for assistance with NMR experiments.

Supplementary Material Available: Tables of crystal data, bond distances and angles, final fractional coordinates, and thermal parameters (8 pages); a listing of observed and calculated structure factors (8 pages). Ordering information is given on any current masthead page.

Characterization of a Stable (η^3-Indenyl)iron Metalate, (η^3-C_9H_7)Fe$(CO)_3^-$. A Role for η^3–η^5 Indenyl Ligand Tautomerization during Alkyl–CO Insertion Reactions

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Summary: The (η^5-indenyl)iron metalate (η^5-In)Fe$(CO)_2^-Na^+$ irreversibly associates carbon monoxide in THF solution and affords the stable CO adduct (η^3-In)Fe$(CO)_3^-Na^+$, which was isolated (77% yield) as its $(Ph_3P)_2N^+$ salt, **4**-PPN$^+$. A single-crystal X-ray diffraction study established the η^3-indenyl coordination: the dihedral fold angle, between planes defined by the η^3-carbons and by the In benzenoid ring, is 22°. In solution, IR and ^{1}H and ^{13}C NMR spectral studies demonstrated that the η^3-indenyl structure endures: its $^{13}C\{^1H\}$ NMR spectrum, for example, exhibits a 29 ppm downfield shift for the ipso C3a,C7a carbons (vs. those on Na^+In^-). Methylating **4**-PPN$^+$ with CH_3I cleanly affords mixtures of (η^5-In)$(CO)_2FeCOCH_3$ and In$(CO)_2FeCH_3$. The yield of the former increases with CO pressure (to 42% at 5.3 atm). Arguments are presented for intermediacy of (η^3-In)$(CO)_3FeCH_3$ in this reaction and for the η^3-to-η^5 indenyl ligand tautomerization driving the alkyl–CO migratory insertion.

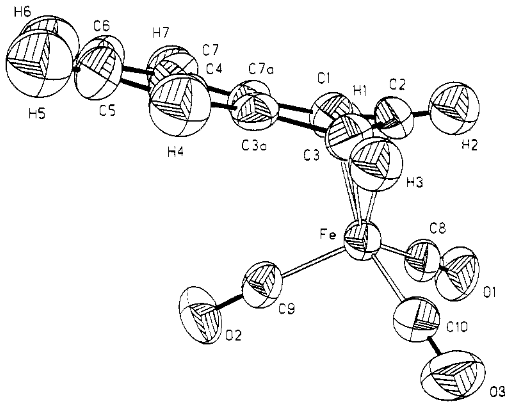
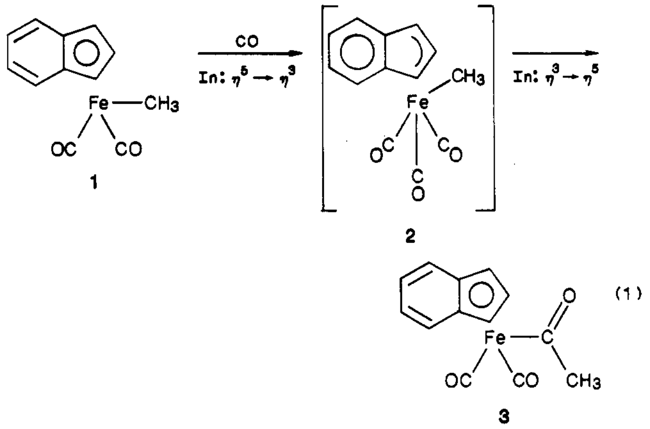


Figure 1. ORTEP plot of 4. Selected structural parameters: Fe–C1, 2.183 (5) Å; Fe–C2, 1.996 (5) Å; Fe–C3, 2.192 (5) Å; Fe–C3a, 2.885 (6) Å; Fe–C7a, 2.869 (7) Å; C1–C2, 1.416 (7) Å; C2–C3, 1.407 (7) Å; C3–C3a, 1.456 (7) Å; C3a–C7a, 1.397 (6) Å; C1–C7a, 1.469 (7) Å; Fe–C8, 1.742 (5) Å; Fe–C9, 1.779 (5) Å; Fe–C10, 1.752 (6) Å; C8–Fe–C10, 93.3 (2)°; C8–Fe–C9, 108.4 (2)°; C9–Fe–C10, 103.2 (2)°.

