Synthesis of Novel Oxime and Oxime Derivatives Phosphazenes from Hexachlorocyclotriphosphazene

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Summary: The new spirocyclophosphazene 2,2-bis(4-benzoylphenoxy)-4,4,6,6-bis[spiro(2',2''-dioxy-1',1''-biphenyl]cyclotriphosphazene (3) was synthesized from the reaction of 2,2-dichloro4,4,6,6-bis[spiro(2',2''-dioxy-1',1''-biphenyl]cyclotriphosphazene (2) with 4-hydroxybenzophenone. The novel oxime-cyclophosphazene containing 2,2''-dioxybiphenyl groups (4) was synthesized from the reaction of 3 with hydroxylamine hydrochloride in pyridine. The reactions of 4 with methyl iodide, benzyl chloride, acetyl chloride, benzoyl chloride, 4-methoxybenzoyl chloride, 2-chlorobenzoyl chloride, propanoyl chloride, 2-bromoethanol and chloroacetyl chloride were studied. Disubstituted compounds were obtained from the reactions of 4 with methyl iodide, benzyl chloride, acetyl chloride, benzyl chloride, 4-methoxybenzoyl chloride, 2-chlorobenzoyl chloride and propanoyl chloride. Pure and defined products could not be obtained from the reaction of 4 with 2-bromoethanol and chloroacetyl chloride. All products were generally obtained in high yields. The structures of the compounds were defined by elemental analysis, IR, 1H, 13C and 31P-NMR spectroscopy.

Keywords: Hexachlorocyclotriphosphazene, phosphazene, oxime, oxime derivatives, oxime-phosphazene

Introduction

Phosphazenes are compounds that contain a framework of alternating phosphorus and nitrogen atoms, either in cyclic or linear form [1]. These compounds are known to exhibit useful thermal properties such as flame retardancy and self-extinguish ability. In addition to thermally favorable properties, the versatility in chemical transformation of P-Cl group of hexachloro-cyclotriphosphazene with organic groups is highly convenient to design functional polymers or molecules [2-8]. Phosphazenes also possess a number of characteristics such as biomedical properties and applications due to their strong antitumor activity [9-13]. Their antimicrobial and biological activities on bacterial and yeast cells have been studied [14-16]. Some applications include model compounds for polyphosphazenes, starting materials for the preparation of cyclolinear and/or cyclomatrix phosphazene substrates, com-mercal polymers with carbon backbones containing pendant cyclophosphazene groups, inorganic hydraulic fluids and lubricants, biologically important substrates such as anti-cancer agents, insect chemosterilants, pesticides and fertilizers, supports for catalysts, dyes, and crown ether phase transfer catalysts for nucleophilic substitution reactions, core substrates for dendrimers, thermal initiators for anionic polymerization reactions and photosensitive materials [17].

Oxime-containing molecules caught attention of researcher, as they appear to be amenable to biotransformation and conjugations with organic and inorganic molecules. The properties of these classes of compounds have been recently exploited with the aim to design and develop novel therapeutic agents that can display acyl group transfer capabilities and serve for the evaluation of novel candidate drugs for the treatment of various diseases. For example, furan oximes were found to inhibit DNA, RNA, and protein synthesis in lipoid leukemia cells [18]. Derivatives of quinoline oximes were also shown to possess antitumor activity, and glucosinolates were suggested as cancer preventive agents [19]. The stability of oxime complexes with various metals has been shown to result in promising compounds with antitumor activity, such as cis and trans platinum complexes and homo- and heteronuclear Cu(II) and Mn(II) oxime complexes [20, 21].

The literature contains reports on the synthesis of different linear, cyclic or poly phosphazenes [22-28]. The synthesis and different reactions of phosphazenes containing 2,2'-dioxybiphenyl groups are reported [29-30]. There are also a large number of literature reports on reactions of the functional groups on phosphazene substituents [31]. Typical of these include coupling reactions of trimeric phosphazene azides with arylxyo, alkoxy and...
dialkylamino cosubstituents [32], N-vinylphosphazenes with azo-decarboxylic and acetylenic esters [33], polymers from 4-formylphenoxo [34-35], maleic [36] and 3,4-methylenedioxyphenoxo substituents [37].

