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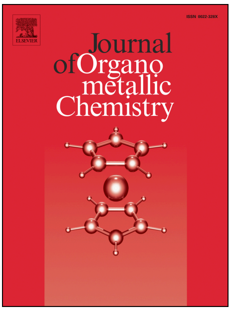

Synthesis and characterization of new (dimethylsilyl)phenoxy and (dimethyl(vinyl)silyl)phenoxy substituted cyclotriphosphazenes

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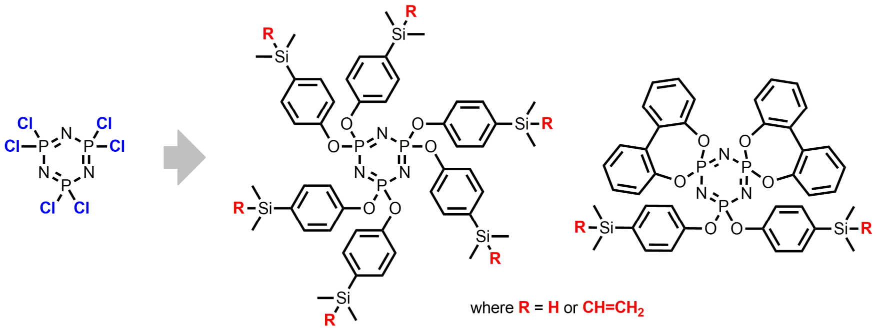

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Synthesis and Characterization of New (dimethylsilyl)phenoxy and (dimethyl(vinyl)silyl)phenoxy Substituted Cyclotriphosphazenes.

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Abstract

The routes of synthesis, as well as, spectroscopic and X-ray diffraction characterization of a new group of bis- or hexakis-4-(dimethyl(vinyl)silyl)phenoxy and 4-(dimethylsilyl)phenoxy-substituted cyclotriphosphazenes as a versatile starting substrates for the synthesis of a variety of organic cyclophosphazene derivatives, via catalytic hydrosilylation or silylative coupling processes, is presented.

Keywords

Cyclophosphazene, nucleophilic substitution, dimethylsilylphenoxy

Introduction

Cyclotriphosphazenes, are considered as one of the most interesting and oldest group of inorganic phosphorus and nitrogen heterocyclic compounds with general formula $N_3P_3R_6$,

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where R can be a chlorine atom or an organic group bonded to the phosphorus[1]. The origins of research on the synthesis, properties and applications of this group of compounds dates back to the mid-nineteenth century when Liebig and Rose used phosphorus pentachloride and ammonia to obtain the first known cyclic phosphazene trimer[2]. Since then hexachlorocyclotriphosphazene, because of the excellent reactivity of the P-Cl bond, was used over the decades as a versatile starting material for the synthesis of cyclotriphosphazene derivatives mainly by nucleophilic substitution reactions, in which chlorine atoms can be exchanged to a variety of functional groups[1,3] influencing, together with the nature of inorganic –P=N– skeleton the unique properties of cyclophosphazene derivatives[4]. Due to the mentioned varied properties cyclophosphazenes have been studied among others as flame retardant additives[5], high temperature fluids[6], clathrates[7], photostabilizers, or antioxidants in organic polymers[8]. They have also been used as phase transfer catalysts, as polypodants and cryptands[9]. Cyclophosphazenes have also been successfully used as biologically active agents[10]. Moreover cyclotriphosphazenes, were used for polymer functionalization and synthesis, as pendant groups or monomers for polyphosphazene synthesis, especially those bearing six chlorine atoms or phenoxy groups[3,11].

The literature on the synthesis and applications of cyclophosphazenes is very diverse, mainly because of mentioned above reactivity of P-Cl bond and functional groups substituted to the cyclotriphosphazene rings, which make them versatile precursors for synthesis of new derivatives and materials easily modifiable by many organic transformations. However, there are a limited number of examples describing their combinations with silicon substrates[12]. Moreover the information about the use of transition metal catalyzed processes (like hydrosilylation[13] or silylative coupling[14]) for the synthesis of functional cyclophosphazenes is even more scant. This is caused mainly by the lack of proper starting substrates for such transformations. Taking into account the lack of silyl cyclophosphazene derivatives having reactive dimethylsilyl or dimethyl(vinyl)silyl groups capable for functionalization via hydrosilylation or silylative coupling processes we decided to undertake attempts to synthesize mentioned above derivatives which can in our opinion affect the research development on the synthesis and possible applications of this group of compounds.

