clometalated product results from nucleophilic atack by OH⁻ at the ortho carbon of a pentafluorophenyl ring. No reaction occurs between uncomplexed PPh₂C₆F₅ and aqueous KOH. The ortho substitution in complex 1 is therefore induced by platinum coordination. Two mechanistic pathways are plausible, each of which involves nucleophilic attack by hydroxide at the ortho carbon of the pentafluorophenyl ring (Scheme I). The first pathway involves an agostic interaction which induces ortho selectivity by the external nucleophile; the second pathway involves prior complexation of the hydroxide with platinum.⁸ We presently have no conclusive evidence to differentiate between intramolecular or external attack at the ortho carbon by the hydroxyl nucleophile. Complex 1 is protonated by HCl to give the ring-opened complex trans- $PtClMe(PPh_{2}C_{6}F_{5})(PPh_{2}C_{6}F_{4}OH-2)$ (2) (eq 4).

$$trans-PtMe(2-OC_6F_4PPh_2)(PPh_2C_6F_5) + HCl \rightarrow trans-PtClMe(PPh_2C_6F_5)(PPh_2C_6F_4OH-2)$$
(4)

Methoxide ion (excess NaOMe in methanol) substitutes the fluorines at the ortho carbon atoms of the pentafluorophenyl ring in trans-[PtMe(THF)(PPh₂C₆F₅)₂]CF₃SO₃ to give trans-Pt- $(OMe)Me(PPh_2C_6F_3(OMe-2,6)_2)_2$ (3) in 82% yield. Complex 3 reacts with NaOH to give trans-PtMe(2-OC₆F₃(OMe-6)- PPh_2)($PPh_2C_6F_3$ (OMe-2,6)₂) (4) (eq 5).⁹ The conversion of 3

trans-IPtMe(THF)(PPh2C6F5)23X + 5NaOMe -



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Supplementary Material Available: Tables of positional pa-

rameters, bond distances, bond angles, general displacement parameters, and torsion angles (13 pages); listing of observed and calculated structure factors (40 pages). Ordering information is given on any current masthead page.

First Structural Evidence for Transannular P-N Bonding in the Phosphine Form of Cyclenphosphorane: An Open Tautomer?

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Evidence has shown that cyclenphosphorane (cyclenPH) exists only in the "closed" tautomer 1a in solution as well as in the solid



and gas phases.¹ Attempts to isolate the "open" form 1b by coordination to transition metals have, thus far, been unsuccessful, except in the rare cases where it is forced into the bidentate structure A.² Reactions of cyclenPH with transition metals usually give the pentacoordinate structure B.3 It has been sug-



gested that this is due to the constraint of the 12-membered cyclen ring which stabilizes the trigonal-bipyramidal (tbp) geometry around phosphorus.^{3,4} We herein report the synthesis and X-ray crystal structure of the first monodentate P-bound transition-metal complex of 1b. The structure of this complex reveals a P-N transannular interaction, which yields a unique geometry for a phosphine ligand, and provides the first structural confirmation of the tbp constraining "bite" of the cyclen ring about phosphorus. Moreover, the geometry explains why this complex undergoes a

⁽⁸⁾ Although we have no direct evidence for an agostic interaction, pre-(8) Although we have no direct evidence for an agostic interaction, pre-cedents exist for both organohalide complexation and C-F cleavage and oxidative addition, see: Richmond, T. G.; Osterberg, C. E.; Arif, A. M. J. Am. Chem. Soc. 1987, 109, 8091-8092. Crabtree, R. H.; Faller, J. W.; Mellea, M. F.; Quirk, J. M. Organometallics 1982, 1, 1361-1366. Crabtree, R. H.; Mellea, M. F.; Quirk, J. M. J. Am. Chem. Soc. 1984, 106, 2913-2917. Gross, M. E.; Johnson, C. E.; Maroney, M. J.; Trogler, W. C. Inorg. Chem. 1984, 22 0268-2023. For supersphilic substitution of fluoronzometics can: 1984, 23, 2968-2973. For nucleophilic substitution of fluoroaromatics see: Tatlow, J. C. Endeavour 1963, 22, 89-95. Allen, J. G.; Burdon, J.; Tatlow, J. C. J. Chem. Soc. 1965, 1045-1051. Banks, R. E. Fluorocarbons and their Derivatives, 2nd ed.; MacDonald: London, 1970; pp 218-221. Attack by external nucleophile is favored by the observation that no reaction occurs with NaOH instead of KOH, but prior hydroxyl coordination better explains the ortho selectivity. An S_{RN} l mechanism fails to explain the selectivity, and futhermore 1 is not formed in the reaction between trans-[PtMe(THF)-