Recently we have synthesized oxime-phosphazene derivatives. For the synthesis of these compounds, we used full and partially substitue products of the hexachlorocyclotriphosphazene [38-44]. In the present study, we synthesized a new cyclophosphazene, 2,2-bis(4-benzoylphen-oxo)-4,4,6,6-bis[spiro(2',2''-dioxy-1',1''-biphenyl)] cyclophosphazene (3). From the reaction of 3 with hydroxylamino hydrochloride, the novel oxime-cyclophosphazene containing 2,2′-dioxobiphenyl groups (4) was synthesized. And we also studied the reactions of 4 with different alkyl or acyl halogens.

**Results and Discussion**

The reaction of 2 with 2 equiv. of 4-hydroxybiphenylphenone in the presence of K2CO3 in acetone gave 2,2-bis(4-benzoylphenoxo)-4,4,6,6-bis[spiro(2',2''-dioxy-1',1''-biphenyl)] cyclophosphazene (3). The novel oxime-cyclophosphazene (4) was synthesized from the reaction of 3 with hydroxylamine hydrochloride in pyridine.

Disubstituted compounds were obtained from the reactions of 4 with methyl iodide, benzyl chloride, acetyl chloride, benzoyl chloride, 4-methoxybenzoyl chloride, 2-chlorobenzoyl chloride and propanoyl chloride in acetone in the presence of K2CO3 via replacement of all the oxime protons with alkyl and acyl groups. Pure and defined products could not be obtained from the reaction of 4 with chloroacetyl chloride and 2-bromoethanol.

The structures of the compounds were elucidated by IR, 1H, 13C and 31P-NMR spectroscopy as well as by elemental analysis. General presentation of the reactions is shown in Scheme 1 and structures of the compounds 2-11 are shown in Scheme 2. All products were generally obtained in high yields. To check the nature of the peaks, example spectra ( IR, 31P, 1H and 13C-NMR spectra of 6 ) were presented in Fig. 1-4 respectively.

The characteristic stretching peaks in the IR spectra of the phosphazenes have been assigned as in experimental section. The P=N stretching vibrations, which are observed between 1171 and 1194 cm–1, are characteristic of cyclophospha-zenes. These peaks are shifted to longer wavelengths for 2–11 than in 1, which appeared at 1218 cm–1. The OH stretching vibration in the IR spectra of 4 indicates the oxime compound. The absence of the OH stretching vibration in the IR spectra of 5–11 indicates that all hydrogen atoms of the OH groups have been replaced by the alkyl and acyl substituents.

The 31P-NMR data for 2–11 are given in Table-1 (AB2 system). It is expected that one doublet and one triplet in the AB2 systems. However, The 31P-NMR spectra of the compounds 2–11 did not show the expected AB2 pattern. There are two peaks in the 31P-NMR spectra of 2–11 as a one doublet and one doublet of doublet. Further splitting was observed, which indicates that the two phosphorus atoms attached to the dioxobiphenyl ring are not magnetically equi-va lent. This non-equivalence of the two phosphorus atoms could be due to the difference in the angle of twist of the two phenyl groups of the biphenyl moieties and their twist in a different direction. The reason for this reversal twist/distortion could be due to the advantageous thermodynamically stable seven-membered dioxobiphenyl ring conformation by imparting reduced 6,6′ hydrogen-hydrogen contacts without broadening the O–P–O angl.

The observation of dd due to the two phosphorus atoms attached to the dioxobiphenyl groups indicates that such a conformation possibly exists in the solution. This may be due to the fact that in solution either averaging of the confor-mational possibilities is not complete or the twisted biphenyls of the dioxobiphenyl seven-membered spiro rings attend kinetically-stable conformations due to the intrinsic nature of the substitution groups. There are also extra peaks, with very weak signals, are observed in the spectra of 4–11. It is assumed that the weak peaks due to the syn and anti isomerism of the –C=N groups. The effects of the syn and anti isomerism are also observed in the 1H and 13C-NMR spectra of 4–11. This data demonstrates that compounds 4–11 consist of a mixture of two isomers.