The η^5-indenyl ligand (In), serving as a replacement for its η^5-cyclopentadienyl (Cp) counterpart, provides a means of promoting associative reactions at the metal center.[1] Reversible slippage of the metal across the five-membered ring[2] thus can form a more reactive η^3-indenyl tautomer that has both benzenoid resonance stablization and an accesible coordination site.[3] Structural studies on a number of η^5-In complexes indeed indicate a minor "slip-fold" distortion already favoring a metal shift away from the indenyl central carbon–carbon bond.[2b,4] Three other coordinatively saturated compounds having η^3-In coordination have been structurally characterized.[5,6] In previous studies, we observed that substituting an η^5-In for η^5-Cp on iron alkyl complexes greatly facilitates their carbonylation (eq 1),[7a] which was attributed to the In ligand promoting an associative reaction (e.g., giving **2**). We now



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[2] (a) Albright, T. A.; Hofmann, P.; Hoffmann, R.; Lillya, C. P.; Dobosh, P. A. *J. Am. Chem. Soc.* **1983**, *105*, 3396. (b) Faller, J. W.; Crabtree, R. H.; Habib, A. *Organometallics* **1985**, *4*, 929.

[3] Corresponding η^3-Cp complexes are plausible intermediates in certain ligand substitution reactions,[1g,3a] but such intermediates are expected to be higher energy species compared to their η^3-In analogues.[3b] (a) Rerek, M. E.; Basolo, F. *Organometallics* **1983**, *2*, 372. Casey, C. P.; O'Connor, J. M.; Haller, K. J. *J. Am. Chem. Soc.* **1985**, *107*, 1241. Yang, G. K.; Bergman, R. G. *Organometallics* **1985**, *4*, 129 and references cited. (b) Belmont, J. A.; Wrighton, M. S. *Organometallics* **1986**, *5*, 1421.

[4] (a) Baker, R. T.; Tulip, T. H. *Organometallics* **1986**, *5*, 839. (b) Allen, S. R.; Baker, P. K.; Barnes, S. G.; Bottrill, M.; Green, M.; Orpen, A. G.; Welch, A. J. *J. Chem. Soc., Dalton Trans.* **1983**, 927. (c) Honan, M. B.; Atwood, J. L.; Bernal, I.; Herrmann, W. A. *J. Organomet. Chem.* **1979**, *179*, 403.

[5] (a) (η^5-In)(η^3-In)W$(CO)_2$, fold angle = 26°: Nesmeyanov, A. N.; Ustynyuk, N. A.; Makarova, L. G.; Andrianov, V. G.; Struchkov, Y. T.; Andrae, S.; Ustynyuk, Y. A.; Malyugina, S. G. *J. Organomet. Chem.* **1978**, *159*, 189. (b) (η^3-In)Ir$(PMe_2Ph)_3$, fold angle = 28°: Merola, J. S.; Kacmarcik, R. T.; Van Engen, D. *J. Am. Chem. Soc.* **1986**, *108*, 329. (c) (η^5-In)(η^3-In)V$(CO)_2$, fold angle = 12°: Kowaleski, R. M.; Rheingold, A. L.; Trogler, W. C.; Basolo, F. *J. Am. Chem. Soc.* **1986**, *108*, 2461.

[6] Existence of several other η^3-In complexes has been inferred from spectroscopic measurements.[1f,3b] (a) Kohler, F. M. *Chem. Ber.* **1974**, *107*, 570. (b) Nakasuji, K.; Yamaguchi, M.; Murata, I.; Tatsumi, K.; Nakamura, A. *Organometallics* **1984**, *3*, 1257.