futhermore 1 is not formed in the reaction between trans-[PtMe(THF)-(PPh₂C₆F₃)₂]X and sodium naphthalenide. (9) ¹H, ³¹Pl¹H}, and ¹⁹F NMR data for 3: δ (CH₃) 0.23 t; ³J(PH) = 6.4 Hz. δ (OCH₃), 3.32 d (12 H); ⁵J(HF) = 2.4 Hz. δ (OCH₃) 3.14 s (3 H); ³J(PtH) = 23 Hz. δ (P) 16.7 s; ¹J(PtP) = 3527 Hz. δ (CF) -151.6 t (2 F), -155.8 d (4 F); ³J(FF) = 20.3 Hz. 4: δ (CH₃) 0.83 dd; ³J(PH) = 7.0 Hz, ³J(PH) = 5.5 Hz. δ (OCH₃) 3.23 d (6 H); ⁵J(HF) = 2.5 Hz. δ (OCH₃) 3.00 d (3 H); ⁵J(HF) = 2.7 Hz. δ (P) 33.8 d, 20.7 d; ²J(PP) = 442 Hz. δ (CF) -150.7 t (1 F), -152.1 t (1 F), -155.6 d (2 F), -163.7 d (1 F), -169.6 d (1 F); ³J(FF) = 20.5 Hz. (10) A similar C-O cleavage reaction has here published

 ⁽¹⁰⁾ A similar C-O cleavage reaction has been published, see: Jones, C.
 E.; Shaw, B. L.; Turtle, B. L. J. Chem. Soc., Dalton Trans. 1974, 992-999.

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Richman, J. E.; Atkins, T. J. Tetrahedron Lett. 1978, 4333. (2) (a) Burns, E. G.; Chu, S. S. C.; de Meester, P.; Lattman, M. Or-ganometallics 1986, 5, 2383. (b) Khasnis, D. V.; Lattman, M.; Siriwardane,

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surprising protonation reaction.

Treatment of cyclenPH with $Mo(CO)_6$ in refluxing toluene yields air-stable crystals of HcyclenPMo(CO)₅, 2, in which cyclenPH is coordinated to the metal (through the phosphorus) in the open form 1b.5,6 The proposed geometry of the product is supported by the ³¹P chemical shift of +116 ppm, which is about 100-150 ppm further downfield than either cyclenPH itself, or the pentacoordinate cyclenP metal derivatives and is indicative of a lower coordination number for phosphorus.^{2,3} The N-H appears as a broad resonance in the ¹H NMR spectrum at δ 1.47 and a weak band in the $\nu_{\rm NH}$ region of the IR spectrum at 3400 cm⁻¹. A direct P-Mo bond is supported by the observation of two-bond PC coupling to the carbonyl carbons in the ${}^{13}C{}^{1}H$ NMR spectrum.⁶ In addition, four resonances appear in the ¹³C spectrum in the methylene region. Isolation of this geometry of cyclenPH is most likely due to the fact that the $Mo(CO)_5$ fragment has only one vacant coordination site.