Results and Discussion

Synthetic procedures applied were based on nucleophilic substitution processes of Cl atom present in hexachlorocyclotriphosphazene substrate with 4-bromophenol and 2,2’-biphenol

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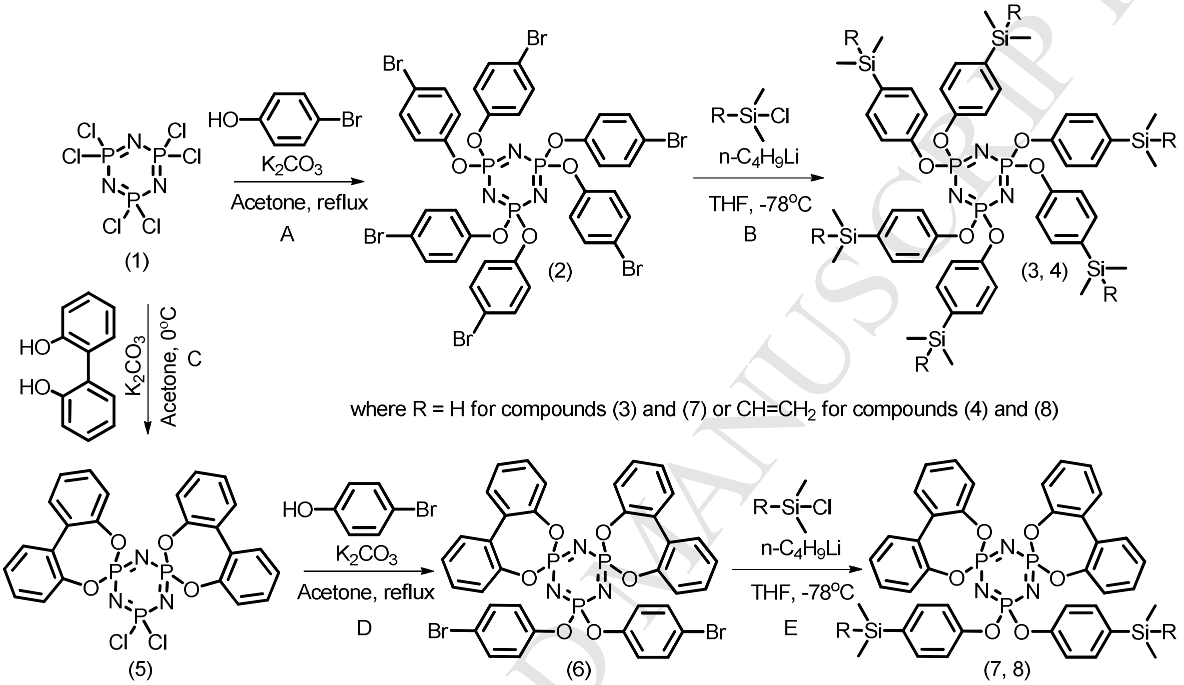
and subsequent reaction of obtained 4-bromophenoxycyclotriphosphazenes with chlorodimethyl silanes to yield the final products. While hexakis(4-(dimethyl(vinyl)silyl)phenoxy) and hexakis(4-(dimethylsilyl)phenoxy) cyclotriphosphazenes were obtained in two step synthesis, to prepare bis(biphenyl-2,2'-diyl)bis(4-(dimethylsilyl)phenoxy) and bis(biphenyl-2,2'-diyl)bis(4-(dimethyl(vinyl)silyl)phenoxy) bisspirocyclic derivatives three step synthesis were applied.



Scheme 1. General scheme of synthesis of bis- and hexakis- 4-(dimethylsilyl)phenoxycyclotriphosphazenes and 4-(dimethyl(vinyl)silyl)phenoxycyclotriphosphazenes.

Path to synthesis of hexakis(4-(dimethylsilyl)phenoxy) and hexakis(4-(dimethyl(vinyl)silyl)phenoxy) cyclotriphosphazenes (3) and (4) is presented on Scheme 1. A and B. In the first step (Scheme 1. A) via nucleophilic substitution of Cl atoms in the substrate (1) with 4-bromophenol in the presence of potassium carbonate hexakis(4-bromophenoxy)cyclotriphosphazene (2) has been synthesized with high 98% yield as a white crystalline powder. Compound 2 was obtained at first by H.R. Allcock et al. using sodium hydride in anhydrous THF.[15] However, employing K_2CO_3 as a base in acetone as solvent resulted in significant reduction in reaction time. Subsequently, in the second stage (Scheme 1. B) obtained intermediate (2) has been reacted with chlorodimethyl- or chlorodimethylvinylsilane in the presence of n-butyllithium to yield hexakis(4-

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(dimethylsilyl)phenoxy)cyclotriphosphazene (3) or hexakis(4-(dimethyl(vinyl)silyl)phenoxy)cyclotriphosphazene (4) as yellowish oils with 92 and 94% yield respectively.