[7] (a) Forschner, T. C.; Cutler, A. R. *Organometallics* **1984**, *4*, 1247. (b) Forschner, T. C.; Cutler, A. R. *Inorg. Chim. Acta* **1985**, *102*, 113.

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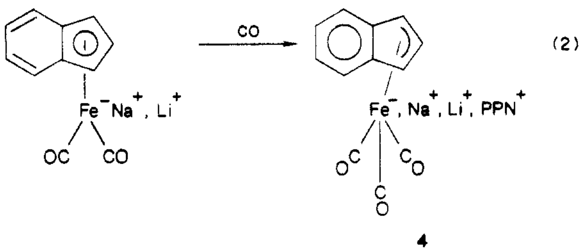
report the synthesis and full characterization of an (η^3-In)Fe complex and the use of its η^3–η^5 in ligand tautomerization to drive an alkyl–CO migratory insertion.

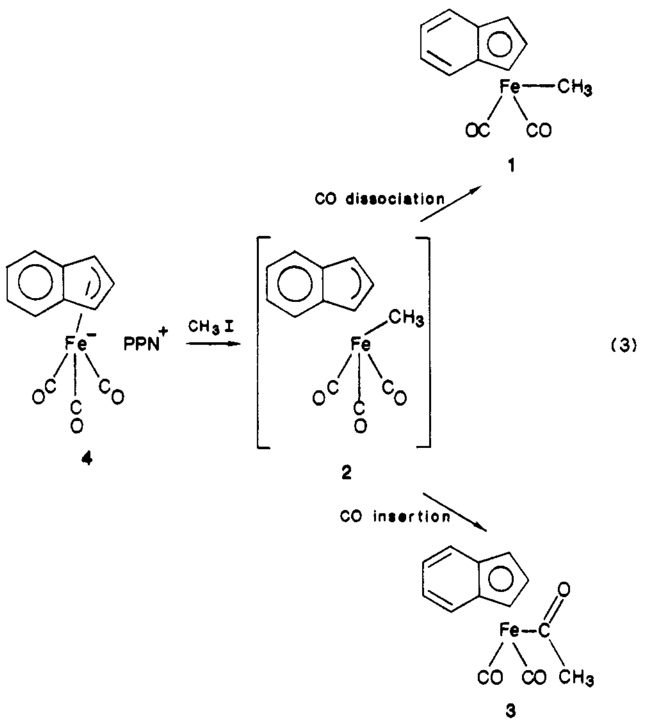
Treating a THF solution of (η^5-In)Fe(CO)$_2^-$Na$^+$ [7b] with carbon monoxide (1 atm) rapidly produced a lighter yellow-brown solution containing (η^3-In)Fe(CO)$_3^-$ (4) (eq 2).



This transformation is complete after a few minutes (as judged by IR spectral monitoring),[8] and metathesis with PPN$^+$Cl$^-$ [PPN$^+$ = (PPh$_3$)$_2$N$^+$] in THF–ether affords stable **4**-PPN$^+$ as brown needles (77% yield).[9] The carbonylation is irreversible: either applying evacuation–N$_2$ flush cycles over THF solutions of **4**-Na$^+$ or **4**-PPN$^+$ or evacuating solid **4**-PPN$^+$ failed to dislodge CO and regenerate starting (η^5-In)Fe(CO)$_2^-$.

