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Note

# A growing family: New structures of coordination polymers containing adamantane-shaped phosphorus-nitrogen cage ligands

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Dedicated to Arnold L. Rheingold

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#### 1. Introduction

Phosphorus(III)-nitrogen compounds (phosphazanes) are well known as effective P-donor ligands in coordination chemistry and host-guest chemistry. Recent work has shown that P-N compounds also display interesting macrocyclic structures and form extended structures with metal ions to make their own types of coordination polymers and networks [1-5]. A long-known reaction of phosphorus trichloride with methylamine yields the adamantane shaped cage molecule shown in Scheme 1. As simple as this reaction seems, the family of P<sub>4</sub>(NR)<sub>6</sub> derivatives that have been isolated remains very small. Only two (methyl and isopropyl) derivatives of this cage have been structurally characterized [6,7]. The R = ethyl derivative has been observed by  ${}^{1}H{}^{31}P$  NMR, but not structurally characterized [8]. The typical preparation shown in Scheme 1 uses excess amine to absorb the hydrogen chloride evolved during the reaction. This can result in a large amount of amine hydrochloride, mixed with a small amount of the desired product. As the alkyl chains are extended (n-propyl...) the hydrochloride salt by-products have significant solubility in organic solvents. This complicates the workup considerably. Still worse, the reaction no longer forms the desired cage exclusively, but a mixture dominated by RN=P(-NHR)<sub>2</sub> and P(-NHR)<sub>3</sub> [9,10]. A more elaborate preparation is reported for the isopropyl analogue [11].

This report details the synthesis of a new member of the P<sub>4</sub>(NR)<sub>6</sub> family according to a two-step, one-pot preparation meth-

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### ABSTRACT

Two new structurally characterized coordination polymers containing the  $P_4(NR)_6$  ligand system are described. A convenient one-pot synthesis of  $P_4(NR)_6$  (R = benzyl) via reaction of lithiated primary amine with phosphorus trichloride demonstrates an expanded scope for the preparation of this adamantanetype structure. Reactions of  $P_4(NR)_6$  (R = Et, Bn) with cuprous iodide yield different products due to the differences in steric demands of the ligands.

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od shown in Scheme 2. This adaptation of synthetic routes used to build some phosphazane macrocycles minimizes the amount of amine required, and eliminates the presence of long-chain hydrochloride salts in the workup [12]. The first step of the process uses lithiated amine both as the nucleophile to form P-N bonds, and the base to neutralize liberated HCl. Stoichiometry prevents all P-Cl bonds from reacting, and allows the formation of the precursors (RNH-PCl<sub>2</sub>), (RNPCl)<sub>2</sub> and (RNPCl)<sub>3</sub>. Later addition of triethylamine liberates the remaining amine from the hydrochloride salt to complete cyclization to P<sub>4</sub>(NR)<sub>6</sub>.

As part of the current effort to explore the structure and coordination chemistry of these ligands, X-ray crystallographic studies of two new P<sub>4</sub>(NR)<sub>6</sub>/CuI coordination compounds are reported. Coordination complexes prepared from the new  $P_4(NR)_6$  ligands show the tunability of this system with respect to steric crowding around the P donor atoms. The core of this ligand can accommodate moderately bulky side chains that may be useful in dictating the chemical and physical properties of complexes formed by this cage ligand. The work below demonstrates one such case.

### 2. Experimental

General considerations: All reactions were carried out under nitrogen atmosphere, either using Schlenk techniques, or working in a nitrogen glovebox. Tetrahydrofuran, toluene and hexanes were all distilled from Na/benzophenone before use. Anhydrous diethyl ether and acetonitrile were used as purchased after storing over molecular sieves. Anhydrous ethylamine was purchased from Sigma-Aldrich and used as purchased. Higher amines were dried over potassium hydroxide or calcium hydride and distilled before use. Proton and phosphorus NMR spectra were acquired on a Bruker

Present address: Bruker-AXS Inc., 5465 E. Cheryl Parkway, Madison, WI 57311, USA.

Avance 300 NMR system (121.5 MHz for  $^{31}$ P) in d8 toluene, and referenced to toluene or 85% H<sub>3</sub>PO<sub>4</sub> external standard, respectively.