Since nitrogens attached to phosphorus usually show reduced basicity,⁷ the N-H nitrogen in **2** would be expected to be the most reactive site toward Lewis acids. However, treatment of **2** with HBF₄ yields a species with a ³¹P chemical shift at δ +35, which is 81 ppm upfield from **2**. The CO region in both the IR and ¹³C NMR spectra are very similar to **2**, suggesting that the Mo(CO)₅ fragment is intact. Moreover, the ¹³C NMR spectrum shows only *two* resonances in the methylene region, which indicates the presence of only two types of carbons. These spectra are consistent not with the simple ammonium salt formation product **3** but rather with a pentacoordinate phosphorus with both axial nitrogens protonated, [(H₂cyclenP)Mo(CO)₅]BF₄, **4**.⁸⁻¹⁰



The unexpected product obtained by protonation led us to examine the structure of 2 more closely to see if it might give insights into the observed reactivity. The X-ray crystal structure

(C₁₃H₁₇MoN₄O₅P) C, H, N. (6) Spectroscopic data for 2: ¹H NMR (CDCl₃) δ 1.47 (br, 1 H), 2.3–3.3 (comp m, 16 H); ³¹P[¹H] NMR (CDCl₃) δ 116; ¹³C[¹H] NMR (THF-d₈) δ 49.3 (CH₂), 50.6 (CH₂), 50.9 (CH₂), 52.5 (CH₂, d, ²J_{PC} = 22 Hz), 207.4 (CO, d, ²J_{PC} = 13 Hz), 212.0 (CO, d, ²J_{PC} = 32 Hz); IR (hexanes, cm⁻¹) ν_{CO} 1945 (vs), 2060 (w); ν_{NH} (KBr) 3400 (vw). (7) Verkade, J. G. *Coord. Chem. Rev.* 1972/3, 9, 1, and references therein.



Figure 1. Computer-generated drawing of HcyclenPMo(CO)₅, 2. Hydrogen atoms omitted for clarity. Selected interatomic distances (Å) are as follows: Mo-P \approx 2.533 (5); P-N(1) = 1.809 (20); P-N(2) = 1.749 (20); P-N(3) = 2.356 (23); P-N(4) = 1.666 (20). Angles (deg) are as follows: Mo-P-N(1) = 107.7 (7); Mo-P-N(2) = 117.6 (9); Mo-P-N(3) = 98.6 (9); Mo-P-N(4) = 120.4 (8); N(1)-P-N(2) = 82.8 (11); N-(1)-P-N(3) = 153.5 (13); N(1)-P-N(4) = 90.7 (9); N(2)-P-N(3) = 82.0 (12); N(2)-P-N(4) = 120.8 (12); N(3)-P-N(4) = 78.8 (13).

of $\mathbf{2}$ was obtained¹¹ and is illustrated in Figure 1, along with selected atomic distances and angles. The geometry about the phosphorus is unique for a phosphine bound to a metal: instead of being close to tetrahedral, the phosphorus is a distorted tbp. The Mo, N(2), and N(4) atoms are at the equatorial positions of the phosphorus, while N(1) occupies one axial position. The other axial site is occupied by N(3), which interacts with P through its lone pair of electrons. Although the P-N(3) distance of 2.356 (23) Å is about 0.4 Å longer than other P-N axial bonds in related cyclenP-metal derivatives,^{2,3} it is well within the sum of the van der Waals radii. More importantly, the phosphorus lies almost in the plane of the Mo, N(2), and N(4) atoms [sum of equatorial angles about P is 359 (2)°]. The N-P-N equatorial and axial angles of 120.8 (12)° and 153.5 (13)°, respectively, are typical for other tbp cyclenPX derivatives in which all four nitrogens show "normal" bond distances to phosphorus. The cyclenP unit adopts a staggered conformation with respect to the Mo-C-O bonds cis to the phosphorus, presumably to minimize steric interactions. It is noted that there is no significant difference between the solution and solid-state structures because the MAS ³¹P chemical shift of **2** appears at δ +109, only 7 ppm shifted from the solution value.12,13

⁽⁵⁾ Synthesis of 2 (inert atmosphere, all solvents dried and deoxygenated). A solution of cyclenPH (0.20 g, 1.0 mmol) and $Mo(CO)_6$ (0.30 g, 1.1 mmol) were refluxed in toluene (5 mL) for 4 h. The volatiles were then pumped off, and the residue was recrystallized from hot hexanes (10 mL), yielding 2 as an off-white, air-stable, crystalline solid (0.41 g, 95%): mp 110-112 °C. Anal. (C₁₃H₁₇MoN₄O₅P) C, H, N.

