spectroscopic studies and find no evidence for water,[15] the characteristic stretching and bending frequencies being absent. The nature of hydrogen in these solids needs to be further investigated.

We have carried out electrical resistivity measurements of $LaMo_6S_8$ and $PbMo_6S_8$ in order to see whether the method of preparation reported here has any effect on the superconducting critical transition temperature (T_c). In Figure 3, we show the variation of resistivity with temperature of these two compounds. We see that zero resistance is attained at 6.79 and 14.05 K for $LaMo_6S_8$ and $PbMo_6S_8$, respectively. The literature values of transition tempertures for these two solids[16,17] prepared by the ceramic method are 4.74–6.76 and 9.5–15.2 K, respectively, depending on the stoichiometry and homogenity of the samples. The T_c values of the present sample suggest that they are nearly stoichiometric. It is clear that the method reported by us has no adverse effect on the T_c. It is particulary noteworthy that single-phase $LaMo_6S_8$ was prepared by us at 1000 °C, while the method reported recently by Tsunekawa et al.[16] requires temperatures as high as 1500 °C.

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Synthesis of a Novel Tetrameric Palladium(II) Complex Having Bridging Chlorides and Corner-Spanning Methylenebis(phosphinic acidate) Ligands

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The tetrakis(μ-pyrophosphito)platinate(II) complex $Pt_2(pop)_4^{4-}$ (pop = μ-$P_2O_5H_2^{2-}$) continues to attract interest because of its unique photophysical properties and photochemical reactivity.[2,3] More recently an analogue diplatinate(II) complex $Pt_2(pcp)_4^{4-}$ (pcp = μ-$P_2CO_4H_4^{2-}$) has been prepared, and its preliminary photophysical and structural properties have been described.[4,5] All previous attempts to prepare the palladium analogue complexes with the pop ligands have failed because the synthetic routes from phosphorous acid cause rapid reduction of the added palladium salts to the free palladium metal. Aqueous solutions of Na_2PdCl_4 and $CH_2(PH(O)OH)_2$ ($pcpH_2$) under all pH conditions also give palladium metal as the final product, but in methanol solvent this reduction does not occur, and we have isolated the first pcp palladium complex.

Table I. Crystal, Data Collection, and Refinement Parameters for **1**

formula	$Pd_4Cl_4P_8Na_2C_4H_{34}O_{24}$
cryst system	monoclinic
space group	$P2_1/n$
a, Å	8.5390 (10)
b, Å	15.713 (2)
c, Å	13.857 (2)
β, deg	106.60 (1)
V, Å^3	1781.7 (8)
D(calcd), g cm^{-3}	2.474
Z	2
temp, °C	26
cryst dimens, mm	0.06 × 0.24 × 0.40
radiation	graphite-monochromated Mo Kα (λ = 0.71073 Å)
diffractometer	Enraf-Nonius CAD4
transmission (max/min)	99.66/76.56
θ limits, deg	1–25
decay, %	0
no. of unique data	3127
no. of unique data with $F_o^2 > 3\sigma(F_o^2)$	2660
GOF	2.205
R, %	2.8
R_w, %	3.9

Experimental Section

Sodium tetrachloropalladate was purchased from Mathey Bishop Inc. Methylenebis(phosphinic acid) was prepared by the literature procedure.[5] Infrared spectra were measured as Nujol mulls on a Perkin-Elmer Model 683 spectrometer. Far-infrared spectra were measured as Vaseline mulls on a Mattson Cygnus 100 FTIR spectrometer.

Sodium Tetrakis(μ-chloro)bis(μ-methylenebis(phosphinic acidato)(2–))bis(μ-methylenebis(phosphinic acidato)(1–))tetrapalladate(II), $Na_2[Pd_4(\mu\text{-}Cl)_4(pcp)_2(pcpH)_2]$. To a 10-mL round-bottom flask was added sodium tetrachloropalladate trihydrate (50 mg, 0.14 mmol), methylenebis(phosphinic acid) (41 mg, 0.28 mmol), and methyl alcohol (1 mL). The solution was heated in an oil bath at 53 °C for 1 h. A precipitate of sodium chloride formed as the brown solution changed color through yellow to almost colorless. The solvent was removed either on a rotary evaporator or by passage of a stream of nitrogen gas. The residue was dissolved in 3–5 mL of aqueous sodium chloride (1 M), and the flask was stoppered. After several hours a pale yellow compound began to precipitate. After 7–10 days the supernatant liquid was removed by pipette, and the residual solid was washed with water (2 × 5 mL). The solid product was dried in vacuo to give the complex as the octahydrate. Yield: 20 mg (10%). Extended heating of the reaction mixture gave a different unidentified compound that shows a red emission at 77 K.