#### 2.1. Preparation of 1a, method 1

Phosphorus trichloride (15.0 mL, 0.175 mol) was added dropwise with mechanical stirring to 70.0 mL (1.06 mol) of ethylamine at -60 °C. The yellow/white reaction mixture was allowed to warm to room temperature. The reaction mixture was then heated to 150 °C to a give a yellow liquid. Cooling the mixture left an amber oily liquid that formed a paste upon standing. Hexanes (3 × 50 mL) were added to extract the product. Filtration and removal of solvent under vacuum yielded a colorless oil that solidifies on standing. (<sup>31</sup>P NMR, 79.5 ppm). Yield: 3.812 g (26.6%). *Anal.* Calc. for: C, 37.7.0; H, 7.91; N, 21.99. Found: C, 37.97; H, 7.48; N, 21.77%..

#### 2.2. Preparation of 1b, method 2

A 250 mL roundbottom flask equipped with a stir bar and rubber septum was charged with 40 mL of tetrahydrofuran, and 1.88 mL (17.2 mmol) of benzylamine. After cooling the solution to 0 °C, 10.8 mL of 1.6 M n-butyllithium were added by syringe. The resulting pink slurry was allowed to warm to room temperature over 1 h. A second round bottom flask containing 20 mL of tetrahydrofuran and 1.0 mL (11.5 mmol) of phosphorus trichloride was cooled to -60 °C in a dry ice/2-propanol bath, and treated dropwise with the lithiated amine. The temperature was maintained for 7 h before the reaction was allowed to warm to 20 °C for an additional 12 h. A syringe was used to add 1.00 mL of triethylamine and the reaction stirred for an additional 24-36 h. A second portion of triethylamine was added and the reaction stirred for another 72 h. Solvent was removed under vacuum and the resulting white paste suspended in dry toluene. Filtration and removal of solvent resulted in a yellow oil. Column chromatography through silica gel, followed by removal of solvent under vacuum vields a colorless oil (<sup>31</sup>P NMR. 80.9 ppm). Yield: 0.69 g. 32%. The ligand was typically used immediately after preparation. Elemental analyses were low for C and N. This was attributed to the possible oxidation during handling. Anal. Calc. for: C, 66.84; H, 5.61; N, 11.14; Found: C, 63.51; H, 5.66; N, 10.06%.

#### 2.3. Preparation of **2**, $[P_4(NEt)_6CuI]_n$

Cuprous iodide (0.091 g, 0.48 mmol) was dissolved in 10 mL of dry acetonitrile and added dropwise to a solution of 0.353 g (0.92 mmol) of **1a** in 15 mL acetonitrile. A white precipitate formed



**Scheme 1.** A simple preparation of the  $P_4(NR)_6$  cage (method 1) that only works well for  $R = CH_3$  and  $C_2H_5$ .

immediately upon addition, then disappeared with stirring. Colorless crystals formed after 2 days. Suction filtration isolated 0.155 g of product (57%). *Anal.* Calc. for: C, 25.16; H, 5.28; N, 14.68; Cu, 10.4; P, 21.6. Found: C, 25.84; H, 4.75; N, 14.63; Cu, 11.1; P, 20.6%.

#### 2.4. Preparation of **3**, $[P_4(NBn)_6(CuI)_2(CH_3CN)_2 \cdot CH_3CN]_n$

A solution of cuprous iodide (0.126 g, 0.662 mmol) in 6 mL of acetonitrile was added to a solution of ligand **1b** (0.451 g, 0.598 mmol) in 10 mL of acetonitrile. X-ray quality crystals of product grew after standing for 3 days. The supernatant was removed by pipet, and the crystals washed with  $3 \times 1$  mL of acetonitrile. Yield: 0.130 g, 30% based on Cu. The crystals of **3** desolvate rapidly, giving a white powder with composition consistent with  $P_4(NBn)_6(Cul)_2(CH_3CN)_2$ . Anal. Calc. for: C, 45.37; H, 3.97; N, 9.20; Cu, 10.44; P, 10.17. Found: C, 46.12; H, 3.75; N, 8.55; Cu, 10.44; P, 10.74%.