⁽⁷⁾ Verkade, J. G. Coord. Chem. Rev. 1972/3, 9, 1, and references therein. (8) Synthesis of 4 (inert atmosphere, all solvents dried and deoxygenated). A stirred solution of 2 (135 mg, 0.309 mmol) in hexanes (3 mL) was treated dropwise with 85% HBF₄·O(C_2H_5)₂ (48 μ L, 0.28 mmol). After stirring for 8, the resulting precipitate was filtered, washed with hexanes and ether, and pumped dry to yield 4 as a white, air-stable, solid (144 mg, 90%): dec 185–187 °C.

⁽⁹⁾ Spectroscopic data for 4: ¹H NMR (CD₃CN) δ 2.3–3.3 (comp m, 16 H), 4.86 (br, 2 H); ³¹Pl¹H} NMR (CD₃CN) δ 35; ¹³Cl¹H} NMR (CD₃CN) δ 45.3 (CH₂, d, ²J_{PC} = 2 Hz), 45.4 (CH₂, d, ²J_{PC} = 2 Hz), 206.1 (CO, d, ²J_{PC} = 13 Hz), 211.0 (CO, d, ²J_{PC} = 41 Hz); IR (CH₃CN, cm⁻¹) ν_{CO} 1937 (vs), 2065 (w).

⁽¹⁰⁾ This H₂cyclenP geometry has been observed before. See ref 3b.

⁽¹¹⁾ X-ray structure determination and refinement. Suitable colorless, cube-shaped crystals of **2** were grown from a solution of hot hexanes. Data were collected at room temperature on a Nicolet R_{3m}/V diffractometer using MoK α radiation. $C_{13}H_{17}N_4O_5Mo$, orthorhombic, space group *Pbca*, a = 12.201 (16) Å, b = 13.432 (15) Å, c = 21.48 (2) Å, V = 3520 (7) Å³, Z = 8, and $d_{calcd} = 1.65$ g cm⁻³. A total of 3750 reflections were collected in the range $3.0^{\circ} \le 2\theta \le 50.0^{\circ}$. Data were corrected for decay (~2%), Lorentz polarization, and absorption. The structure was solved by direct methods using SHELXTL-PLUS (Sheldrick, G. M. *Structure Determination Software Programs*, Nicolet Instrument Corp., 1988) and subsequent difference Fourier methods. Final full-matrix least-squares refinement using 1892 observed reflections based on $F > 6.0\sigma(F)$ converged to R = 0.062 and $R_w = 0.069$; GOF = 2.27, $(\Delta/\sigma)_{max} = 0.10 \Delta \rho_{(max, min)} = 0.63, -0.82$ e/Å³, the function minimized being $\Sigma w(|F_o| - |F_c|)^2$, the weight used being $w^{-1} = \sigma(F^2) + 0.0013F^2$. All non-hydrogen atoms were refined anisotropically; hydrogens on carbon atoms were included in calculated positions with a riding model with fixed isotropic thermal parameters and were included in the final refinement. Scattering factors for C, H, N, O, P, and Mo used were those stored in SHELXTL-PLUS.

SHELXTL-PLUS. (12) The ³¹P chemical shift of **2** does not, at first, suggest a high coordination for phosphorus because of the far downfield position. However, a possible model for comparison would be $(Me_2N)_3PMo(CO)_5$, a species where no increased coordination of the phosphorus is possible. The ³¹P shift of this compound is δ +145 (Andrews, G. T.; Colquhoun, I. J.; McFarlane, W.; Grim, S. O. J. Chem. Soc., Dalton Trans. 1982, 2353), about 30 ppm downfield of **2**; this difference may reflect somewhat of an upfield shift of the phosphorus in **2** due to the interaction of the nitrogen lone pair with phosphorus. However,

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CCH

The X-ray structure of 2 shows that the cyclen ring does have the correct bite for a tbp geometry around phosphorus and indeed adopts that orientation via a P-N transannular interaction. This provides the first direct structural evidence for this often-suggested ability.^{1-3,14} Moreover, in view of the geometry about phosphorus, the electronics of cyclenP might be best described as a zwitterionic phosphoranide^{3d} ligand C.