Crystals of $Na_2[Pd_4Cl_4(CH_2PO_4H_2)_2(CH_2PO_4H_3)_2]\cdot 8H_2O$ suitable for x-ray diffraction were obtained directly from the synthetic procedure. A crystal was placed in a capillary tube for data collection. Data were collected on an Enraf-Nonius CAD4 diffractometer equipped with Mo Kα radiation and a graphite monochromator by $\omega/2\theta$ scans of variable rate designed to yield $\sigma(I) = 0.02I$ for all observable data. A maximum of 120 s was used for the scan time of weak reflections. The data were corrected for Lp, background, and absorption effects. The absorption correction was based on ψ scans of reflections near $\chi = 90°$. Data having $I > 3\sigma(I)$ were used in the refinement. Crystal details are collected in Table I. The structure was initially solved by heavy-atom techniques. The tetramer lies on a center of symmetry in $P2_1/n$, and the crystals contain eight molecules of water per tetramer. The charge balance was accommodated by a model where both oxygen atoms on P(1) are protonated. All H atoms were located, but they did not successfully refine; in the final model only the hydrogens on CH_2 were refined. The oxygens O(1)–O(5) are linked by a hydrogen bond; O(2) donates a hydrogen bond to a neighboring tetramer (O(2)–O(3)′ = 2.651 (3) Å).

The crystal has a slight disorder where the entire anion is rotated by 90° without greatly affecting the P and Cl atom positions. This happens for approximately 4% of the molecules. Refinement was by full-matrix least squares with weights $w = \sigma^{-2}(F_o)$. Final residuals are given in Table I. Non-hydrogen atomic positions are given in Table II, and selected bond distances and angles are collected in Table III.

Results and Discussion

The colorless residue from the reaction of $PdCl_4^{2-}$ and $CH_2(PH(O)OH)_2$ in methanol gives, on heating in aqueous sodium chloride solution and allowing the mixture to stand for a few days, yellow crystals of a new complex. This insoluble complex shows

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Table II. Coordinates for Pd Tetramer **1**

atom[a]	*x*	*y*	*z*
Pd(1)*	0.44936 (4)	0.35937 (2)	0.91850 (3)
Pd(2)*	0.30979 (4)	0.57073 (2)	0.86220 (3)
Pd(1)′*	0.72930 (11)	0.4201 (6)	0.9851 (7)
Pd(2)′*	0.44213 (11)	0.3831 (6)	1.08152 (7)
Cl(1)	0.2156 (1)	0.44280 (8)	0.92804 (10)
Cl(2)	0.5163 (1)	0.47827 (8)	0.82479 (9)
P(1)	0.6691 (1)	0.28889 (8)	0.90505 (9)
P(2)	0.1092 (1)	0.64668 (8)	0.89426 (9)
P(3)	0.3724 (1)	0.25649 (8)	1.00561 (9)
P(4)	0.4053 (1)	0.67920 (8)	0.79188 (9)
Na(1)	0.0000 (3)	0.0267 (1)	0.8769 (2)
O(1)*	0.6908 (4)	0.1956 (2)	0.9429 (3)
O(2)*	0.6820 (4)	0.2917 (2)	0.7938 (2)
O(3)*	0.9245 (4)	0.2630 (2)	1.1503 (3)
O(4)*	1.0541 (4)	0.4055 (2)	1.1335 (3)
O(5)*	0.4671 (4)	0.1704 (2)	1.0165 (3)
O(6)*	0.1870 (4)	0.2359 (2)	0.9654 (2)
O(7)*	0.3119 (4)	0.7660 (2)	0.7809 (3)
O(8)*	0.4247 (4)	0.6541 (2)	0.6888 (2)
O(1W)	0.0045 (5)	0.1020 (3)	0.0359 (3)
O(2W)	0.2073 (5)	0.4444 (2)	0.3215 (3)
O(3W)	0.2133 (5)	0.5065 (3)	0.5554 (4)
O(4W)	0.5346 (6)	0.0644 (3)	0.1868 (4)
C(1)*	0.8527 (6)	0.3451 (3)	0.9695 (4)
C(2)*	0.3899 (6)	0.2941 (3)	1.1316 (3)

[a] Asterisk denotes a 0.96 population; star denotes a 0.04 population.