#### 2.5. X-ray Crystallography

Crystals were handled under oil and the specimen crystals were affixed to the aperture of a MiTeGen MicroMount mounted to a tapered copper pin. This assembly was transferred to the goniometer of a Bruker CCD diffractometer equipped with an Oxford Cryosystems low-temperature apparatus operating at 100 K (200 K for **3**). The unit cell and orientation matrix were determined using reflections harvested from three sets of 12  $0.5^{\circ}$   $\phi$  scans, such that  $I > 20\sigma(I)$ . Final unit cell parameters were determined using reflections harvested from each entire data set. All data were corrected for Lorenz and polarization effects, as well as for absorption. Structure solution and refinement utilized the SHELXTL software package as implemented in the APEX2 software suite [13]. Figures were con-

able 1									
ummary	of	crystal	data	for	com	pounds	<b>2</b> ar	ıd	3.

Compound	2	3
CSD#	775 613	775 614
Formula	$C_{24}H_{60}Cu_2I_2N_{12}P_8$	$C_{48}H_{51}Cu_2I_2N_9P_4$
Fw	1145.48	1258.74
Space group	$P2_1/n$	$P2_1/n$
a (Å)	11.4755(4)	12.9771(12)
b (Å)	34.0911(12)	19.0563(16)
<i>c</i> (Å)	11.9048(4)	21.011(2)
α (°)	90	90
β(°)	111.712(2)	92.626(3)
γ (°)	90	90
$V(Å^3)$	4326.9(3)	5190.5(8)
Ζ	4	4
$\rho_{\rm calc}  ({\rm g/cm^{-3}})$	1.758	1.611
F(0 0 0)	2288	2504
$\mu$ (Mo K $\alpha$ ) (mm <sup>-1</sup> )	2.741	2.175
λ (Å)	0.71073	0.71073
Temperature (°C)	-173	-73
R; wR	0.0469; 0.1111	0.0406; 0.1164
Goodness-of-fit (GOF)	1.305	1.044



Scheme 2. Reaction to prepare P<sub>4</sub>(NR)<sub>6</sub> (R = benzyl) (method 2). {<sup>1</sup>H}<sup>31</sup>P NMR spectra of reaction mixture are included in supplementary material.

structed and rendered using ORTEP-3 for Windows [14] and POV-RAY for Windows 3.6 [15]. A summary of crystallographic data for all compounds is provided in Table 1 and details of the crystallography are provided in Supplementary material.

 Table 2

 Selected bond lengths and angles for compounds 2 and 3.

2		3	
Cu1-P5	2.2465(8)	Cu1-N7	1.998(4)
Cu1-P1	2.2508(8)	Cu1-P1	2.2170(13)
Cu1-I1	2.5336(4)	Cu1–I1(i)	2.6567(7)
Cu2-P7	2.2406(8)	Cu1-I1	2.6922(7)
Cu2-P3(i)	2.2451(8)	Cu2-N8	2.010(5)
Cu2–I2	2.5268(4)	Cu2-P2	2.2117(13)
		Cu2-I2(ii)	2.6346(8)
		Cu2–I2	2.6857(7)
P5-Cu1-P1	124.79(3)	N7-Cu1-P1	116.24(13)
P5-Cu1-I1	117.65(3)	N7-Cu1-I1(i)	104.08(12)
P1-Cu1-I1	117.53(2)	P1-Cu1-I1(i)	118.81(4)
P7-Cu2-P3	127.17(3)	N7-Cu1-I1	102.75(13)
P7-Cu2-I2	119.06(2)	P1-Cu1-I1	106.05(4)
P3-Cu2-I2	113.72(2)	I1-Cu1-I1(i)	107.52(2)
		N8-Cu2-P2	119.12(13)
		N8-Cu2-I2	106.12(14)
		P2-Cu2-I2	112.56(4)
		N8-Cu2-I2	102.77(13)
		P2-Cu2-I2	115.37(4)
		I2-Cu2-I2(ii)	98.43(2)
		Cu1-I1-Cu1(i)	72.48(2)
		Cu2-I2-Cu2(ii)	81.57(2)



**Fig. 1.** Thermal ellipsoid plot of the asymmetric unit of **2**. Ellipsoids drawn at 50% probability. Hydrogen atoms are removed for clarity.