Acknowledgment. We thank the Robert A. Welch Foundation for generous financial support. D.V.K. thanks Prof. Narayan S. Hosmane for helpful discussions. We thank Prof. Robert H. Neilson for obtaining the ¹³C NMR spectrum of 4.

Supplementary Material Available: Tables of summary of X-ray analysis, atomic coordinates, anisotropic thermal parameters, bond distances, bond angles, and hydrogen atom coordinates for 2 (6) pages); tables of observed and calculated structure factors for 2 (11 pages). Ordering information is given on any current masthead page.

(13) As an alternative explanation to that in ref 12, the ${}^{31}P$ chemical shift may not indicate any transannular interaction since (Me₂N)₃PMo(CO)₅ may not be a suitable comparison standard. The upfield position of 2 compared to $(Me_2N)_3PMo(CO)_5$ may be due simply to incorporation of the phosphorus into two five-membered rings. We thank a referee for pointing out this possibility.

(14) Richman, J. E.; Kubale, J. J. J. Am. Chem. Soc. 1983, 105, 749.

Gas-Phase Ion Chemistry of the Acetic Acid Enolate Anion [CH₂CO₂H]⁻

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Deprotonation of acetic acid forms mainly the acetate anion $CH_3CO_2^{-} [\Delta G^{\circ}_{acid}(CH_3CO_2H)] = 341.5 \text{ kcal mol}^{-1}]^2$ The isomeric enolate ion $[CH_2CO_2H]^{-3}$ has not been studied in the gas phase, although it is implicated in condensed phase reactions.⁴ We wish to report that in our tandem flowing afterglow-selected

 ^{(3) (}a) For early ab initio calculations, see: Wagner, H.-U.; Boche, G. Z. Naturforsch. 1982, 37b, 1339; Helv. Chim. Acta 1983, 66, 842. (b) Ab initio Calculations on [CH₂CO₂H]⁻ give the geometry (RHF/6-31+G*) and energy (MP2/6-31+G*//6-31+G*) indicated below (GAUSSIAN 82, Release H—Frisch, M.; Binkley, J. S.; Schlegel, H. B.; Raghavachari, K.; Martin, R.; Stewart, J. J. P.; Bobrowicz, F.; DeFrees, D.; Seeger, R.; Whiteside, R.; Fox, D.; Fluder, E.; Pople, J. A. Carnegie Mellon University). Results are as follows (hartrees, Å, deg): HO_1 (0.048), O_1C (1.392), CO_2 (1.244), CC (1.374), CH_1 (1.075), CH_2 (1.075), HO_1C (103.68), O_1CC (114.94), O_2CC (130.68), CCH₂ (119.36), CCH₁ (120.96), planar.



(4) Angelo, M. B. C. R. Acad. Sci. Paris, C 1973, 293. Nesmeyanov, A. N.; Surkov, B. A.; Sazonova, V. A.; Zaimovskaya, T. A. Dokl. Akad. Nauk. SSSR 1974, 219, 614. ion flow tube (FA-SIFT),⁵ the acetic acid enolate anion can readily be prepared by the reaction shown in eq 1.6

$$F^{-}$$
 + (CH₃)₃SiCH₂CO₂H \rightarrow HO-C $\begin{pmatrix} O \\ - \\ CH_2 \end{pmatrix}$ + (CH₃)₃SiF (1)
CH₂

Enolate ion I deprotonates acetone $[\Delta G^{\circ}_{acid} = 361.9 \text{ kcal mol}^{-1}]^7$ but not methyl acetate $[\Delta G^{\circ}_{acid} = 365.1 \text{ kcal mol}^{-1}]$;⁷ this brackets $\Delta G^{\circ}_{acid}(CH_3CO_2H)$ at $363 \pm 3 \text{ kcal mol}^{-1}$. Ion I undergoes quite different reactions from that of its isomer the acetate anion II. For example, (i) I (m/z 59) reacts readily with CH₃OD to form m/z 60 (eq 2);⁸ the acetate anion undergoes no deuterium incorporation under these conditions;9 (ii) I undergoes the characteristic nucleophilic aromatic substitution with $C_6F_6^{10}$ shown in eq 3;¹¹ and (iii) I may act as an ambident nucleophile with SO_2 as shown in eq 4 and 5.12

