can be ruled out since the pyridine lowest lying triplet state (29 650 cm^{-1})[7] is energetically too high. Thus, exciplex formation as described above would appear to be an attractive alternative mechanism for MLCT* quenching. This proposal draws support from the effects of pyridine substituents in the sterically significant ortho position.[8] Plots of τ_0/τ vs [2,6-dimethylpyridine] and [2,6-di-*tert*-butylpyridine] (Figure 1 (parts b and c, respectively)) depict the marked decreases in quenching rates with increases in substituent steric bulk. The corresponding k_q values are $9.5 \pm 0.9 \times 10^8$ and $\leq 10^6$ M^{-1} s^{-1}.

During the quenching studies, the MLCT emission lifetime also proved to be sensitive to the concentration of I, but the MCC emission lifetime was invariant over the same range of conditions. Figure 2 demonstrates the linear dependence between $[Cu_4I_4(py)_4]$ and the reciprocal of the lifetime (τ^{-1}). The slope of this plot gives the self-quenching rate constant with $k_q^s = 4.5 \pm 0.4 \times 10^9$ M^{-1} s^{-1} and an intercept (τ_0^{-1}) of $2.2 \pm 0.2 \times 10^6$ s^{-1} from which was calculated $\tau_0 = 0.45 \pm 0.04$ μs.

One potential explanation of the self-quenching behavior is that this phenomenon is a reflection of quenching by pyridine liberated by dissociation from I, e.g.,

$$\underset{\text{I}}{Cu_4I_4(py)_4} \xrightleftharpoons{K_1} \underset{\text{II}}{Cu_4I_4(py)_3} + py \qquad (1)$$

For this mechanism to be valid, a K_1 value about 3×10^{-3} M would be required by the data in Figure 2 and the k_q for pyridine (5.9×10^9 M^{-1} s^{-1}). This would lead to [II]/[I] ratios ranging from 83 to 3.1 over the concentrations used in Figure 2, a proposal inconsistent with linear Beer's law plots (e.g., Abs(360 nm) vs [I]) over this concentration range.[9] Thus, since the self-quenching is exclusive to the higher energy MLCT state, we propose that the self-quenching mechanism involves bimolecular energy transfer as illustrated in eq 2.[10] Unfortunately, detection of the enhanced MCC* emission thereby expected is precluded by the small fraction of the total excited state population represented by the MLCT* under these conditions.

$$I^*(MLCT^*) + I \rightarrow I + I^*(MCC^*) \qquad (2)$$

In summary, two different quenching mechanisms are reported here for the metal-to-ligand charge-transfer excited state of the copper(I) cluster $Cu_4I_4py_4$ in benzene. Quenching by the Lewis base pyridine appears to be operating by complex formation with the Cu(II) center of the MLCT* as previously described for charge-transfer excited states of the mononuclear Cu(I) complex $Cu(dmp)_2^+$.[1,2] A competitive mechanism is quenching of the MLCT* by another I, a process proposed to be facile owing to the presence of the MCC* at a lower energy in the manifold of electronic states. In contrast, the MCC emission is not quenched by either mechanism. That the higher energy MLCT* can be quenched by bimolecular pathways, especially the self-quenching mechanism, in competition with unimolecular internal conversion to the MCC state represents a particularly dramatic demonstration of the poor coupling between these two types of excited states in this cluster.

Acknowledgment. This work was supported by Grant No. CHE87-22561 from the National Science Foundation.

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[8] The observation that quenching also occurs with Lewis bases such as methanol and tetrahydrofuran is consistent with the proposed exciplex mechanism.

[9] (a) Linear Beer's law behavior would also preclude significant contributions from equilibria between the Cu_4 cluster and other copper species of lower nuclearity. These data would not eliminate an equilibrium between the step and cube forms of $Cu_4I_4py_4$.[9b] (b) That the MLCT* and MCC* emissions arise solely from the cube form of $Cu_4I_4py_4$ has been demonstrated by solid-state emission spectroscopy. Both emissions are still present in samples which have been shown by crystallography to be *closo*-$Cu_4I_4py_4$.[9c] (c) Raston, C. L.; White, A. H. *J. Chem. Soc., Dalton Trans.* **1976**, 2153.

