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Synthesis and spectroscopic properties of geminal-bis(*tert*-butylamino)cyclotriphosphazenes obtained by the reaction of spiro and ansa phenoxycyclotriphosphazenes with the *tert*-butylamine and the crystal structure of 4,4'-bis(*tert*-butylamino)-2,6',6',10--tetrachloro-4',4',6',6'-tetrahydrospyro[12*H*-dibenzo[*d*,*g*]-[1,3,2]dioxaphosphocin-6,2'λ^5-[1,3,5,2,4,6]-triazaphosphorine]

DİĞDEM ERDENER[1], MUSTAFA YILDIZ[1]*, HÜSEYİN ÜNVER[2], NAZAN OCAK İSKELELİ[3] and TAHSİN NURİ DURLU[2]

[1]*Department of Chemistry, Faculty of Science and Arts, Çanakkale Onsekiz Mart University, TR-17100 Çanakkale,* [2]*Department of Physics, Faculty of Science, Ankara University, TR-06100 Tandoğan, Ankara and* [3]*Department of Physics, Faculty of Science and Arts, Ondokuz Mayıs University, TR-55139 Kurupelit, Samsun, Turkey*

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Abstract: The condensation reactions of partly substituted spiro and ansa phenoxycyclotriphosphazenes **1** and **2** with *tert*-butylamine produce disubstituted geminal-bis(*tert*-butylamino)phenoxycyclotriphosphazene derivatives (**3** and **4**). The structures of these compounds were characterized by elemental analysis, and IR, ^{1}H-, ^{13}C-, ^{31}P-NMR and mass spectroscopic techniques. Compound **3** was also examined by X-ray crystallography and found to be crystallized in the monoclinic space group $P2_1/n$ with the unit cell parameters: a = = 10.842(4), b = 9.375(5), c = 29.104(11) Å, β = 99.25(3)°, V = 2920(2) Å^3, D_x = 1.404 g cm^{-3}.

Keywords: phenoxyphosphazenes; spectroscopy; spiro; ansa; crystal structure.

INTRODUCTION

The reactions of trimeric phosphazene ($N_3P_3Cl_6$) with primary and secondary amines, diamines, polyamines [1–4] and aryloxides [5–14] under different conditions have been studied. The phenoxy derivatives of trimeric phosphazene ($N_3P_3Cl_6$) and tetrameric phosphazene ($N_4P_4Cl_8$) were used in the synthesis of new, small-molecule organocyclophosphazenes.[15] Polymeric phosphazene derivatives with inorganic backbones and aryloxy side groups are widely used as high refractive index glasses,[16] ferroelectric and non-linear optical devices,[17]