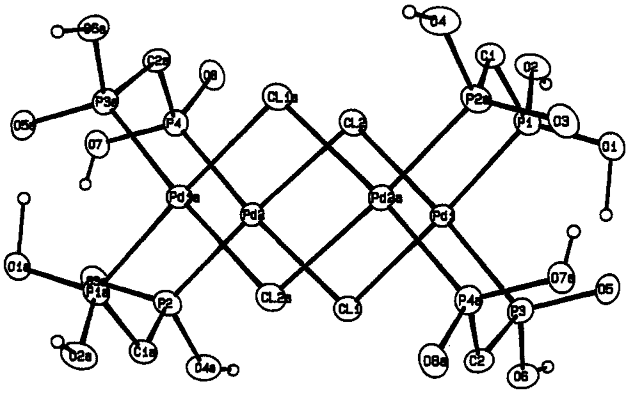

Figure 1. ORTEP drawing of the $[Pd_4Cl_4(P_2CH_4O_4)_2(P_2CH_5O_4)_2]^{2-}$ complex anion.

absorption bands due to $\nu(P—O)$, $\nu(P═O)$, and $\nu(Pd—Cl)$ at 935, 1175, and 250 cm^{-1}, respectively, and a tetrameric structure **1** by X-ray crystallography (eq 1). The complex is formed by

$$4Na_2PdCl_4 + 4pcpH_2 \rightarrow \underset{\mathbf{1}}{Na_2[Pd_4(\mu\text{-}Cl)_4(\mu\text{-}pcp)_2(\mu\text{-}pcpH)_2]} + 6HCl + 6NaCl \quad (1)$$

partial substitution of the chlorides in $PdCl_4^{2-}$ by the pcp ligand. This complex is one of the few structurally characterized palladium tetramers and is the first isolated palladium complex with a pop-type ligand.[6]

Table III. Selected Bond Distances and Angles

Distances (Å)			
Pd(1)–Pd(2)	3.539 (1)	Pd(1)–P(1)	2.232 (1)
Pd(1)–Pd(2)′	3.332 (1)	Pd(1)–P(3)	2.226 (1)
Pd(1)–Cl(1)	2.423 (1)	Pd(2)–P(2)	2.234 (1)
Pd(1)–Cl(2)	2.435 (1)	Pd(2)–P(4)	2.230 (1)
Pd(2)–Cl(1)	2.437 (1)	P(1)–C(1)	1.798 (3)
Pd(2)–Cl(2)	2.452 (1)	P(2)–C(1)	1.827 (4)
P(3)–C(2)	1.809 (3)	P(4)–C(2)	1.816 (3)
P(1)–O(1)	1.550 (3)	P(3)–O(5)	1.561 (3)
P(1)–O(2)	1.577 (2)	P(3)–O(6)	1.554 (2)
P(2)–O(3)	1.542 (3)	P(4)–O(7)	1.565 (2)
P(2)–O(4)	1.568 (2)	P(4)–O(8)	1.536 (2)
Angles (deg)			
Pd(1)–Pd(2)–Pd(1)	91.55 (1)	Pd(2)–Pd(1)–Pd(2)	88.45 (1)
Cl(1)–Pd(1)–Cl(2)	86.37 (3)	Cl(2)–Pd(1)–P(1)	90.29 (3)
Cl(1)–Pd(1)–P(1)	176.65 (3)	Cl(2)–Pd(1)–P(3)	175.66 (3)
Cl(1)–Pd(1)–P(3)	89.30 (3)	P(1)–Pd(1)–P(3)	94.04 (3)
Cl(1)–Pd(2)–Cl(2)	85.68 (3)	Cl(2)–Pd(2)–P(2)	175.76 (3)
Cl(1)–Pd(2)–P(2)	90.71 (3)	Cl(2)–Pd(2)–P(4)	89.18 (3)
Cl(1)–Pd(2)–P(4)	174.23 (3)	P(2)–Pd(2)–P(4)	94.32 (3)
Pd(1)–Cl(1)–Pd(2)	93.48 (3)	Pd(1)–Cl(2)–Pd(2)	92.82 (3)
P(1)–C(1)–P(2)	114.86 (19)	P(3)–C(2)–P(4)	116.06 (19)
O(1)–P(1)–O(2)	108.78 (14)	O(5)–P(3)–O(6)	107.49 (14)
O(1)–P(1)–C(1)	107.05 (16)	O(5)–P(3)–C(2)	106.97 (16)
O(2)–P(1)–C(1)	100.19 (15)	O(6)–P(3)–C(2)	102.09 (15)
O(3)–P(2)–O(4)	109.40 (14)	O(7)–P(4)–O(8)	108.70 (14)
O(3)–P(2)–C(1)	107.85 (16)	O(7)–P(4)–C(2)	103.55 (16)
O(4)–P(2)–C(1)	100.03 (15)	O(8)–P(4)–C(2)	105.07 (15)