#### 3. Results and discussion

#### 3.1. New ligands

The preparation of  $P_4(NBn)_6$  can be achieved by reaction of the lithiated amine with phosphorus trichloride. When phosphorus trichloride is treated with a suspension of lithiated primary amine in a ratio of 2–3, the reaction initially forms a mixture of BnNHPCl<sub>2</sub> (doublet  $\delta$  158 ppm, I = 50 Hz) and trimeric (BnNPCl)<sub>3</sub> ( $\delta$  134, 125 and 104 ppm) and  $(BnNPCl)_2$  ( $\delta$  227) [16,17]. Treatment of this mixture with triethylamine deprotonates the hydrochloride byproduct from the first step to complete the slow formation of the  $P_4(NR)_6$  structure. Monitoring of the reaction by <sup>31</sup>P NMR shows that the reaction progresses through a complicated series of intermediates that collapse to the appropriate resonance near 80 ppm. Numerous unidentified intermediates are evident, with resonances that are not consistent with previously suggested precursors to the  $P_4(NR)_6$  cage. It is noteworthy that yields and purity of products from this reaction are best when there is a slight excess of PCl<sub>3</sub>. This is easily achieved by using slightly less than the reguired amount of butyllithium and triethylamine. It is not clear from this study what route is taken to reach the  $P_4(NR)_6$  structure. The <sup>31</sup>P NMR spectra of reaction mixtures show resonances appropriate for the coexistence of multiple intermediates, with no clear distinction of whether dimer, trimer, or both are the direct precursor of P<sub>4</sub>(NBn)<sub>6</sub>. This aspect of the experiment warrants its own detailed study.

# 3.2. Preparation and structural characterization of Cul coordination polymers

All crystals were grown from acetonitrile reaction mixtures. Bond lengths and angles for the Cu coordination sphere, shown in Table 2, and  $P_4(NR)_6$  cage are consistent with other known structures. A slight shortening of P–N bond lengths is noted when P is bound to copper. Some significant lengthening of P–N bond distances is noted in **3**.

#### 3.2.1. Structure of **2**, $[P_4(NEt)_6CuI]_n$

Compound **2** crystallizes in the monoclinic space group  $P2_1/n$ . The asymmetric unit contains two cage ligands and two trigonal planar copper ions as shown in Fig. 1. Both copper ions are coordinated by two P donor atoms and a terminal iodide. The polymer chains run parallel to the *a*-axis of the unit cell as shown in Fig. 2. One ethyl group, attached to N1, shows evenly distributed positional disorder over two sites. Calculated cone angles for  $P_4(NEt)_6$  were 157° for P atoms bound to Cu [18]. The P–N bonds within the cage fall in a normal range for those in previously determined structures with an average for **2** of 1.708 Å. This suggests lit-



Fig. 2. Polymer segment of 2 along *a*-axis. Symmetry operations: (i) *x*, *y*, *z* – 1 (ii) *x*, *y*, *z* + 1. Thermal ellipsoids drawn at 50% probability. H atoms removed for clarity.



**Fig. 3.** Asymmetric unit of **3**. Thermal ellipsoids of Cu, I, N and P drawn at 50% probability. Benzyl group carbons drawn as stick models for clarity. Hydrogen atoms removed for clarity.

tle change to the ligand core as a result of larger R groups. The N atoms in the cage ligands are generally close to planar. The average for the sum of angles around N is  $354^\circ$ , with a minimum value of  $346^\circ$ .

#### 3.2.2. Structure of **3**, $[P_4(NBn)_6(CuI)_2(CH_3CN)_2 \cdot CH_3CN]_n$

Crystals of **3** grow in the monoclinic space group  $P2_1/n$  from a solution of hot acetonitrile. Fig. 3 shows that the asymmetric unit consists of one cage ligand bridging two metal centers. The benzyl group attached to N3 occupies two sites with relative occupancies

of about 2:1. Adjacent to the disordered benzyl group is one uncoordinated acetonitrile solvent, also disordered over two orientations. The tetrahedral coordination environment of Cu is completed by a bound acetonitrile as the fourth ligand. Steric crowding near P prevents multiple cage ligands from binding Cu. As a consequence, the 1:1 reaction of  $P_4(NBn)_6$  and CuI produces a linear coordination polymer that propagates by formation of a Cu<sub>2</sub>I<sub>2</sub> dimer as shown in Fig. 4. The Cu-Cu distances of 3.146 and 3.476 Å for Cu1 and Cu2, respectively, suggest no Cu-Cu bonding is present. The Cu–P distances are comparable to other  $P_4(NR)_6$ complexes. The P-N bond lengths and angles in the core of the ligand also display a typical distribution of values, with the exception of the P3–N3 and P4–N5 bonds. These bonds undergo significant distortion, deviating from the average P-N bond distance by 0.047 and 0.026 Å, respectively. The N atoms of the cage are generally planar, with the greatest deviations seen for N3 and N5 where the sums of angles were 346° and 350°, respectively.