[10] Qualitative studies demonstrate that biphenyl with a triplet state energy of 23 000 cm^{-1}, which is close to the 0-0 energy of the MLCT state, quenches the MLCT emission lifetime but not that of the MCC state.

Preparation, Crystal Structures, and Spectroscopic Characterization of Diaminochalcogenophosphonium Cations[1]

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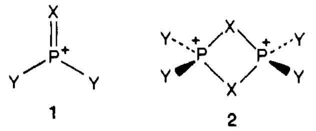
We view the tricoordinate phosphonium electronic environment **1** (isovalent to the carbon center in ethene) as a fundamental key to the development of group 15 chemistry. However, examples of **1** are rare, and characterization is incomplete. Phosphoryl chloride is postulated to autoionize giving **1a**,[4] and the nitrogen **1b**,[5] carbon **1c**,[6] and thio **1d**[4b,7] analogues of **1** have recently been identified spectroscopically in solution. Alternative covalent structures involving phosphoryl-[4] and thiophosphoryl-Lewis acid adducts[8] have also been studied. Here we report attempts to investigate diaminothiophosphonium and tetraethyldiaminoselenophosphonium cations and reveal them to adopt dimeric ring structures **2** in the solid state. The derivatives of **2** represent the first examples of heterocyclic chalcogen-phosphorus cations. In addition, we present new spectroscopic evidence for the solution behavior of the chalcogenophosphonium systems.



1 2

a, X = O; Y = Cl
b, X = NR; Y = NR_2
c, X = CR_2; Y = NR_2
d, X = S; Y = Ph, Y = NR_2
e, X = S; Y = NEt_2;
e', Y = NMe_2
f, X = Se; Y = NEt_2

Reactions of Y_2XPCl (**e**, **e'**,[9] and **f**[10]) with $AlCl_3$ in CH_2Cl_2 at room temperature provided good yields (**e**, 86%; **e'**, 77%; **f**, 95%) of crystalline solids (**e**, **e'**, white; **f**, yellow) with the empirical formula $1AlCl_4^-$.[11] The ionic nature of these extremely air-

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Table I. Selected Bond Lengths (Å) and Angles (deg) for the Dicationic Units **2e** and **2f**

2e		**2f**			
P-S	2.109 (2)	P1-Se1	2.257 (3)	P1-Se2	2.249 (3)
P[a]-S	2.083 (2)	P2-Se1	2.238 (3)	P2-Se2	2.262 (3)
P-N1	1.601 (5)	P1-N1	1.614 (9)	P1-N2	1.58 (1)
P-N2	1.607 (5)	P2-N3	1.60 (1)	P2-N4	1.625 (9)
P[a]-S-P	84.0 (1)	P1-Se1-P2	82.9 (1)	Se1-P1-Se2	97.2 (1)
S[a]-P-S	96.0 (1)	P1-Se2-P2	82.6 (1)	Se1-P2-Se2	97.3 (1)
N1-P-S	113.1 (2)	N1-P1-Se1	118.0 (4)	N2-P1-Se2	113.2 (4)
		N1-P1-Se2	110.1 (4)	N2-P1-Se1	109.4 (4)
N2-P-S	108.8 (2)	N3-P2-Se1	110.2 (4)	N4-P2-Se2	108.7 (4)
		N3-P2-Se2	112.9 (4)	N4-P2-Se1	116.1 (4)
N2-P-N1	112.0 (3)	N1-P1-N2	108.7 (5)	N3-P2-N4	111.0 (5)

[a] Atoms related by a crystallographic center of inversion.