The anion of **1** shows a planar arrangement of palladium atoms slightly distorted toward a diamond geometry (Pd(1)–Pd(2)–Pd(1) = 91.55 (1)°, Pd(2)–Pd(1)–Pd(2) = 88.45 (1)°) with bridging chlorides and corner-spanning methylenebis(phosphinic acidate) ligands (Figure 1). The nonbonded palladiums are separated by 3.539 (1) Å across the Pd_2Cl_2 bridge (Pd(1)–Pd(2)) and by 3.332 (1) Å across the $Pd_2(pcp)_2$ bridge (Pd(1)–Pd(2)′). The Pd–Cl distances 2.437 [12] (1) Å are long due to the strong trans influence of the pcp ligand. The unexpected stoichiometry as a disodium salt rather than a tetrasodium salt results from two of the corner-spanning ligands being doubly deprotonated $(CH_2(P(O)OH)_2^{2-})$ and two being singly deprotonated $(CH_2(P(O)OH)(P(OH)_2)^-)$ anions, where P(1) in the latter ligand has both oxygens protonated. This is the first example of unsymmetrical protonation of pop or pcp ligands in a complex, although the situation has been previously observed for platinum(III) complexes that have bridging phosphate ligands.[7]

The Pd–Pd distances in complex **1** are intermediate between those previously found in palladium tetramers. In one group of complexes there are organic ligands directly between the palladiums, and consequently the interpalladium distances are greater than 4.5 Å. In a second group of compounds there is a direct Pd–Pd bond, and the intermetallic distances are approximately 2.5 Å.[6] The observed Pd–Pd distances (~3.5 Å) for **1** do not fall within either range but correspond more closely to a nonbonded Pd–Pd separation. A related structure in mercury chemistry is $Na_4[Hg_4(\mu\text{-}Cl)_4\{P(O)(OEt)_2\}_8]\cdot 6H_2O$, which has a symmetrical tetrameric arrangement with bridging chlorides and two diethyl phosphonato-*P* ligands complexed to each mercury atom.[8]

The isolation of this tetrameric palladium complex with bridge-spanning ligands structurally represents the "halfway step" to the formation of the target dimer $Pd_2(pcp)_4^{4-}$. The isolation of this new tetrameric complex suggests that, for ligands which have no tendency to form chelate complexes with a single metal center, it is plausible for dimers to be the final products in substitution reactions that proceed via tetrameric intermediates.[9]

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(9) Complex **1** shows no emission down to 77 K, but other palladium complexes showing either red or yellow emissions have been obtained with $pcpH_2$.

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Supplementary Material Available: Tables of coordinates and isotropic thermal parameters for hydrogen atoms and anisotropic thermal parameters (2 pages); listings of observed and calculated structure factors (14 pages). Ordering information is given on any current masthead page.

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Charge-Transfer Absorption and Emission of $Cu(NN)_2^+$ Systems