The 1H and 13C-NMR data also confirm the structures of 2–11 (Scheme 2). In the 1H-NMR spectra, the OH proton is observed at 11.56 ppm for 4. It is understood from the integral intensities that there are two OH protons in 4, which is the original oxime-phosphazene containing 2,2′-dioxobiphenyl groups. The aromatic protons for all the compounds appear between 7.16 and 8.17 ppm.
Pure and defined products could not be obtained.

Scheme 1: General presentation of the reactions
Scheme 2: The structures of the compounds (2-11)

Fig. 1: The I.R Spectrum of 6.
Fig. 2: The $^{31}$P-NMR Spectrum of 6.

Fig. 3: The $^1$H-NMR Spectrum of 6.

Fig. 4: The $^{13}$C-NMR. Spectrum of 6.

Table 1: The $^{31}$P-NMR data of 2-11. (AB$_2$ system.)

<table>
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<td>25.28</td>
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Table 1: The $^{31}$P-NMR data of 2-11. (AB$_2$ system.)

The detailed $^{13}$C-NMR spectral data are given in experimental section. The carbonyl carbon atom for 3 is observed at 190.2 ppm. Since the carbonyl group changed to imin group, the Carbonyl peaks were not observed for 4-11. The imine carbon peaks for 4-11 are observed at 154.88, 155.77, 156.16, 163.29, 163.05, 163.99, 162.89 and 163.52 ppm respectively.

In this paper we report on the preparation of oxime-cyclophosphazene containing 2,2'-dioxybiphenyl groups from 2,2-bis(4-benzoylphenoxy)-4,4,6,6-bis[spiro(2',2''-dioxy-1',1''biphenyl)cyclotriphosphazene, and studies on its reactions with different alkyl and acyl halogenes. We hope that this original work is potentially an useful addition to the literature and can lead to some high polymer works.

**Experimental**

Solvents and other liquids used in the experimental works were dried by conventional m-methods. Hexachlorocyclotriphosphazene [N$_3$P$_3$Cl$_6$] (1) was recrystallized from hexane. Other chemicals were used as purchased. 2,2-dichloro-4,4,6,6bis[sp-
ior(2',2''-dioxo-1',1'''-biphenylyl)cyclotriphosphazene (2) was prepared as described by Carriedo and et al [26]. The reaction of [N3P3Cl6] with the biphenyl-2,2'-diol was carried out under dry nitrogen. IR spectra were recorded on an ATI Unicam Mattson 1000 FTIR spectrometer. 1H, 13C, and 31P-NMR spectra were recorded using a Bruker DPX-300 spectrometer operating at 300.13, 75.46 and 121.49 MHz, respectively. The 1H and 13C-NMR chemical shifts were measured using SiMe4 as an internal standard, whereas those for 31P were measured using 85% H3PO4 as an external standard. Chemical shifts downfield from the standard were assigned positive δ values. Microanalysis was carried out by a LECO 932 CHNS-O apparatus.

Synthesis of Compound 2

A mixture of 1 (10.20 g, 29.34 mmol), biphenyl-2,2'-diol (10.70 g, 57.46 mmol), and K2CO3 (9.84 g, 71.34 mmol) was stirred in acetone (100 ml) at 0°C and then was reacted at ambient temperature for 24 h. The solvent was removed under vacuum. The residue was extracted with CH2Cl2 (4x75 ml). After the solvent was removed, a white solid (92% yield). IR (KBr), ν/cm⁻¹: 3034, 3071, 1194, 942. 1H-NMR (DMSO-d6) δ/ppm: 7.68 (d, 4H, J=7.45, H-5), 7.55 (t, 4H, J=7.60, H-3), 7.40 (m, 8H, H-2, H-4). 13C-NMR (DMSO-d6) δ/ppm: 147.30 (C-1), 130.73 (C-5), 130.52 (C-3), 129.15 (C-5), 129.03 (C-5, is), 128.83 (C-14), 128.72 (w) (C-14, is), 128.31 (C-6), 127.43 (C-14, is), 126.99 (C-4), 121.96 (C-2). EA (calculated/found for C24H16Cl2N3O4P3) (MW=574.22): % C, 50.20-6.92; H, 2.81; N, 7.32.