The presence of benzyl groups on the P<sub>4</sub>N<sub>6</sub> core significantly increases steric crowding. Calculated estimates of the cone angles for P1 and P2 are 185° and 179°, respectively. Such crowding serves to limit to one the number of ligands that may fit around a metal center. Fig. 4 also shows how the benzyl groups near P1 and P2 are oriented away to allow coordination of the copper atoms and form the pocket that accommodates the acetonitrile ligand. While intra-chain  $\pi$ - $\pi$  interactions between benzyl groups and bound acetonitrile are observable, there is little evidence for  $\pi$ -stacking between phenyl rings - either within or between chains. The close proximity of the benzyl groups around the cage requires that if any one benzyl group changes its conformation, at least one other must move to accommodate it. Estimates of the steric crowding at P3 and P4 were calculated by placing dummy atoms at a distance of 2.24 Å from P and computing cone angles as for P-Cu bonds. The estimated values for these cone angles were 215° and 211°, respectively. The contrast in cone angles for structures 2 and 3 is shown in Table 3. Such crowding coincides with the distortion in the core structure of the ligand. The lengthening of the P3-N3 and P4-N5 bonds coincides with the closest approach of benzyl groups to P3 and P4 atoms.

Without P–Cu–P links, compound **3** is moderately soluble. It can be recrystallized from hot acetonitrile with minimal decomposi-



**Fig. 4.** Polymer segment of **3.** Symmetry operations: (i) -x + 2, -y + 2, -z + 2 (ii) -x + 2, -y + 1, -z + 2. Thermal ellipsoids drawn at 50% probability. Hydrogens removed and benzyl groups shown as ball and stick for clarity.

Table 3	
Summary of cone angles for coordinated and	non-coordinated P atoms in ${\bf 2}$ and ${\bf 3}$

Compound, R	Donor	Cone angle	Cone angle
	atoms	P–Cu–H° <sup>a</sup>	P-Dummy-H° <sup>b</sup>
<b>2</b> , C <sub>2</sub> H <sub>5</sub>	2P, I <sup>−</sup>	157	160
<b>3</b> , CH <sub>2</sub> (C <sub>6</sub> H <sub>5</sub> )	P, 2I <sup>−</sup> , CH <sub>3</sub> C <b>N</b>	182	213

<sup>a</sup> Calculated from crystals structure data based on P-Cu-H<sub>phenyl</sub> (on ligand) angle (Ref. [18]).

<sup>b</sup> Dummy atom placed at 2.24 Å and angle computed identical to P-donor atoms.

tion. Crystals grown in such a manner had identical structure to those originally isolated from the reaction mixture. The steric crowding of the non-coordinated P atoms may not only limit the number of ligands that can bind to a cuprous ion, but also prevent the  $P_4(NBn)_6$  ligand from binding more than two metal ions.

#### 4. Conclusion

Reactions of closo-tetraphosphorushexaalkylimide ligands  $P_4(NR)_6$  (R = ethyl, benzyl) with cuprous iodide has afforded two new structures, expanding the group of structurally characterized examples to four. The structural evidence in  $[P_4(NEt)_6Cul]_n$  (2) suggests that extending the length of the primary alkyl group does not affect the structure of the  $P_4N_6$  core. By modification of a well-known reaction to form P–N clusters and cages, the preparation of the sterically hindered  $P_4(NBn)_6$  cage ligand can be achieved by a simple process in moderate yield. The structural characterization of the Cul complex **3** shows that this ligand can bind multiple metal ions. But connectivity of **3** differs from **2** and  $[P_4(NCH_3)_6Cul]_n$  [4], in part, due to congestion around the  $P_4N_6$  core. Such observations suggest a way to control the connectivity of coordination polymers and networks that might be formed with this family of ligands.

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#### **Appendix A. Supplementary material**

CCDC 775613 and 775614 contain the supplementary crystallographic data for compounds **2** and **3** respectively. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/ retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk X-ray. NMR spectra from reaction are also supplied as supplemental figures. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.ica.2010.08.052.

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