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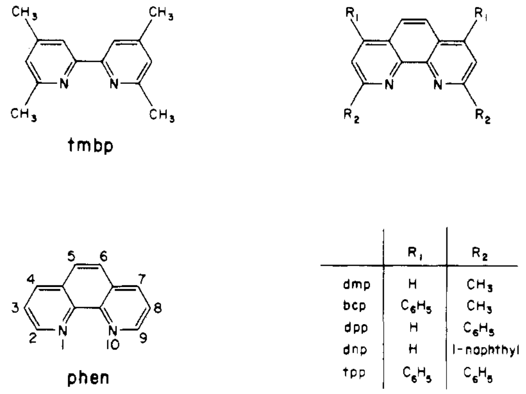
Luminescence from $Cu(NN)_2^+$ systems, where NN denotes a substituted 1,10-phenanthroline ligand, has been reported for pure solids,[1] as well as for complexes dissolved in fluid-solution[2,3] or in glassy media.[4,5] The emission originates from d–π* metal-to-ligand charge-transfer (CT) excited states; more specifically, temperature-dependent emission studies have established that at least two different excited states are involved and that they differ in spin multiplicity.[6] The photophysical properties of $Cu(NN)_2^+$ systems have also been found to be strongly affected by substituent groups on the ligand moieties. Steric[3,5] and electronic[7] effects are involved, but the steric effects are very dramatic because the excited state is subject to a novel type of quenching wherein a Lewis base attacks the "Cu(II)" center, possibly forming a short-lived excited-state complex (exciplex).[2,5,8] Sterically active groups on the complex or the incoming nucleophile introduce "F-strain"[9] and inhibit exciplex quenching, thereby enhancing the excited-state lifetime.[5,10] Other aspects of the photochemistry and photophysics of these systems are also being pursued.[3,11–20] In order to characterize the electronic structures and the excited-state dynamics of these systems in more detail, we have carried out systematic studies of the absorption and emission properties of a number of complexes of NN ligands (Figure 1) in fluid solution and frozen matrices.

Experimental Section

Materials. The $[Cu(NN)_2]BF_4$ salts were prepared by a method previously described.[21] The dmp (2,9-dimethyl-1,10-phenanthroline) and bcp (2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline) ligands were obtained from Aldrich, while phen (1,10-phenanthroline) was purchased from G. F. Smith; each was used without purification. dpp (2,9-diphenyl-1,10-phenanthroline) and tpp (2,4,7,9-tetraphenyl-1,10-phenanthroline) were synthesized as before,[22] and dnp (2,9-bis(1-naphthyl)-1,10-phenanthroline) was prepared analogously.[23] tmbp (4,4′,6,6′-tetramethyl-2,2′-bipyridine) was synthesized by the method of Linnell.[24] Spectral grade solvents were used for all the physical studies.

Apparatus. Absorption measurements were made on a Cary 17D spectrophotometer. The emission spectra were obtained with a Perkin-Elmer MPF-44B fluorescence spectrophotometer, while lifetimes were measured with a Princeton Applied Research Model 2100 N_2-pumped dye laser source and a data-collection system that have previously been described.[12] Measurements in the glass were obtained with either a quartz finger Dewar or an Oxford Instruments DN-704 cryostat. For measurements in fluid solution, temperature control was achieved with a Lauda K-2/RD circulating water bath.



	R_1	R_2
dmp	H	CH_3
bcp	C_6H_5	CH_3
dpp	H	C_6H_5
dnp	H	1-naphthyl
tpp	C_6H_5	C_6H_5

Figure 1. NN ligands.

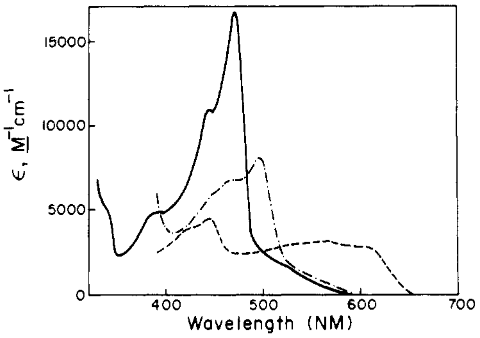


Figure 2. Absorption spectra at 90 K in 4:1 EtOH/MeOH glass of $Cu(dmp)_2^+$ (—), $Cu(dnp)_2^+$ (-•-), and $Cu(dpp)_2^+$ (---).

Table I. Absorption Data for $Cu(NN)_2^+$ Systems in 4:1 EtOH/MeOH at 90 K

NN[a]	band	λ, nm	ε,[b] M^{-1} cm^{-1}
phen	I	500 (sh)	
	IIa	463	17 200
	IIb	435	12 500
	III	380	8 100
dmp	I	510 (sh)	
	IIa	460	16 500
	IIb	440	10 800
	III	390	4 800
bcp	I	540 (sh)	
	IIa	495	31 600
	IIb	460	20 200
	III	425	10 700
dnp	I	530 (sh)	
	IIa	485	8 100
	IIb	460	7 100
tmbp	I	510 (sh)	
	IIa	477	13 770
	IIb	447	10 270
dpp		600	2 800
		560	3 100
		436	4 400
		413	3 900
tpp		590	9 800
		460	10 300

[a] See Figure 1 for abbreviations. [b] The ε values are not corrected for solvent contraction.

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