Synthesis of Compound 3

A mixture of 2 (10 g, 17.41 mmol), 4-hydroxybenzophenone (6.40 g, 32.29 mmol), and K2CO3 (9.84 g, 71.34 mmol) was stirred in acetone (100 ml) at 0°C and then refluxed for 4 h. The solvent was removed under vacuum. The residue was extracted with CH2Cl2 (4x75 ml). After the solvent was removed, a white solid (3) formed. 1H-NMR (DMSO-d6) δ/ppm: 7.68 (d, 4H, J=7.45, H-5), 7.55 (t, 4H, J=7.60, H-3), 7.40 (m, 8H, H-2, H-4). 13C-NMR (DMSO-d6) δ/ppm: 147.30 (C-1), 130.73 (C-5), 130.52 (C-3), 129.00 (C-6), 126.99 (C-4), 121.96 (C-2). EA (calculated/found for C52H40N5O8P3) (MW=955.82): % C, 65.34; H, 4.68; N, 7.32.

Synthesis of Compound 4

A mixture of 3 (10.00 g, 11.26 mmol) and hydroxylamine hydrochloride (2.2 g, 31.65 mmol) was refluxed in pyridine (15 mL) for 3.5 h. After the reaction was complete, the mixture was allowed to cool and was slowly poured into water (100 mL) and precipitated twice from water. The white solid (4) was washed with alcohol and dried at 50°C in a vacuum. Yield: 9.73 g (93%). IR (KBr), ν/cm⁻¹: 3299, 1602, 1175, 932. 1H-NMR (DMSO-d6) δ/ppm, (w: weak, is: isomer): 11.56 (s, 2H, H-16), 11.47 (w) (H-16, is), 7.67 (d, 4H, J=7.46, H-13), 7.61-7.10 (m, 30H, H-2, H-3, H-4, H-5, H-8, H-9, H-14, H-15), 13C-NMR (DMSO-d6) δ/ppm: 145.88 (C-11), 150.77 (J=5.56, C7), 150.15 (w) (C-7, is), 147.64 (J=7.23, C-1), 147.60 (w) (C-1, is), 137.06 (C-12), 134.87 (C-15), 133.76 (C-9), 131.38 (C-10), 130.71 (C-13), 130.33 (w) (C-13, is), 129.45 (C-3), 129.30 (w) (C-3, is), 129.15 (C-5), 129.03 (w) (C-5, is), 128.83 (C-14), 128.72 (w) (C-14, is), 128.31 (C-6), 127.43 (C-4), 127.15 (w) (C-4, is), 121.12 (J=10.83, C-2), 121.46 (C-8), 121.21 (w) (C-8, is), EA (calculated/found for C90H33N5O3P3) (MW=927.77): % C, 66.73; H, 3.91(4.08), N, 7.55(7.80).

Reaction of 4 with Methyl Iodide

A solution of 1.00 mL (2.28 g, 16.06 mmol) methyl iodide in acetone (10 mL) was slowly added dropwise to a stirred and cooled (0-5°C) mixture of 4 (0.70 g, 0.75 mmol) and K2CO3 (1.00 g, 7.24 mmol) in acetone (30 mL). The reaction was carried out at room temperature for 3 h and then was refluxed for 12 h. After the reaction was complete, the precipitate was filtered off and the solvent was removed. The product was dissolved in very little amount of acetone and was precipitated with water several times. The white solid (5) was washed with alcohol and dried at 50°C in a vacuum. Yield: 0.54 g (75%). IR (KBr), ν/cm⁻¹: 1601, 1179, 936. 1H-NMR (DMSO-d6) δ/ppm, (w: weak, is: isomer): 7.68 (d, 4H, J=6.00, H-13), 7.59-7.10 (m, 30H, H-2, H-3, H-4, H-5, H-8, H-9, H-14, H-15), 3.89 (s, 6H, H-16), 3.82 (w) (H-16, is). 13C-NMR (DMSO-d6) δ/ppm: 155.77 (C-11), 150.36 (C-7), 147.58 (C-1), 136.01 (C-12), 133.79 (C-15), 133.22 (C-9), 131.28 (C-10), 130.72 (C-13), 130.40 (w) (C-13, is), 130.06 (C-3), 129.52 (C-5), 129.42 (w) (C-5, is), 129.09 (C-14), 128.88 (w) (C-14, is), 128.31 (C-6), 127.72 (C-4), 127.18 (w) (C-4, is), 121.12 (J=10.83, C-2), 121.46 (C-8), 121.21 (w) (C-8, is), EA (calculated/found for C90H33N5O3P3) (MW=955.82): % C, 65.34(65.60), H, 4.22(4.00), N, 7.33(7.05).
**Reaction of 4 with Benzyl Chloride**