* Corresponding author. E-mail: myildiz@comu.edu.tr
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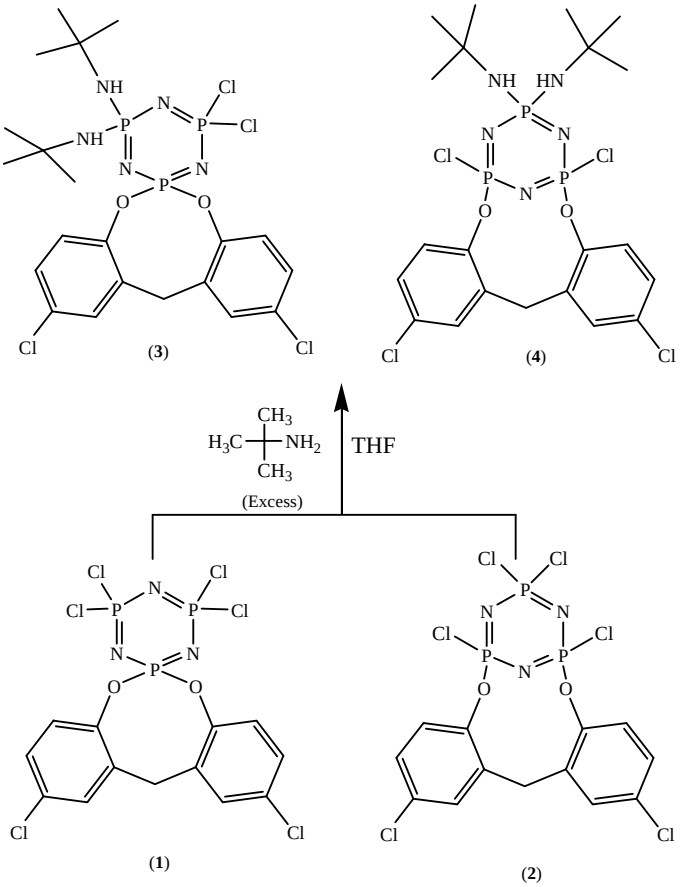
liquid crystalline materials,[18] and as biomedical materials.[19] Additionally, they can be used to create small molecule models for the corresponding linear phosphazene macromolecules. The organic, inorganic or organometallic side groups are highly effective in determining the specific physical or chemical properties of phosphazene polymers. Although the partial and complete aminolysis reactions of *tert*-butylamine [$NH_2C(CH_3)_3$] were studied[20] to obtain geminal *tert*-butylamino products, there have not been sufficient studies yet on the partial and complete aminolysis reactions of *tert*-butylamine for spiro or ansa phosphazene compounds. In the present study, the reaction of 2,4',4',6',6',10-hexachloro--4',4',6',6'-tetrahydrospiro[12*H*-dibenzo[*d*,*g*][1,3,2]dioxaphosphocin-6,2'λ^5--[1,3,5,2,4,6]triazaphosphorine] (spiro) (**1**) and 2,6,8,8,10,14-hexachloro-8,8--dihydro-6*H*-6λ^5,10λ^5-nitrilo-10*H*,16*H*-dibenzo[*h*,*k*][1,7,3,5,2,4,6]-dioxadiazatriphosphacyclododecine (ansa) (**2**) with *tert*-butylamine (Scheme 1) were examined and the new *gem*-diamino-spiro (**3**) (Fig. 1) and ansa (**4**) phenoxycyclotriphosphazene derivatives were isolated.



Scheme 1. Synthesis of compounds **3** and **4**.

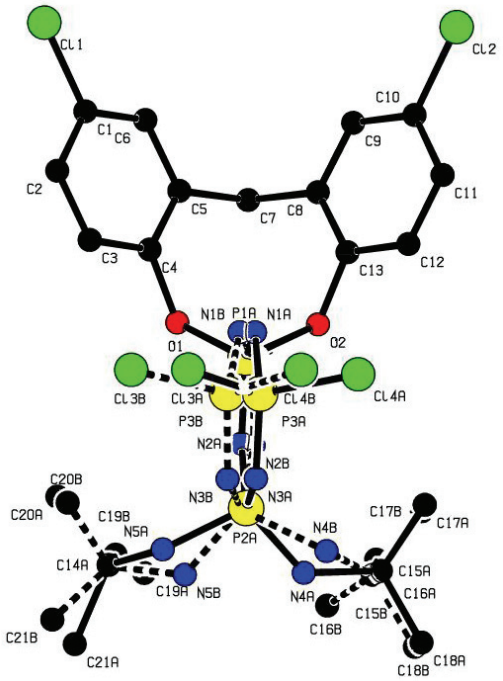


Fig. 1. The molecular structure of compound **3**; the H atoms have been omitted for clarity.[21]