The η^3-In coordination on **4**-PPN$^+$ was confirmed by a single-crystal X-ray diffraction study of its mono THF solvate.[10] An ORTEP plot for the organoiron anion (Figure 1) depicts overall pseudotetrahedral coordination of iron to the nonplanar 1–3-η-indenyl system and to three terminal (linear) carbonyls.[11] No intermolecular interactions with either counterion or THF are evident. The extreme slip distortion or η^3-allyl–ene bonding to the In ligand[5] is further defined by values of (a) 0.75 Å for the difference between the averages of the metal–carbon distances to C$_1$, C$_2$, and C$_3$ and those to ring-junction carbons C3a and C7a and (b) 22° between the planes defined by the η^3-carbons and by the In benzenoid ring.[2b,4]

The η^3-In structure of **4**-PPN$^+$ endures in solution. Its IR spectrum, for example, closely resembles that of (η^3-C$_3$H$_5$)Fe(CO)$_3^-$Na$^+$, although IR spectra of **4**-Na$^+$ and **4**-Li$^+$ further indicate the absence of significant ion pairing.[12] The ^{1}H NMR spectrum of **4**-PPN$^+$,[9] exhibiting an upfield shift for the H1,3 doublet and downfield shift for H$_2$, is consistent with the presence of an η^3-In complex.[5b] [Corresponding indenyl resonances for (η^5-In)(CO)$_2$Fe-alkyl complexes occur at δ 4.5 and 5.6, respectively.[7b]] An especially sensitive probe for slip-fold distortion of the In ligand, and thus reduced bonding between the iron and ring-junction carbons C3a,C7a, corresponds to the ^{13}C NMR chemical shifts of these carbons.[4a,5a,6a] Their observed 29 ppm downfield shift on **4**-PPN$^+$ (vs. those on Na$^+$In$^-$ at δ 128) also indicates localized η^3-In bonding.



Alkylating **4**-PPN$^+$ with methyl iodide affords mixtures of the (η^5-indenyl)iron methyl (**1**) and acetyl (**3**)[7b] complexes (eq 3). Yields of the acetyl product **3** increase with CO pressure: 13% **3**, 80% **1** (1 atm of N$_2$); 26% **3**, 71% **1** (1 atm of CO); 42% **3**, 52% **1** (5.3 atm of CO).[15] Attempts at directly observing **2**, the presumed intermediate from methylating **4**,[16] proved unsuccessful. At −15 °C, the reaction between **4**-PPN$^+$ and CH$_3$I in CD$_3$CN was sluggish; NMR spectral monitoring nevertheless detected only **4** plus **1** and **3**.[17] A plausible interpretation is that al-

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[8] CpFe(CO)$_2^-$Na$^+$ or CpFe(CO)$_2^-$Li$^+$ in THF solutions do not react with CO (80 psig). Also, InFe(CO)$_2^-$Na$^+$ is inert toward PPh$_3$ or P(OMe)$_3$. The high stability of (η^3-In)Fe(CO)$_3^-$ (**4**) presumably derives from charge delocalization over three π-acceptor carbonyls. One other η^3-In complex, (η^3-In)Ir(PMePh$_2$)$_3$,[5b] has been obtained by incorporating another ligand onto its η^5-In precursor.

[9] **4**-PPN$^+$: IR (THF) ν(CO) 1951, 1871, 1852 cm^{-1}; ^{1}H NMR (CD$_3$CN) δ 7.49 (m, 31 H, PPN$^+$ and In H2), 7.6 (H2, corresponding maximum observed NOE enhancement in ^{13}C{^{1}H} double irradiation), 6.03 and 5.92 (AA′BB′, 4 H, In:benzo), 3.54 (d, J = 3.0 Hz, 2 H, H1,3); ^{13}C{^{1}H} (APT) (CD$_3$CN) δ 223.4 (CO), 157.3 (C3a,7a), 120.0 and 113.3 (C4–C7), 91.3 (C2), 55.6 (C1,3). Anal. Calcd for mono THF solvate C$_{52}$H$_{45}$FeNO$_4$P$_2$: C, 72.13; H, 5.25. Found: C, 72.28; H, 5.26.