$$HO - C = + CH_3OD - (CH_2CO_2H) + OOCH_3 = - CH_2$$

$$_2 DCO_2 H (OCH_3) \longrightarrow CH_2 DCO_2 + CH_3 OH (2)$$



We have shown that thermalized ions I and II are discrete species with quite different reactivities. However, if enolate I is excited (for example, by collisional activation), will it convert to the more stable acetate anion by 1,3 proton transfer?¹⁴ This

^{(1) (}a) On leave at Boulder from the University of Adelaide. (b) Boulder. (c) Adelaide.

⁽²⁾ Cumming, J. B.; Kebarle, P. Can. J. Chem. 1978, 58, 56.

⁽⁵⁾ Van Doren, J. M.; Barlow, S. E.; DePuy, C. H.; Bierbaum, V. M. J. Am. Chem. Soc. 1987, 109, 4412.

^{(6) (}a) The generation of gas-phase anions from trimethylsilyl compounds is reported by the following: DePuy, C. H.; Bierbaum, V. M.; Flippin, L. A.; Grabowski, J. J.; King, G. K.; Schmitt, R. J.; Sullivan, S. A. J. Am. Chem. Soc. 1980, 102, 5012. (b) The $(M - H^+)^-$ ion is also a product of the reaction between F⁻ and Me₃SiCH₂CO₂H (branching ratio 0.10). (c) Me₃SiCH₂CO₂H is a commercial product.

⁽⁷⁾ Bartmess, J. E.; McIver, R. T. The Gas Phase Acidity Scale. In Gas Phase Ion Chemistry; Bowers, M. T., Ed.; Academic Press: New York, 1979; Vol. 2, p 99. Lias, S. G.; Bartmess, J. E.; Liebman, J. F.; Holmes, J. L.; Levin, R. D.; Mallard, W. G. J. Phys. Chem. Ref. Data 1988, 17 (Suppl. 1).
(8) (a) The ion [(CH₂CO₂H)⁻ + CH₃OD] is also detected in this exper-

iment. (b) Anion I undergoes slow incorporation (one deuterium) with \dot{D}_2O at high D_2O flows.

^{(9) (}a) The acetate ion is made by the reaction $F + (CH_3)_3 SiOCOCH_3$ \rightarrow CH₃CO₂⁻ + (CH₃)₃SiF. (b) The only detectable product of the reaction between CH₃CO₂⁻ and CH₃OD is [CH₃CO₂⁻ + CH₃OD].

⁽¹⁰⁾ For other nucleophilic aromatic substitution reactions of perfluoro-

⁽¹⁰⁾ For other nucleophilic aromatic substitution reactions of perfluoro-benzenes, see: Ingemann, S.; Nibbering, N. M. M.; Sullivan, S. A.; DePuy, C. H. J. Am. Chem. Soc. 1982, 104, 6520. (11) (a) The ion $[(CH_2CO_2H)^- + C_6F_6]$ is also noted: branching ratio $[(CH_2CO_2H)^- + C_6F_6]$. C₆F₅CH₂CO₂⁻ = 1:2. (b) CH₃CO₂⁻ reacts with C₆F₆ to give only $[CH_3CO_2^- + C_6F_6]$. (12) (a) The ion $[(CH_2CO_2H)^- + SO_2]$ is also noted: branching ratio $[(CH_2CO_2H)^- + SO_2]$:HOCO₂⁻ = 4:4:1. (b) CH₃CO₂⁻ reacts with SO₂ to give only $[CH_3CO_2^- + SO_2]$. (13) Bowie, J. H.; Blumenthal, T. J. Am. Chem. Soc. 1975, 97, 2959. Szuleiko, J. E. Bowie, I. H. Howe, I. Beynon, I. H. Int. I. Mass Spectrom

Szulejko, J. E.; Bowie, J. H.; Howe, I.; Beynon, J. H. Int. J. Mass Spectrom. Ion Phys. 1980, 34, 99.