It should be noted that functionalization of compound (2) with organolithium reagents was studied by Allcock et al. They established a condition enabling complete substitution of bromine atoms by lithium. Moreover, obtained organolithium cyclotriphoshazene derivative was reacted with CO_2, Ph_2PCl, Ph_2CO, C_4H_9Br, Ph_3SnCl, Ph_3PAuCl and D_2O[15]. These results definitely have broadened the knowledge of the reactivity of cyclophosphazenes, however, in our point of view incorporation of reactive organosilicon moieties to the cyclophosphazene rings should also have a similar impact on the field of cyclophosphazene chemistry.

Path to the synthesis of bis(biphenyl-2,2'-diyl)bis(4-(dimethylsilyl)phenoxy)cyclotriphosphazene (7) and bis(biphenyl-2,2'-diyl)bis(4-(dimethyl(vinyl)silyl)phenoxy) cyclotriphosphazene (8) is presented on Scheme 1. C, D and E. In this case the first step (Scheme 1. C) based on nucleophilic substitution of Cl atoms in substrate (1) with two equimolar amounts of 2,2'-biphenol in the presence of potassium carbonate bis(biphenyl-2,2'-diyl)bischlorocyclotriphosphazene (5) has been synthesized with 92% yield as a white solid according to the published method[16]. Next in the second stage (Scheme 1. D) obtained intermediate (5) has been reacted with 4-bromophenol to yield bis(biphenyl-2,2'-diyl)bis(4-bromophenoxy)cyclotriphosphazene (6) with 95% yield also as a white solid. Finally, in the third step (Scheme 1. E) intermediate (6) has been reacted with chlorodimethyl or chlorodimethylvinylsilane in the presence of n-butyllithium to prepare respectively bis(biphenyl-2,2'-diyl)bis(4-(dimethylsilyl)phenoxy)cyclotriphosphazene (7) and bis(biphenyl-2,2'-diyl)bis(4-(dimethyl(vinyl)silyl)phenoxy)cyclotriphosphazene (8) with 94 and 96% yield as a white crystals.

Structures of all obtained intermediates and final products were confirmed with NMR spectroscopy. The ^1H and ^{13}C NMR spectra were recorded using Bruker Ultrashield 300 MHz spectrometer while ^{29}Si and ^{31}P ones were recorded using Bruker Ascend 400 spectrometer. All spectra were collected at room temperature in $CDCl_3$ or toluene-d8 as solvents. Detailed analytical results, as well as, spectra recorded are available in Electronic Supplementary Information (ESI). Additionally because intermediate (6), as well as, products (7) and (8) formed white, crystalline solids, after recrystallization from diethyl ether or methylene chloride, X-ray structure analysis was performed.

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Diffraction data were collected at 100(1) K, by the ω-scan technique on Agilent Technologies Xcalibur four-circle diffractometer with Eos CCD detector and graphite-monochromated MoK$_\alpha$ radiation (λ=0.71069 Å). The data were corrected for Lorentz-polarization, as well as, for absorption effects[17]. Precise unit-cell parameters were determined by a least-squares fit of 973 (6), 7203 (7), and 13517 (8) reflections of the highest intensity, chosen from the whole experiment. The structures were solved with SIR92[18] and refined with the full-matrix least-squares procedure on F2 by SHELXL-2013[19]. All non-hydrogen atoms were refined anisotropically, Si-H hydrogen atoms in (7) were found in the difference Fourier map and their position was further refined. All other hydrogen atoms were placed in idealized positions and refined as 'riding model' with isotropic displacement parameters set at 1.2 (1.5 for methyl groups) times U$_{eq}$ of appropriate carrier atoms. In structure of (7) large voids filled with diffused electron density were found; and as the modelling of solvent molecules was unsuccessful, the SQUEEZE procedure[20] was applied. Some soft restraints were applied for the shape and size of thermal ellipsoids in this structure. The relevant experimental data and refinement details are listed in ESI. Perspective views of the molecules are shown on Figure 1. and selected geometrical characteristics are listed in Table 1. In all structures, the P_3N_3 rings are almost flat and P-O bonds are directed above and below the mean ring plane. It might be noted that NPN bond angles are systematically smaller than PNP ones.

