. 52 (2001) 335;
(j) C.W. Allen, Coord. Chem. Rev. 130 (1994) 137;
(k) M. Witt, H.W. Roesky, Chem. Rev. 94 (1994) 1163;
(l) C.W. Allen, Chem. Rev. 91 (1991) 119;
(m) S.S. Krishnamurthy, A.C. Sau, M. Woods, Adv. Inorg. Chem. Radiochem. 21 (1978) 41.
[2] (a) K.R. Carter, M. Calichman, C.W. Allen, Inorg. Chem. 48 (2009) 7476;
(b) V. Chandrasekhar, Inorganic and Organometallic Polymers, Springer-Verlag, Heidelberg, Germany, 2005;
(c) P.I. Richards, A. Steiner, Inorg. Chem. 44 (2005) 275;
(d) J.-H. Jung, S.K. Potluri, H. Zhang, P. Wisian-Neilson, Inorg. Chem. 43 (2004) 7784;
(e) J.E. Mark, H.R. Allcock, R. West, Inorganic Polymers, Prentice Hall, Englewood Cliffs, NJ, 1992;
(f) R.A. Shaw, Phosphorus Sulfur Silicon 45 (1989) 103;
(g) J.-F. Labarre, Top. Curr. Chem. 129 (1985) 173;
(h) H.R. Allcock, K.D. Lavin, G.G. Riding, Macromolecules 18 (6) (1985) 1341.
[3] (a) H.R. Allcock, Chemistry and Applications of Polyphosphazenes, Wiley Interscience, New York, 2003;
(b) K.R.J. Thomas, V. Chandrasekhar, P. Pal, S.R. Scoot, R. Hallford, A.W. Cordes, Inorg. Chem. 32 (1993) 606;
(c) K.R.J. Thomas, V. Chandrasekhar, S.R. Scott, R. Hallford, A.W. Cordes, J. Chem. Soc., Dalton Trans. (1993) 2589;
(d) K.R.J. Thomas, P. Tharmaraj, V. Chandrasekhar, Polyhedron 14 (1995) 977;
(e) K.R.J. Thomas, V. Chandrasekhar, Polyhedron 14 (1995) 1607;
(f) H.R. Allcock, Chemistry and Applications of Polyphosphazenes, Wiley, Hoboken, NJ, 2003;
(g) M. Gleria, R. De Jaeger, Phosphazenes-a World Wide Insight, NovaScience, New York, 2004.
[4] (a) V. Chandrasekhar, K.R.J. Thomas, Appl. Organomet. Chem. 7 (1993) 1;
(b) K.V. Katti, V. Sreenivasa Reddy, P.R. Singh, Chem. Soc. Rev 97 (1995);
(c) K.V. Katti, P.R. Singh, C.L. Barnes, Inorg. Chem. 31 (1992) 4588;
(d) E.W. Ainscough, A.M. Brodie, C.V. Depree, J. Chem. Soc., Dalton Trans. (1999) 4123;
(e) E.W. Ainscough, A.M. Brodie, C.V. Depree, C.A. Otter, Polyhedron 25 (2006) 2341;
(f) R. Horvath, C.A. Otter, K.C. Gordon, A.M. Brodie, E.W. Ainscough, Inorg. Chem. 49 (2010) 4073;
(g) E.W. Ainscough, A.M. Brodie, A. Derwahl, S. Kirk, C.A. Otter, Inorg. Chem. 46 (2007) 9841;
(h) V. Chandrasekhar, S. Nagendran, Chem. Soc. Rev. 30 (2001) 193;
(i) V. Chandrasekhar, V. Krishnan, in: M. Gleria, R. De Jaeger (Eds.), Applicative Aspects of Cyclophosphazenes, Nova Science, New York, 2004, p. 159;
(26)(j) H.R. Allcock, J.L. Desorcie, G.H. Riding, Polyhedron 6 (1987) 119.