[10] Crystal data for (In)Fe(CO)$_3^-$·PPN$^+$·OC$_4$H$_8$ (**4**): as brown plates by slow diffusion of hexane into a THF solution; 0.44 × 0.44 × 0.65 mm, monoclinic $P2_1/c$ (No. 14): a = 9.714 (1) Å, b = 19.980 (5) Å, c = 23.260 (4) Å; β = 101.78 (1)°; V = 4419 (1) Å^3; z = 4; ρ(calcd) = 1.301 g cm^{-3}; Nicolet R3m diffractometer; μ = 4.55 cm^{-1} (Mo Kα): 0 °C; $2\theta_{max}$ = 45°: N_{refl} = 6826, ($I > 3\sigma_I$) = 4285; R = 0.054, R_w = 0.056; heavy-atom solution, blocked cascade refinement, all non-hydrogen atoms anistropic, all hydrogen atoms as idealized, isotropic contributions; SHELXTL (Rev. 5.1) computer programs (Nicolet Corp., Madison, WI).

[11] For a related structure, (η^3-C$_7$H$_7$)Fe(CO)$_3^-$AsPh$_4^+$: Sepp, E.; Pürzer, A.; Thiele, G.; Behrens, H. *Z. Naturforsch., B: Anorg. Chem., Org. Chem.* **1978**, *33B*, 261.

[12] (η^3-C$_3$H$_5$)Fe(CO)$_3^-$Na$^+$:[13] IR (THF) ν(CO) 1937, 1854, 1807 cm^{-1}. **4**-Na$^+$: IR (THF) ν(CO) 1949, 1871, 1850 cm^{-1}. **4**-Li$^+$: IR (THF) ν(CO) (THF) 1950, 1870, 1852 cm^{-1}. Ion pairing for **4**-Na$^+$ or **4**-Li$^+$ would engender additional absorptions; those corresponding to an isocarbonyl interaction would shift to lower energies.[14] Thus (η^5-In)Fe(CO)$_2^-$Na$^+$ [ν(CO) 1882, 1816 and 1866, 1779 cm^{-1} (THF)] contains a 40:60 mixture of tight ion pairs (Fe–Na) and of species containing ion pairing through the terminal carbonyl (i.e., isocarbonyl interaction), respectively. (These assignments rest on those made for the analogous CpFe(CO)$_2^-$Na$^+$.[14b])

[13] Gubin, S. P.; Denisovich, L. I. *J. Organomet. Chem.* **1968**, *15*, 471. Nesmeyanov, A. N.; Kritskaya, I. I. *J. Organomet. Chem.* **1968**, *14*, 387. Also see IR data for (cycloheptadienyl)Fe(CO)$_3^-$, which presumably has an 1–3-η-C$_7$H$_9$ ligand: Williams, G. M.; Rudisill, D. E. *J. Am. Chem. Soc.* **1985**, *107*, 3357.

[14] (a) Darensbourg, M. Y. *Prog. Inorg. Chem.* **1985**, *33*, 221. (b) Pannell, K. H.; Jackson, D. *J. Am. Chem. Soc.* **1976**, *98*, 4443. Nitay, M.; Rosenblum, M. *J. Organomet. Chem.* **1977**, *136*, C23.

[15] Yields of **1** and **3** are after column chromatography; no other organometallic products were detected. Reactions of **4**-Li$^+$ and **4**-Na$^+$, also in THF, with CH$_3$I gave identical results. Methyl complex **1** neither carbonylates to **3** under these conditions[7a] nor does it react with **4**. The product of the instantaneous reaction between (In)Fe(CO)$_2^-$ and **1**, a fully characterized binuclear acetyl anion (In)$_2$(CO)$_3$Fe$_2$(COCH$_3$)$^-$, was not detected in these studies (Forschner, T. C.; Cutler, A. R., to be submitted for publication).

[16] Assignment of an analogous "slipped-Cp" structure, (η^3-Cp)(CO)$_3$FeCH$_3$, to the product originating during photolysis of Cp(CO)$_2$FeCH$_3$ in a CO matrix[16a] has been disputed.[16b] (a) Fettes, D. J.; Narayanaswamy, R.; Rest, A. J. *J. Chem. Soc., Dalton Trans.* **1981**, 2311. Mahmoud, K. A.; Rest. A. J.; Alt, H. G. *Ibid.* **1985**, 1365. (b) Blaha, J. P.; Wrighton, M. S. *J. Am. Chem. Soc.* **1985**, *107*, 2694.