A solution of 1.00 mL (1.10 g, 8.69 mmol) benzyl chloride in acetone (10 mL) was slowly added dropwise to a stirred and cooled (0-5 °C) mixture of 4 (0.70 g, 0.75 mmol) and K₂CO₃ (1.00 g, 7.24 mmol) in acetone (30 mL). The reaction was carried out at room temperature for 24 h. After the reaction was complete, the precipitate was filtered off and the solvent was removed. The product was dissolved in very little amount of acetone and was precipitated with alcohol several times. The white solid (6) formed. Yield: 0.69 g (83%). IR (KBr), ν/cm⁻¹: 1603, 1173, 937. ¹H-NMR (DMSO-d₆) δ/ppm: (w: weak, is: isomer): 7.68 (d, 4H, J=6.99, H-13), 7.53-7.13 (m, 44H, H-2, H-3, H-4, H-5, H-8, H-9, H-14, H-15, H-18, H-19, H-20), 5.18 (s, 4H, H-16). ¹³C-NMR (DMSO-d₆) δ/ppm: 156.16 (C-11), 150.42 (C-7), 147.56 (C-1), 138.29 (C-12), 136.05 (C-17), 133.81 (C-15), 133.18 (C-9), 131.28 (C-10), 130.58 (C-13), 130.38 (C-13, is), 130.13 (C-3), 129.56 (C-5), 129.50 (C-5, is), 128.90-128.11 (5C, C-6, C-14, C-18, C-19, C-20), 127.78 (C-4), 127.18 (C-4, is), 122.08 (C-2), 121.58 (C-8), 121.29 (C-8, is), 76.25 (C-16). EA (calculated/ found for C₆₄H₄₈N₅O₁₀P₃) (MW=1108.02): % C, 69.37(69.00), H, 4.37(4.08), N, 6.32(6.40).

**Reaction of 4 with Acetyl Chloride**

A solution of 1.00 mL (1.20 g, 15.28 mmol) acetyl chloride in acetone (10 mL) was slowly added dropwise to a stirred and cooled (0-5 °C) mixture of 4 (0.70 g, 0.75 mmol) and K₂CO₃ (1.00 g, 7.24 mmol) in acetone (30 mL). The reaction was carried out at room temperature for 24 h. After the reaction was complete, the precipitate was filtered off and the solvent was removed. The product was dissolved in very little amount of acetone and was precipitated with alcohol several times. The white solid (7) formed. Yield: 0.63 g (82%). IR (KBr), ν/cm⁻¹: 1772, 1602, 1173, 936. ¹H-NMR (DMSO-d₆) δ/ppm, (w: weak, is: isomer): 7.70-7.18 (m, 34H, H-2, H-3, H-4, H-5, H-8, H-9, H-13, H-14, H-15, H-18), 7.65-7.10 (m, 34H, H-2, H-3, H-4, H-5, H-8, H-9, H-13, H-14, H-15, H-19, H-20). ¹³C-NMR (DMSO-d₆) δ/ppm: 164.81 (C-16), 164.56 (C-16, is), 163.05 (C-11), 162.63 (C-11, is), 152.36 (C-7, is), 151.96 (J=8.26, C-7), 147.35 (C-1), 135.41 (C-12), 134.32 (C-20), 134.10 (C-20, is), 132.13 (C-15), 131.98 (C-15, is), 131.64 (C-9, is), 130.80-128.06 (9C, C-10, C-13, C-3, C-5, C-6, C-17, C-18, C-19), 127.00 (C-4), 121.88 (C-2), 121.73 (J=4.52, C-8), 121.35 (C-8, is). EA (calculated/found for C₄₆H₄₄N₅O₁₀P₃) (MW=1135.98): % C, 67.67(68.00), H, 3.90(4.18), N, 6.17(6.35).