RESULTS AND DISCUSSION

Analytical, IR and MS spectral data

*4,4'-Bis(*tert*-butylamino)-2,6',6',10-tetrachloro-4',4',6',6'-tetrahydrospyro-[12*H*-dibenzo[*d,g*][1,3,2]dioxaphosphocin-6,2'λ^5-[1,3,5,2,4,6]-triazaphosphorine] (spiro)* (**3**). Yield: 32 %; m.p.: 238 °C; Anal. Calcd. for $C_{21}H_{28}O_2N_5P_3Cl_4$: C, 40.84; H, 4.53; N, 11.34 %. Found: C, 40.99; H, 4.63; N, 11.31 %. IR (KBr, cm^{-1}): 3382 (*s*, N–H), 3081 (*w*, Ar–H), 2969 (*s*, C–H, aliphatic), 1481 (*s*, C=C), 1383 (*m*, C–O), 1276 (*s*, P–O), 1222 (*s*, P=N), 1178–1156–1110 (*s*, P–O–C), 581 + 525 (*s*, P–Cl). MS (highest peak in multiplet, based on Cl^{35}) (*m*/*z*): 617 (M^+, 100 %), 543 ((M – *tert*-BuNH), 10 %), 279 ((M – $CH_2(OArCl)_2Cl_2$), 13 %).

*8,8-Bis(*tert*-butylamino)-2,6,10,14-tetrachloro-8,8-dihydro-6*H*-6λ^5,10λ^5-nitrilo-10*H*,16*H*-dibenzo[h,k][1,7,3,5,2,4,6]-dioxadiazatriphosphacyclododecine (ansa) (**4**)*. Yield: 30 %; m.p.: 261 °C; Anal. Calcd. for $C_{21}H_{28}O_2N_5P_3Cl_4$: C, 40.84; H, 4.53; N, 11.34 %. Found: C, 40.89; H, 4.55; N, 11.33 %. IR (KBr, cm^{-1}): 3446 (*m*, N–H), 3086 (*w*, Ar–H), 2927 (*m*, C–H, aliphatic), 1481 (*s*, C=C), 1393 (*w*, C–O), 1261 (*s*, P–O), 1205 (*s*, P=N), 1159–1110 (*s*, P–O–C), 562 + 530 (*s*, P–Cl). MS (highest peak in multiplet, based on Cl^{35}) (*m*/*z*): 617 (M^+, 18 %), 543 ((M – *tert*-BuNH), 3 %), 279 ((M – $CH_2(OArCl)_2Cl_2$), 15 %).

IR, ^{1}H-NMR, ^{13}C-NMR, ^{31}P-NMR and MS spectroscopy

In the IR spectra, the characteristic P=N, P–O and P–O–C bands with wave numbers 1222 , 1205 (ν(P=N)), 1276, 1261 (ν(P–O)) and 1178–1156–1110, 1159––1110 (ν(P–O–C)) were observed for compounds **3** and **4**, respectively. In addition, the asymmetric and symmetric vibrations of PCl_2 were found at 581 and 562, and 525 and 530 for **3** and **4,** respectively.