[17] Reaction between **4**-PPN$^+$ and Ph$_3$SnCl in THF solution produces only (η^5-In)(CO)$_2$FeSnPh$_3$ (77% isolated yield), which is a known compound: Faller, J. W.; Johnson, B. V.; Schaeffer, C. D., Jr. *J. Am. Chem. Soc.* **1976**, *98*, 1395.

kylation of 4 occurs in a rate-limiting step to give 2, which then either rapidly dissociates CO (leaving 1) or migrates methyl to ligated CO (giving 3).[18] Studies by Belmont and Wrighton[3b] have established that η^3- to-η^5 indenyl tautomerization can drive CO dissociation, and it is likely that the alkyl–CO migratory insertion also benefits from this indenyl transformation.

Acknowledgment. Continuing support from the Department of Energy, Office of Basic Energy Sciences, is gratefully acknowledged. We also thank Professor M. Wrighton and Dr. R. T. Baker for supplying preprints of ref 3b and 4a.

Registry No. 1, 71498-19-6; 3, 96412-64-5; 4-PPN$^+$, 106799-48-8; 4-Na$^+$, 106799-49-9; 4-Li$^+$, 106799-50-2; 4-PPN$^+$·OC$_4$H$_8$, 106820-58-0; (η^5-In)Fe(CO)$_2^-$Na$^+$, 52326-30-4.

Supplementary Material Available: ORTEP drawings, a unit cell diagram, and tables of bond distances, bond angles, anisotropic thermal parameters, H-atom coordinates, and fractional coordinates (12 pages). Ordering information is given on any current masthead page.

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[18] Alkyl migration apparently predominates for the ruthenium analogue of 2. Reducing [In(CO)$_2$Ru]$_2$[19] with Na(Hg) in THF solution (and under 1 atm of CO) affords a red-yellow solution containing a metalate that we tentatively formulate as (η^3-In)Ru(CO)$_3^-$Na$^+$: IR ν(CO) 1998, 1946 (br) cm^{-1}. Attempts at isolating and purifying it thus far have proved unsuccessful. Treating these solutions, nevertheless, with CH$_3$I (1 atm N$_2$) affords (η^5-In)(CO)$_2$RuCOCH$_3$[20] (79% isolated yield) and only a trace of the corresponding methyl compound.

[19] McArdle, P. *J. Chem. Soc. A* **1970**, 2128. Humphries, A. P.; Knox, S. A. R. *J. Chem. Soc., Chem. Commun.* **1973**, 326. Gansow, O. A.; Burke, A. R.; Vernon, W. D. *J. Am. Chem. Soc.* **1976**, *98*, 5817.

[20] For (In)(CO)$_2$RuCOCH$_3$ (yellow crystals, decomp pt 124 °C): IR (CH$_2$Cl$_2$) ν(CO) 2030, 1969, 1646 cm^{-1}; ^{1}H NMR (CDCl$_3$) δ 7.43, 7.13 (AA′BB′, 4 H, In:benzo), 5.97 (d, J = 3.0 Hz, 2 H, H1,3), 5.53 (t, J = 3.0 Hz, H2), 2.44 (s, 3 H, COCH$_3$). Anal. Calcd for C$_{13}$H$_{10}$O$_3$Ru: C, 49.56; H, 3.19. Found: C, 49.48; H, 3.32.

Observation of a Facile Isomerization of an Early-Transition-Metal η^2-Iminoacyl Group via a 1,2-Hydrogen Shift

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Summary: The insertion of 2,6-dimethylphenyl isocyanide (ArNC) into the metal–carbon bonds of the complexes Cp$_2$M(CH$_2$-py-6-Me)$_2$ (M = Zr, Hf; CH$_2$-py-6-Me = 2-(6-methylpyridyl)methyl) leads to the vinylamide complexes Cp$_2$M(ArNCH=CH-py-6-Me)(CH$_2$-py-6-Me) and Cp$_2$M(ArNCH=CH-py-6-Me)$_2$. The vinylamide ligand is formed by a facile 1,2-hydrogen shift in intermediate η^2-iminoacyl complexes.