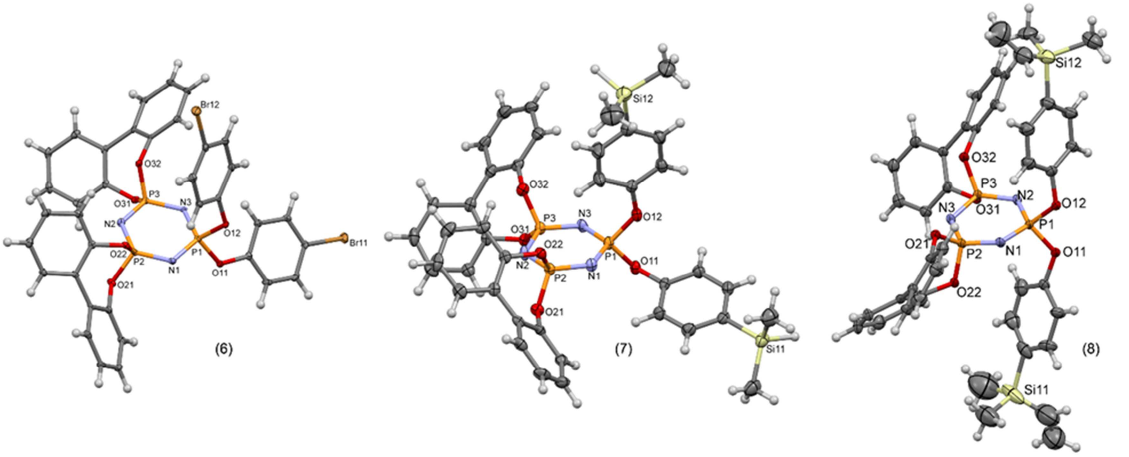

Figure 1. A perspective view of molecules of (6) (7) and one of the symmetry-independent molecule of (8); ellipsoids are drawn at the 50% probability level, hydrogen atoms are shown as spheres of arbitrary radii.

Dihedral angles in biphenyl fragments (substituents at P2 and P3) are similar in all structures while the disposition of the substituents at P1 - dihedral angles between two phenyl rings -

strongly depending on the size and kind of substitution at this ring. In the crystal structure of (6) characteristic triangular motifs created by halogen Br···Br interactions can be observed (see ESI Fig. 30).[19] Additionally, in this structure the disordered (across the threefold axis) solvent CH_2Cl_2 molecule is present, which fills the voids in the crystal structure (see ESI Fig. 31). Structures (7) and (8) are almost solely defined by van der Waals dispersive interactions, with no hydrogen bonding involved in the crystal architecture determination. Additionally in the structure of (8) there are two symmetry-independent molecules in the crystal structure, very similar in geometry.

Table 1. Selected geometrical parameters (Å, °) with s.u.’s in parentheses.

	(6)	(7)	(8)A	(8)B
NPN angles	117.79(19) 118.83(19) 118.17(19)	117.64(12 118.29(12) 118.48(12)	116.8(2) 118.0(2) 118.3(2)	117.3(2) 118.5(2) 118.3(2)
PNP angles	121.2(2) 121.6(2) 122.1(2)	122.11(14) 121.29(15) 121.46(14)	122.1(3) 122.0(3) 120.8(3)	121.5(3) 120.3(3) 121.6(3)
d_1	29.14(13)	79.64(8)	84.4(2)	85.35(17)
d_2	42.76(13)	41.65(10)	41.4(3)	44.23(18)
d_3	42.31(11)	41.98(9)	42.51(18)	42.4(3)
dev	0.038(2)	0.0518(12)	0.101(3)	0.106(3)

d_1, d_2 and d_3 denote the dihedral angles between the planes of phenyl rings at P_1 and biphenyl rings at P_2 and P_3, respectively, and dev is maximal deviation from P_3N_3 plane.

Conclusions

Presented methodologies enabled us to obtain with high yield a group of versatile starting substrates for the synthesis of a variety of organic cyclophosphazene derivatives via catalytic transformations. Potentially they can also be useful alternatives for tetramethyldisiloxanes or tetramethylcyclotetrasiloxanes, commonly used as silicone rubber crosslinkers. Moreover they can also play the role of co-monomers or seeds in linear or star shaped polymer and hybrid materials synthesis.

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Supporting Information

Detailed experimental procedures; Spectroscopic analysis of isolated compounds; Crystallographic data. This material is available free of charge via the Internet at

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Highlights

- Path of synthesis of dimethylsilylphenoxy substituted cyclophosphazenes is presented
- Spectral and x-ray characteristic of new derivatives is given
- Obtained compounds are useful substrates for their further catalytic transformations
- These compounds can be alternatives for conventional silicone rubber cross-linkers

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