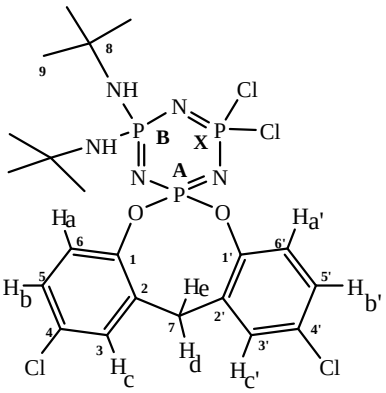
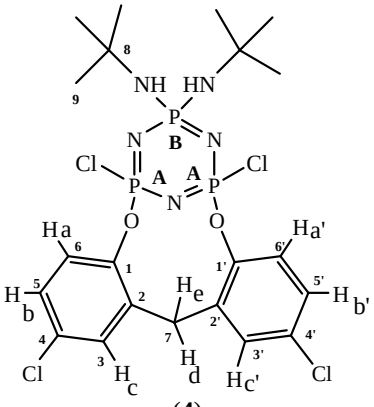
The NMR spectral data for compounds **3** and **4** are presented in Table I. In solution, the structures are symmetrical according to the ^{1}H-NMR and ^{13}C-NMR spectral data. The Ar–CH_2–Ar protons are quartered due to the geminal proton couplings with the P atom ($^{2}J_{\mathbf{HCH}}$ = 13.52 Hz and $^{2}J_{\mathbf{HCH}}$ = 13.73 Hz for **3** and **4**, respectively) and the five bond couplings ($^{5}J_{\mathbf{P}OCCC\mathbf{H}}$ = 2.91 Hz and $^{5}J_{\mathbf{P}OCCC\mathbf{H}}$ = = 2.01 Hz for **3** and **4**, respectively). The difference of these protons in compounds **3** and **4** may be explained by the hydrogen bonding with N atoms on the cyclotriphosphazene ring. According to the crystallographic data, the –CH_2– protons in compound **1** form weak intermolecular hydrogen bonds with nitrogen atoms of the cyclotriphosphazene ring.[5] The protons of each *tert*-butyl group give a singlet (δ 1.38, 1.40 and δ 1.28, 1.59 ppm). The NH signal is broad for compound **3**, while it is a doublet (J = $^{2}J_{\mathbf{PNH}}$ = 5.92 Hz) for compound **4**. The phenyl protons **Haa'** and **Hbb'** are doublets of doublets (6.94 and 7.04 ppm and 7.17 and 7.22 ppm) because of the three and four bond coupling ($^{3}J_{\mathbf{HCCH}}$ = 6.69 and 8.62 Hz, $^{4}J_{\mathbf{HCCCH}}$ = 2.23 and 2.05 Hz for **3** and **4,** respectively). The coupling of **Haa'** with the P atom is $^{4}J_{\mathbf{P}OCC\mathbf{H}}$ = 5.17 and 8.61 Hz for **3** and **4**, respectively. The **Hcc'** protons of compounds **3** and **4** gave a doublet at δ 7.29 and 7.32 ppm, respectively.

According to the proton decoupled ^{13}C-NMR spectra, compounds **3** and **4** have nine signals (Table I). C9 is coupled with the P atom (δ 31.45 ppm, *d*, 6C, $^{3}J_{\mathbf{PNC}}$: 5.0 Hz) in compound **3**, while it is not coupled with the P atom (δ 26.25 ppm, *s*, 6C) in compound **4**.

The proton decoupled ^{31}P-NMR spectra of compounds **3** and **4** were interpreted as a result of a simple ABX and A_2B spin system (Table I). According to the pattern of the proton coupled ^{31}P-NMR spectra of compounds **3** and **4**, it was concluded that only *gem*-diamino architectures are possible.

The electron impact MS spectrum of compounds **3** and **4** showed a well-defined parent ion at *m/z* 617 (100 and 18 %) with the expected isotope pattern. The peaks at *m/z* values of 543 and 279 correspond to the loss of *tert*-BuNH)and $CH_2(OArCl)_2Cl_2$ groups, respectively. The N_3P_3 ring system in **3** and **4** is not stable (the dominant ion was not observed at *m/z* 134) during the fragmentation, which indicates the loss of *tert*-butylamino, phenoxy and chloride fragments. The fragmentation pattern of **3** was similar to that of **4**.

TABLE I. ^{1}H-NMR, ^{13}C-NMR and ^{31}P-NMR spectral data in $CDCl_3$; chemical shifts (δ) are reported in ppm