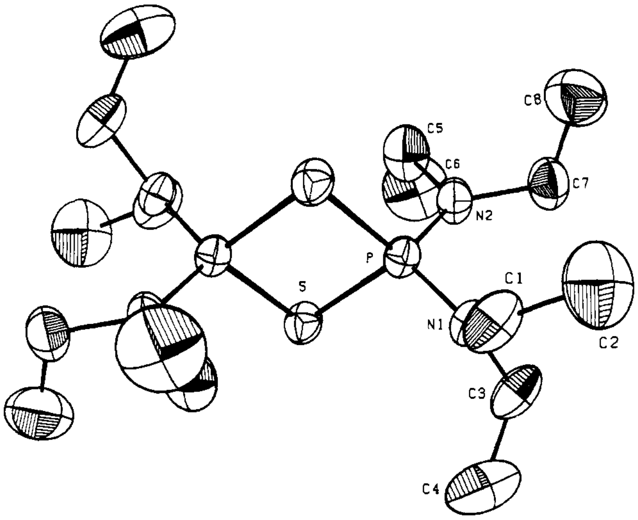


Figure 1. A view of dication **2e**.

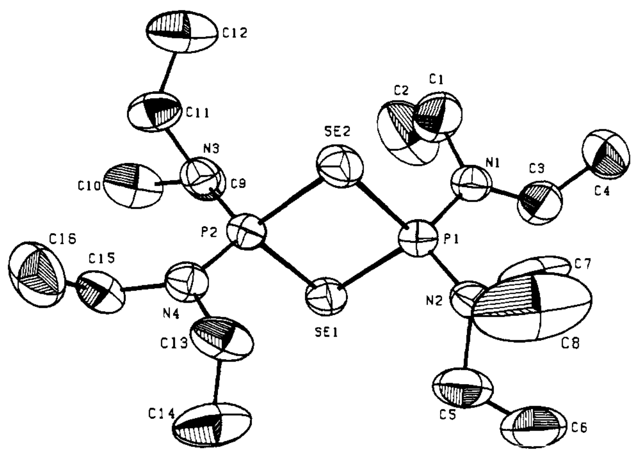
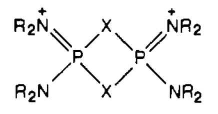
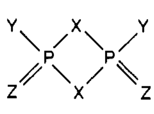


Figure 2. A view of dication **2f**.

sensitive solids is evident from the infrared band at 485 cm^{-1}, characteristic of $AlCl_4^-$.[12] Discrete cationic dimeric units **2** with similar conformations are observed in the X-ray crystal structures[13] of **e** (Figure 1) and **f** (Figure 2). Selected bond lengths and angles are presented in Table I. In common with phosphetane structures,[14] the planar P_2X_2 ring is perpendicular to the plane of the four substituent centers. The amine units (C_2NP) are individually planar indicating the presence of a substantial P-N π-interaction. This is supported by the short P-N bond lengths, which average 1.60 (2) Å in **2f** and 1.604 (3) Å in **2e** (P-N single bond 1.77 Å).[15] A similar shortening is observed for other aminophosphonium centers.[16] The P-X bond lengths are typical for single bonds (P-Se = 2.252 (9) Å average; P-S = 2.10 (1) Å average),[15] and the overall molecular bonding is best viewed as resonance structures of **3**. In this respect, **2e** and **2f** are isovalent and structurally similar to known phosphetane derivatives **4**.[14]



3



4

Freshly prepared solutions (CH_2Cl_2) of **2e** and **2e′** as well as preparative reaction mixtures exhibit a single ^{31}P NMR[17] signal

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(11) $C_4H_{12}N_2PSAlCl_4$: Anal. Calcd: C, 15.01; H, 3.78; N, 8.75; P, 9.68; S, 10.01. Found: C, 15.11; H, 3.76; N, 8.67; P, 9.51; S, 9.90. IR (CsI plates, Nujol mull) 1300, 1160, 1055, 1010, 985, 705, 635, 485 (br) cm^{-1}. $C_8H_{20}N_2PSAlCl_4$: Anal. Calcd: C, 25.55; H, 5.36; N, 7.45. Found: C, 25.61; H, 5.36; N, 7.30. IR (CsI plates, Nujol mull) 1300, 1205, 1150, 1100, 1065, 1025 (br), 960, 930, 790, 675, 630, 490 (br) cm^{-1}. $C_8H_{20}N_2PSeAlCl_4$: Anal. Calcd: C, 22.72; H, 4.77; N, 6.62; P, 7.32; Se, 18.67. Found: C, 22.67; H, 4.96; N, 6.62; P, 7.31; Se, 18.63. IR (CsI plates, Nujol mull) 1290, 1205, 1145, 1100, 1050, 1015, 980, 955, 925, 795, 660, 540, 485 (br) cm^{-1}.