[5] (a) V. Chandrasekhar, V. Krishnan, A. Steiner, J.F. Bickley, Inorg. Chem. 43 (2004) 166;
(b) V. Chandrasekhar, B.M. Pandian, R. Azhakar, Inorg. Chem. 45 (2006) 3510;
(c) P.I. Richards, A. Steiner, Inorg. Chem. 43 (2004) 2810;
(d) M. Harmjanz, B.L. Scoot, C.J. Burns, Chem. Commun. (2002) 1386;
(e) K.R.J. Thomas, V. Chandrasekhar, P. Pal, S.R. Scoot, R. Halford, A.W. Cordes, Inorg. Chem. 32 (1993) 606;
(f) E.W. Ainscough, A.M. Brodie, B. Moubaraki, K.S. Murray, C.A. Otter, Dalton Trans. 20 (2005) 3337;
(g) E.W. Ainscough, A.M. Brodie, C.V. Depree, G.B. Jameson, C.A. Otter, Inorg. Chem. 44 (2005) 7325;
(h) A.J. Heston, M.J. Panzner, W.J. Youngs, C.A. Tessier, Inorg. Chem. 44 (2005) 6518;
(i) K. Inoue, T. Itaya, N. Azuma, Supramol. Sci. 5 (1998) 163;
(j) P. Sozzani, A. Comotti, S. Bracco, R. Simonutti, Angew. Chem., Int. Ed. 43 (2004) 2792;
(k) M. Gleria, R. Bertani, R. De Jaeger, J. Inorg. Organomet. Polym. 14 (2004) 1;
(l) S. Kumaraswamy, M. Vijjulatha, C. Muthiah, K.C. Kumara Swamy, U. Engelhardt, J Chem. Soc., Dalton Trans. (1999) 891;
(m) P. Kommana, S. Kumaraswamy, K.C. Kumara Swamy, Inorg. Chem. Commun. 6 (2003) 394;
(n) N.S. Kumar, K.C. Kumara Swamy, Polyhedron 24 (2004) 979;
(o) N.S. Kumar, K.C. Kumara Swamy, Acta Crystallogr., Sect. C 57 (2001) 1421.
[6] (a) M. Rajeswara Rao, R. Bolligarla, R.J. Butcher, M. Ravikanth, Inorg. Chem. 49 (2010) 10606;
(b) S. Sumitra, Tetrahedron Lett. 44 (2003) 7281;
(c) K. Muralidharan, P. Venugopalan, A.J. Elias, Inorg. Chem. 42 (2003) 3176;
(d) C.N. Myer, C.W. Allen, Inorg. Chem. 41 (2002) 60;
(e) G. Franc, S. Mazeres, C.-O. Turrin, L. Vendier, C. Duhayon, A.-M. Caminade, J.-P. Majoral, J. Org. Chem. 72 (2007) 8707;
(f) O. Mongin, A. Pla-Quintana, F. Terenziani, D. Drouin, C.L. Droumaguet, A.-M. Caminade, J.-P. Majoral, M. Blanchard-Desce, New J. Chem. 31 (2007) 1354;
(g) T.R. Krishna, M. Parent, M.H.V. Werts, L. Moreaux, S. Gmouh, S. Charpak, A.-M. Caminade, J.-P. Majoral, M. Blanchard-Desce, Angew. Chem., Int. Ed. 45 (2006) 4645;
(h) L. Brauge, G. Veriot, G. Franc, R. Deloncle, A.-M. Caminade, J.-P. Majoral, Tetrahedron 62 (2006) 11891;
(i) O. Mongin, T.R. Krishna, M.H.V. Werts, A.-M. Caminade, J.-P. Majoral, M. Blanchard-Desce, Chem. Commun. (2006) 915.
[7] (a) J. Jo'elle Mitjaville, A.-M. Caminade, J.-C. Daran, B. Donnadieu, J.-P. Majoral, J. Am. Chem. Soc. 117 (1995) 1712;
(b) S. Beşli, S.J. Coles, D.B. Davies, A.O. Erkovan, M.B. Hursthouse, Inorg. Chem. 47 (2008) 5042;
(c) R. Boomishankar, P.I. Richards, A.K. Gupta, A. Steiner, Organometallics 29 (2010) 2515.
[8] V. Chandrasekhar, S. Nagendran, Chem. Soc. Rev. 30 (2001) 193.
[9] (a) I. Porwolik-Czomperlik, K. Brandt, A. Clayton, D.B. Davies, R.J. Eaton, R.A. Shaw, Inorg. Chem. 41 (2002) 4944;
(b) K. Brandt, I. Porwolik, M. Siwy, T. Kuoka, R.A. Shaw, D.B. Davies, M.B. Hursthouse, G.D. Sykara, J. Am. Chem. Soc. 119 (1997) 1143.
[10] V. Chandrasekhar, G.T. Senthil Andavan, R. Azhakar, B. Murugesa Pandian, Tetrahedron Lett. 47 (2006) 8365.