Atom	Compound	
	 (**3**)	 (**4**)
Haa'	6.94 (2H, *d–d*, $^{3}J_{\mathbf{HH}}$ = 6.69 Hz, $^{4}J_{\mathbf{POArH}}$ = 5.17 Hz)	7.04 (2H, *d–d*, $^{3}J_{\mathbf{HH}}$ = 8.62 Hz, $^{4}J_{\mathbf{POArH}}$ = 8.61 Hz)
Hbb'	7.17 (2H, *d–d*, $^{3}J_{\mathbf{HH}}$ = 6.69 Hz, $^{4}J_{\mathbf{HH}}$ = 2.23 Hz)	7.22 (*d–d*, 2H), ($^{3}J_{\mathbf{HH}}$ = 8.62 Hz, $^{4}J_{\mathbf{HH}}$ = 2.05 Hz)
Hcc'	7.29 (2H, *d*, $^{4}J_{\mathbf{HH}}$ = 2.23 Hz)	7.32 (2H, *d*, $^{4}J_{\mathbf{HH}}$ = 2.05 Hz)
Hd, He	4.01 (2H, *q*, $^{2}J_{\mathbf{HH}}$ = 13.52 Hz, $^{5}J_{\mathbf{POCH}}$ = 2.91Hz)	4.00 (2H, *q*, $^{2}J_{\mathbf{HH}}$ = 13.73 Hz, $^{5}J_{\mathbf{POCH}}$ = 2.01 Hz)
CH_3	1.38 (9H, *s*), 1.40 (9H, *s*)	1.28 (9H, s), 1.59 (9H, *s*)
NH	2.40 (2H, broad)	3.70 (2H, *d*, $^{2}J_{\mathbf{PNH}}$= 5.92 Hz)
C11'	147.21 (*d*, $^{2}J_{\mathbf{POC}}$ = 8.92 Hz)	146.6 (*d*, $^{2}J_{\mathbf{POC}}$ = 7.73 Hz)
C22'	131.02 (*d*, $^{3}J_{\mathbf{POC}}$ = 2.7 Hz)	131.69 (*s*)
C33'	129.94 (*s*)	130.13 (*s*)
C44'	133.57 (*d*, $^{5}J_{\mathbf{POC}}$ = 3.7 Hz)	133.37 (*s*)
C55'	128.54 (*s*)	128.79 (*s*)
C66'	124.04 (*d*, $^{3}J_{\mathbf{POC}}$ = 5.0 Hz)	123.95 (*d*, $^{3}J_{\mathbf{POC}}$ = 4.52 Hz)
C7	51.76 (*s*)	33.24 (*s*)
C8	33.35 (*s*)	29.71 (*s*)
C9	31.45 (*d*, $^{3}J_{\mathbf{PNC}}$ = 5.0 Hz)	26.25 (*s*)
Spin system	ABX	A_2B
$\mathbf{P_A}$	0.76 (1P, *q*, $^{2}J_{\mathbf{PANPB}}$ = 70.49 Hz,	1.65 (2P, *d*, $^{2}J_{\mathbf{PANPB}}$ = 73.22 Hz)
$\mathbf{P_B}$	$^{2}J_{\mathbf{PANPX}}$ = 71.96 Hz)	25.53(1P, *t*, $^{2}J_{\mathbf{PANPB}}$ = 73.22 Hz)
$\mathbf{P_X}$	5.58 (1P, *q*, $^{2}J_{\mathbf{PANPB}}$ = 70.96 Hz, $^{2}J_{\mathbf{PBNPX}}$ =71.15 Hz) 21.65 (1P, *q*, $^{2}J_{\mathbf{PANPX}}$ = 71.96 Hz, $^{2}J_{\mathbf{PBNPX}}$ = 71.15 Hz)	–

X-ray crystallography

The conditions employed for crystal data collection and the parameters of the refinement process are summarized in Table II and selected bond distances