Early-transition-metal,[2] lanthanide,[3] and actinide η^2-acyl and η^2-iminoacyl groups have been shown to be important organometallic functionalities. Of particular synthetic interest has been their ability to undergo a range of carbon–carbon bond forming reactions.[5] We wish to report here our initial observations concerning a facile isomerization of a group 4 metal η^2-iminoacyl group into a vinylamide substituent, a reaction that essentially halts any further organometallic chemistry of this ligand.[6]

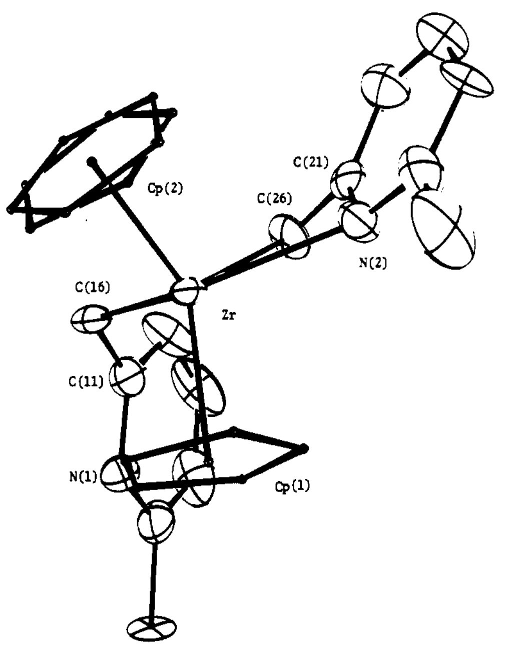
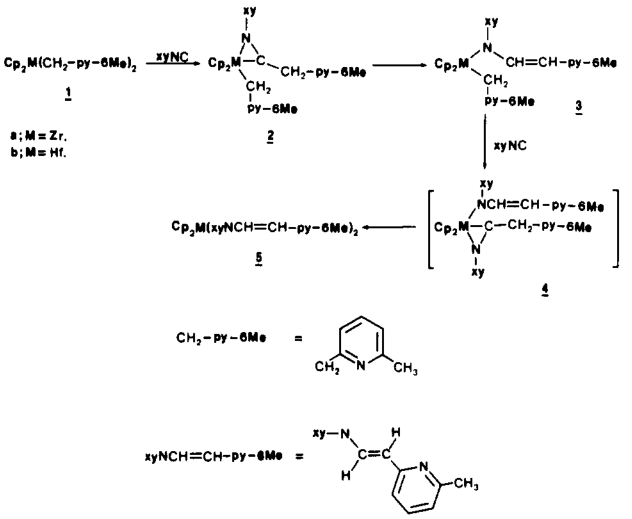


Figure 1. ORTEP view of Cp$_2$Zr(CH$_2$-py-6-Me)$_2$ (**1a**) with hydrogens omitted. Some selected bond distances (Å) and angles (deg): Zr–N(2) = 2.407 (4), Zr–C(16) = 2.422 (4), Zr–C(26) = 2.406 (5), Zr–CEN(1) = 2.214 (6), Zr–CEN(2) = 2.25 (1), Zr–C(16)–py = 121.5 (3), Zr–C(26)–py = 88.5 (3), CEN(1)–Zr–CEN(2) = 128.1 (3), C(16)–Zr–C(26) = 76.2 (2).

Scheme I



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[1] (a) Camille and Henry Dreyfus Teacher-Scholar, 1985–1990. (b) Fellow of the Alfred P. Sloan Foundation, 1986–1990.

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