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(13) Crystal data for $C_{16}H_{40}N_4P_2S_2Al_2Cl_8$ (**2e**): $M = 752.19$, monoclinic, space group $P2_1/n$, $a = 10.598$ (2) Å, $b = 8.976$ (2) Å, $c = 19.370$ (4) Å, $\beta = 98.65$ (2)°, $V = 1822$ Å^3, $Z = 2$, $D_c = 1.37$ g cm^{-3}, $\mu_{calc} = 8.1$ cm^{-1}, λ(Mo Kα) = 0.71073 Å. The intensities of 3598 reflections were measured on an Enraf-Nonius CAD-4 diffractometer at room tempeature ($2 < 2\theta < 50°$). The structure was solved by direct methods (MULTAN) and refined by full-matrix least-squares methods using 1544 unique reflections [$F_o > 5\sigma(F_o)$]. The reflection:parameter ratio was 9:1. The final residuals were $R = 0.052$ and $R_w = 0.056$. Crystal data for $C_{16}H_{40}N_4P_2Se_2Al_2Cl_8$ (**2f**): $M = 846.0$, triclinic, space group $P\bar{1}$, $a = 10.635$ (7) Å, $b = 12.335$ (8) Å, $c = 15.159$ (9) Å, $\alpha = 95.94$ (8)°, $\beta = 93.46$ (7)°, $\gamma = 110.99$ (9)°, $V = 1836.5$ Å^3, $Z = 2$, $D_c = 1.53$ g cm^{-3}, λ (Mo Kα) = 0.71073 Å, $\mu_{calc} = 26.3$ cm^{-1}. An empirical absorption correction was applied. The intensities of 5621 reflections were measured on an Enraf-Nonius CAD-4 diffractometer at room temperature ($2 < 2\theta < 50°$). The structure was solved by direct methods (MULTAN) and refined by full-matrix least-squares methods using 3276 unique observed reflections [$F_o > 5\sigma(F_o)$]. The reflection:parameter ratio was 10:1. The final residuals were $R = 0.066$ and $R_w = 0.069$.

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(72.5 ppm and 78.6 ppm, respectively) slightly upfield from those of the corresponding precursor thiophosphoryl chlorides (84.6 and 91.5 ppm). Reaction of $AlCl_3$ with an equimolar mixture of the precursors (Y_2XPCl) **e** and **e′** gives ^{31}P NMR signals at 72.3 and 78.2 ppm and shows no phosphorus-phosphorus coupling (i.e., no asymmetric dimers) indicating that these signals correspond to monomeric species. However, while the ^{27}Al NMR spectra contain a sharp signal at 102 ppm ($AlCl_4^-$),[18] the more substantial (>50%) signal is a broad peak slightly downfield (106 ppm). These ^{27}Al spectra are similar to those obtained for $(NMe_2)_3P{=}S{\cdot}AlCl_3$ and $Ph_3P{=}S{\cdot}AlCl_3$.[19] We conclude that, in solution, chloride ion reabstraction occurs allowing formation of a covalent Lewis acid-base adduct ($Y_2PSCl{\cdot}AlCl_3$)[21] and that the ionic nature of **2e** and **2e′** is dependent on a crystal lattice factor. In contrast, a single sharp signal at 102 ppm ($AlCl_4^-$)[18] is observed in the ^{27}Al NMR spectrum of solutions of **2f**. Moreover, the ^{31}P spectrum includes signals for $(Et_2N)_2P^+$ (264.0 ppm)[20] and **2f** (−7 ppm, $J_{P\text{-}Se}$ = 341 Hz, identified as a triplet in the ^{77}Se NMR spectrum $J_{P\text{-}Se}$ = 340 Hz) and other unidentified signals at 79–80 ppm, demonstrating complex equilibria involving only ionic species.