and angles are listed in Table III. The molecular structure with the atom-numbering scheme is shown in Fig. 1.[21] The structure of **3** shows pseudo-mirror symmetry. The mirror plane runs through the atoms N3, P1, C7, as confirmed by the torsion angles. The dihedral angle between the cyclotriphosphazene ring and phenyl ring planes is 58.98(7)°. The endocyclic bond angles (123.9(6)° and 118.5(5)°) for compound **3** are larger than those of the standard compound, $N_3P_3Cl_6$ (121.4(3)° and 118.3(2)°).[22] In compound **3**, the endocyclic angles of the substituents (*tert*-butylamine) that are bonded to the phosphorus atoms, N2A––P2A–N3A, N2B–P2B–N3B are smaller than the other endocyclic angles. The exocyclic N4A–P2A–N5A, N4B–P2B–N5B angles (105.5(4)°; 105.3(6)°) are larger than the other exocyclic angles due to the replacement of the bulky *tert*-butylamino group by Cl atoms. According to the study of Bullen and Tucker,[23] the phosphazene bond lengths are correlated with the electronegativities of exocyclic substituents bound to a particular phosphazene core. The lengths of the P–N bonds depend on the electronegativities of the substituents. In compound **3**, the 2,2'-methylenebis(4-chlorophenoxy) group attached to P1 seems to have a strong electron withdrawing character while the *tert*-butylamino group attached to P2 seems to have a strong electron releasing character. Thus, the lengths of the P–O and P–N bonds are changed considerably. The P–N bond lengths in $N_3P_3R_6$ are generally the same provided that all the substituents R are the same. However when R is a difunctional bulky substituent[24] or contains different substituents, the P–N bonds may show significant variations.[25,26] In the structure **3**, the P–N bond lengths vary between 1.547(4) and 1.632(5) Å due to the influence of the difunctional bulky substituent and the *tert*-butylamino group. According to the earlier report of Allcock *et al.*,[12] the P–O bond lengths are not unusual, however the P1A–O1 (1.542(4)] and the P1B–O2 [1.493(8)) bond lengths are shorter than P–O single bonds of 1.577(2) and 1.61 Å.[5] These lengths are shorter than the P–O single bond (1.61 Å), which suggests some exocyclic delocalization of electrons. The determined P–Cl bond lengths are in good agreement with the expected values.[5,27–29]

The crystal structure is stabilized by a weak intermolecular hydrogen bond and the molecules are stacked *via* weak C–H…π ring interactions in compound **3**.

TABLE II. Crystal data and structure refinements of compound **3**

Formula	$C_{21}H_{28}O_2N_5P_3Cl_4$
Formula weight	617.19
Crystal system	Monoclinic
Space group	P 21/n
Crystal dimension	0.20 x 0.39 x 0.70 mm^3
Temperature collection	296(2) K
Unit cell parameters	a = 10.842(4) Å, b = 9.375(5) Å, β = 99.25(3)°, c = 29.104(11) Å

TABLE II. Continued

V	2920(2) Å^3
Z	4
D_C / g cm^{-3}	1.404
μ(MoKα) / mm^{-1}	0.598
F(000)	1272
$2\theta_{max}$	52.00°
h, k, l Range	$-13 \leq h \leq 13$; $-11 \leq k \leq 11$; $-35 \leq l \leq 35$
No. of measured reflections	41048
No. of independent reflections	5752
No. of observed reflections	3469
Data / restraints / parameters	3469 / 350 / 479
Goodness-of-fit on F^2	0.991
Measurement	STOE IPDS 2
Program system	STOE X-AREA
Structure determination	SHELXS-97
Refinement method	Full-matrix least-squares on F^2
R, R_W ($I > 2\sigma(I)$)	0.0545, 0.1844
$(\Delta\rho)_{max}$–$(\Delta\rho)_{min}$ / e Å^{-3}	0.451–0.524

TABLE III. Some selected bond lengths (Å) and bond angles (°) of compound **3**

Lengths			
Cl3A–P3A 2.001(3)	P2A–N4A 1.575(5)	Cl3B–P3B 1.987(6)	P1B–O1 1.665(6)
Cl4A–P3A 2.019(3)	P2A–N2A 1.591(5)	Cl4B–P3B 2.006(6)	P2B–N2B 1.596(8)
P1A–O1 1.542(4)	P2A–N3A 1.632(5)	P1B–O2 1.493(8)	P2B–N3B 1.619(8)
P1A–N2A 1.561(5)	P2A–N5A 1.654(6)	P1B–N2B 1.559(8)	P2B–N4B 1.685(8)
Angles			
O1–P1A–N2A 105.1(2)		N3B–P2B–N4B 101.9(4)	
N2A–P1A–O2 107.4(2)		C13–O2–P1B 126.8(4)	
O1–P1A–N1A 115.0(2)		N3B–P3B–N1B 122.5(5)	
N2A–P2A–N5A 105.8(3)		C13–O2–P1A 124.8(3)	
O1–P1A–O2 104.5(2)		N3B–P3B–Cl4B 110.3(3)	
N1B–P1B–O1 98.9(3)		P1A–N2A–P2A 123.9(3)	
O2–P1B–O1 104.6(4)		N1B–P3B–Cl4B 105.5(3)	
N2B–P2B–N3B 115.6(5)		P3A–N3A–P2A 122.0(3)	