**Reaction of 4 with 4-methoxybenzoyl Chloride**

A solution of 1.00 mL (1.20 g, 8.60 mmol) benzoyl chloride in acetone (10 mL) was slowly added dropwise to a stirred and cooled (0-5 °C) mixture of 4 (0.70 g, 0.75 mmol) and K₂CO₃ (1.00 g, 7.24 mmol) in acetone (30 mL). The reaction was carried out at room temperature for 24 h. After the reaction was complete, the precipitate was filtered off and the solvent was removed. The product was dissolved in very little amount of acetone and was precipitated with alcohol several times. The white solid (8) formed. Yield: 0.71 g (83%). IR (KBr), ν/cm⁻¹: 1750, 1601, 1174, 937. ¹H-NMR (DMSO-d₆) δ/ppm, (w: weak, is: isomer): 8.12 (d, 4H, J=7.33, H-18), 7.79-7.14 (m, 40H, H-2, H-3, H-4, H-5, H-8, H-9, H-13, H-14, H-15, H-19, H-20). ¹³C-NMR (DMSO-d₆) δ/ppm: 164.81 (C-16), 164.56 (C-16, is), 163.05 (C-11), 162.63 (C-11, is), 152.36 (C-7, is), 151.96 (J=8.26, C-7), 147.35 (C-1), 135.41 (C-12), 134.32 (C-20), 134.10 (C-20, is), 132.13 (C-15), 131.98 (C-15, is), 131.64 (C-9, is), 130.80-128.06 (9C, C-10, C-13, C-3, C-5, C-6, C-17, C-18, C-19), 127.00 (C-4), 121.88 (C-2), 121.73 (J=4.52, C-8), 121.35 (C-8, is). EA (calculated/found for C₄₆H₄₄N₅O₁₀P₃) (MW=1135.98): % C, 67.67(68.00), H, 3.90(4.18), N, 6.17(6.35).
114.76 (C-19), 114.58 (C-19, is), 56.23 (C-21), 56.03 (w) (C-21, is). EA (calculated/found for C_{64}H_{42}Cl_{2}N_{5}O_{10}P_{3}) (MW=1196.03): % C, 66.28 (66.47), H, 4.05 (4.20), N, 6.74 (6.88).

Reaction of 4 with 2-chlorobenzoyl Chloride

A solution of 1.00 mL (2.31 g, 13.20 mmol) 2-chlorobenzoyl chloride in acetone (10 mL) was slowly added dropwise to a stirred and cooled (0-5 °C) mixture of 4 (0.70 g, 0.75 mmol) and K_{2}CO_{3} (1.00 g, 7.24 mmol) in acetone (30 mL). The reaction was carried out at room temperature for 24 h. After the reaction was complete, the precipitate was filtered off and the solvent was removed. The product was dissolved in very little amount of acetone and was precipitated with alcohol several times. The white solid (10) formed. Yield: 0.76 g (91%). IR (KBr), v/cm⁻¹: 1760, 1690, 1174, 937. ¹H-NMR (DMSO-d₆) δ/ppm, (w: weak, is: isomer): 8.09 (d, 2H, H-22), 8.09-6.80 (m, 40H, H-2, H-3, H-4, H-5, H-8, H-9, H-13, H-14, H-15, H-19, H-20, H-21). ¹³C-NMR (DMSO-d₆) δ/ppm: 171.89 (C-16), 171.75 (w) (C-16, is), 134.76 (C-3), 133.10 (C-4), 129.87 (C-5), 129.87 (w) (C-5, is), 128.96 (C-14), 128.96 (w) (C-14, is), 128.70 (C-15), 128.70 (w) (C-15, is), 128.57 (C-16), 128.57 (w) (C-16, is), 128.44 (C-17), 128.44 (w) (C-17, is), 128.31 (C-18), 128.31 (w) (C-18, is), 128.28 (C-19), 128.28 (w) (C-19, is), 128.15 (C-20), 128.15 (w) (C-20, is), 128.02 (C-21), 128.02 (w) (C-21, is), 127.73 (C-13), 127.73 (w) (C-13, is), 127.60 (C-8), 127.60 (w) (C-8, is). EA (calculated/found for C_{66}H_{48}N_{5}O_{12}P_{3}) (MW=1196.03): % C, 66.28 (66.47), H, 4.05 (4.20), N, 6.74 (6.88).

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References