The identification of derivatives of **2** in the solid state further demonstrates the dominant stability of the phosphetane framework.[14] However, this new cationic system has unique dissociative properties in solution, with the nature of the species dependent upon the chalcogen (X). We anticipate the stabilization of derivatives of **1** through the suitable choice of substituents X and Y.

Supplementary Material Available: Crystal structures, experimental details, unit cell packing diagrams, and tables of crystal data, atomic positional parameters, anisotropic thermal parameters, bond lengths, bond angles, and least-squares planes for $[(Et_2N)_2PS]_2(AlCl_4)_2$ and $[(Et_2N)_2PSe]_2(AlCl_4)_2$ (16 pages); table of observed and calculated structure factors (10 pages). Ordering information is given on any current masthead page.

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Binding of 4′,6-Diamidino-2-phenylindole (DAPI) to GC and Mixed Sequences in DNA: Intercalation of a Classical Groove-Binding Molecule

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DNA intercalators typically contain planar, fused-aromatic systems that slide between base pairs in the complex.[1] Molecules that bind in the minor groove generally contain unfused-aromatic systems with terminal basic functions.[2] Detailed molecular models for both binding modes have been developed from X-ray crystallographic and molecular modeling results.[3,4] For example, (i) binding in the DNA minor groove is sterically inhibited by the 2-NH_2 group of G[2,4] and (ii) several aromatic diamidines, 4′-6-diamidino-2-phenylindole[5] (DAPI, Figure 2), berenil,[1b] and hydroxystilbamidine,[6] are AT specific minor groove-binding agents.[2] Early investigations with DAPI suggested that it binds to DNA by intercalation,[5e] but more detailed studies indicated that it binds specifically to AT base pairs in the minor groove.[2,5a-d]

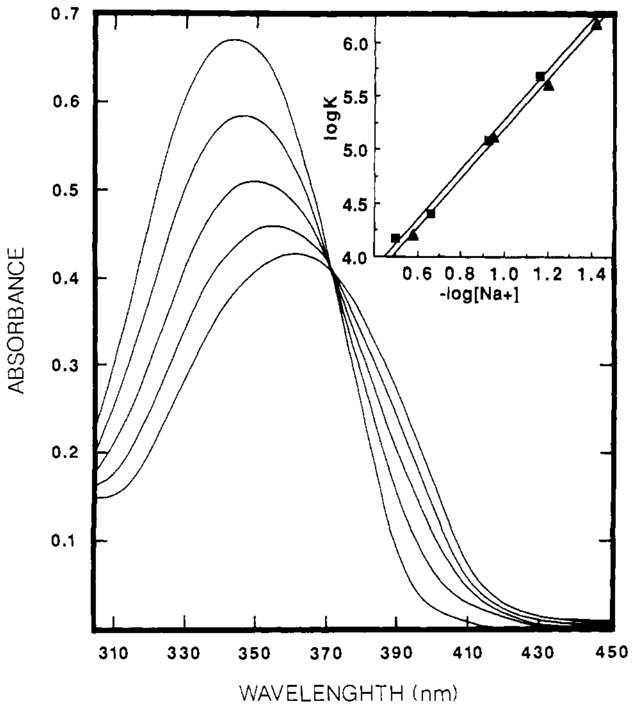

Figure 1. Spectral shifts of DAPI on titration with polyd(G-C)$_2$. The DAPI concentration was 2.5 × 10^{-5} M, and the polyd(G-C)$_2$ base-pair molarity increased as follows (top to bottom curves at 340 nm): 0, 1.4, 2.7, 4.0, 6.0 × 10^{-5}. The titration was conducted in a 1-cm cuvette in MES buffer on a Cary 2200 spectrophotometer. The inset shows plots of log *K* vs −log [Na^+] (NaCl added to MES buffer) for DAPI (■) and quinacrine[12] (▲).

The intercalation with DNA of a group of unfused-aromatic cations, similar in structure to classical groove-binding molecules, has led to the conclusion that intercalation and groove-binding modes should be viewed as two variable depth potential wells on a continuous energy surface.[7] This result requires the investigation

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