EXPERIMENTAL

Reagents and techniques

The ^{1}H-, ^{13}C- and ^{31}P-NMR spectra were recorded on a Bruker DPX FT-NMR spectrometer operating at 400, 101.6 and 161.99 (spectral data in $CDCl_3$) MHz, respectively. The ^{1}H- and ^{13}C-NMR chemical shifts were measured using $SiMe_4$ as an internal standard; the ^{31}P-NMR chemical shifts were measured using 85 % H_3PO_4 as an external standard. The infrared absorption spectra were recorded on a Perkin Elmer BX II spectrometer in KBr discs and are reported in cm^{-1} units. The electron impact (70 eV, *ca.* 1.12×10^{-17} J) mass spectra were obtained on a VG-ZAPSPEC spectrometer with an ion source temperature of 240 °C. Carbon, nitrogen and hydrogen analyses were performed on a LECO CHNS-932 analyzer.

The melting points were measured on an Electro Thermal IA 9100 apparatus using a capillary tube. Hexachlorocyclotriphosphazene was purchased from Aldrich. It was recrystallized from hexane and purified by fractional vacuum sublimation at 55 °C before use. Tetrahydrofuran and hexane were purchased from Merck, distilled over sodium/benzophenone and stored over molecular sieves. Sodium (Merck), *tert*-butylamine (Merck), 2,2'-methylenebis(4-chlorophenol) (Aldrich), and silica gel (Aldrich, 70–230 mesh, 60 Å) were used as received. All reactions were monitored using Kieselgel 60 F 254 (silica gel) pre-coated TLC plates. All reactions and manipulations were performed under an atmosphere of dry argon.

*Synthesis of 2,4',4',6',6',10-hexachloro-4',4',6',6'-tetrahydrospiro[12*H*-dibenzo[*d,g*]-[1,3,2]dioxaphosphocin-6,2'λ^5-[1,3,5,2,4,6]triazaphosphorine] (spiro) (**1**) and 2,6,8,8,10,14-hexachloro-8,8-dihydro-6*H*-6λ^5,10λ^5-nitrilo-10*H*,16*H*-dibenzo[*h,k*]-[1,7,3,5,2,4,6]-dioxadiazatriphosphacyclododecine (ansa) (**2**)*

Compounds (**1**) and (**2**) were prepared according to the published procedure.[5]

*Synthesis of 4,4'-bis(*tert*-butylamino)-2,6',6',10-tetrachloro-4',4',6',6'-tetrahydrospyro[12*H*--dibenzo[*d,g*][1,3,2]dioxaphosphocin-6,2'λ^5-[1,3,5,2,4,6]-triazaphosphozine] (spiro) (**3**)*

tert-Butylamine (0.670 g; 9.20×10^{-3} mol) in dry THF (50 mL) was added drop wise to a stirred solution of compound **1** (0.50 g; 9.2×10^{-4} mol) in dry THF (150 mL) at –20 °C over 1 h, with argon flowing over the reaction mixture. Then the mixture was allowed to come to ambient temperature, boiled under reflux (24 h) using a condenser fitted with a $CaCl_2$ drying tube. The precipitated amine hydrochloride was filtered off and the solvent removed by rotary evaporation. The crude product was left to dry under *vacuo* and chromatographed (silica gel, 60 g, eluent; $CHCl_3$/*n*-hexane, 3:1) to give compound **3**. Then, it was recrystallized from $CHCl_3$/petroleum ether (50:70) by the slow diffusion method whereby a white solid formed, yield 0.182 g.

*Synthesis of 8,8-Bis(*tert*-butylamino)-2,6,10,14-tetrachloro-8,8-dihydro-6*H*-6λ^5,10λ^5-nitrilo-10*H*,16*H*-dibenzo[*h,k*][1,7,3,5,2,4,6]-dioxadiazatriphosphacyclododecine (ansa)(**4**)*

tert-Butylamine (0.61 g; 8.30×10^{-3} mol) in dry THF (50 mL) was added drop wise to a stirred solution of compound **2** (0.45 g; 8.30×10^{-4} mol) in dry THF (150 mL) at –20 °C for over 1 h, with argon flowing over the reaction mixture. Compound **4** was prepared and purified as compound **3,** white solid, yield 0.153 g.

Crystal structure

The data collection for **3** was performed on a STOE IPDS-2 diffractometer employing graphite-monochromatized MoKα radiation (λ = 0.71073Å). The collected intensities were corrected for Lorentz and polarization factors; absorption correction was performed (μ = 0.598 mm^{-1}) by the integration method *via* X-RED software [30] and the cell parameters were determined using X-AREA software.[30] The structure was solved using direct methods in the WINGX implementation of SHELXS-97 [31] and refined with SHELXL-97.[32] A total of 479 crystallographic parameters were refined. Throughout the refinement process, the Cl3, Cl4, P1, P2, P3, N1, N2, N3, N4, N5, C14, C15, C16, C17, C18, C19, C20, C21 atoms were treated as a disordered group. The site occupation factors of the disordered atoms were refined to 0.71(2) and 0.29(2). All hydrogen atoms were located geometrically using a riding model. The crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication No. CCDC 644277.[33]

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ИЗВОД

СИНТЕЗА И СПЕКТРОСКОПСКЕ КАРАКТЕРИСТИКЕ ГЕМИНАЛНИХ-БИС(*терц*-БУТИЛАМИНО)ФОСФАЗЕНА ДОБИЈЕНИХ РЕАКЦИЈОМ СПИРО И АНСА ФЕНОКСИФОСФАЗЕНА СА *терц*-БУТИЛАМИНОМ И СТРУКТУРА КРИСТАЛА 4,4'-БИС(*терц*-БУТИЛАМИНО)-2,6',6',10-ТЕТРАХЛОРО-4',4',6',6'-ТЕТРАХИДРОСПИРО[12*H*-ДИБЕНЗО[*d*,*g*][1,3,2]ДИОКСАФОСФОЦИН-6,2'λ^5-[1,3,5,2,4,6]-ТРИАЗАФОСФОРИНА]

DİĞDEM ERDENER[1], MUSTAFA YILDIZ[1], HÜSEYİN ÜNVER[2], NAZAN OCAK İSKELELİ[3] и TAHSİN NURİ DURLU[2]

[1]*Department of Chemistry, Faculty of Science and Arts, Çanakkale Onsekiz Mart University, TR-17100 Çanakkale,* [2]*Department of Physics, Faculty of Science, Ankara University, TR-06100 Tandoğan, Ankara и* [3]*Department of Physics, Faculty of Science and Arts, Ondokuz Mayıs University, TR-55139 Kurupelit, Samsun, Turkey*

Реакција кондензације супституисаних спиро и анса феноксициклотрифосфазена **1** и **2** са *терц*-бутиламином даје дисупституисане геминалне-бис(*терц*-бутиламино)феноксициклотрифосфазенске деривате (**3** и **4**). Структура ових једињења је окарактерисана елементалном анализом, IC, ^{1}H-, ^{13}C-, ^{31}P-NMR спектроскопијом и масеном спектрометријом. Структура једињења **3** испитана је и кристалографском анализом и утврђено је да једињење кристалише у моноклиничној просторној групи $P2_1/n$ са јединичним параметрима: a = = 10,842(4), b = 9,375(5), c = 29,104(11) Å, β = 99,25(3)°, V = 2920(2) Å^3, D_x = 1,404 g cm^{-3}.

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33. Further information may be obtained from: Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB21EZ, UK, by quoting the depository number CCDC 644277 (E-mail: deposit@ccdc.cam.ac.uk).