Acknowledgment. Support of this work by the National Science Foundation (Grant RII-8610671) and the Commonwealth of Kentucky (EPSCoR Program) is gratefully acknowledged. We thank Wesley C. Roberts (an NSF-REU summer fellow) for synthetic assistance. Partial support of this work by the Department of Energy, Division of Chemical Sciences (Office of Basic Energy Sciences) is also gratefully acknowledged.

Isolation of a Bis(η^4-arene)dipalladium(I) Complex during the Annelation of Palladated Aryl Groups with Diphenylacetylene[1]

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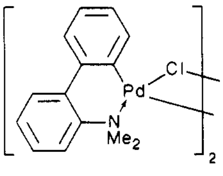
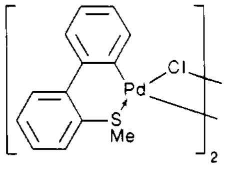
Summary: The palladated aryl units obtained via cyclopalladation of methyl 2-biphenylyl sulfide or *N*,*N*-dimethyl-2-biphenylamine undergo annelation reactions with diphenylacetylene. From the reaction of the sulfur containing palladocycle with the alkyne, an organometallic intermediate has been isolated in which two arene moieties are η^4-coordinated to a dipalladium(I) unit.

In the final stage of palladium-mediated stoichiometric or catalytic reactions in organic synthesis, the metal is usually recovered as palladium(II) or palladium(0) through reductive elimination reactions during which the metal is reduced by two-electron processes.[2] We wish here to report a rare example of such a reaction in which the metal is found as a palladium(I) complex together with the functionalized organic substrate.

We have shown earlier that the reactions between cyclopalladated compounds and internal alkynes can afford selectively heterocyclic compounds.[3] While investigating the potentiality of this reaction for the purpose of organic synthesis, we became interested in the behavior of such compounds in which the palladium atom is part of six-membered rings.[4] The new compounds **1** and **2** have thus been synthesized in almost quantitative yields through classical palladation[5] of the corresponding ligands using palladium acetate as the metalating agent followed by metathesis of the acetato groups with lithium chloride. The chloride-bridged dimer **1** was converted in situ into the cationic species **3** by abstracting the chloride ion with $AgSO_3CF_3$ in a THF solution. After removal of AgCl and the solvent, the residue thus obtained was treated in CH_2Cl_2 with excess of diphenylacetylene (PhC≡CPh:**3** > 4) to afford after 5 min of stirring at room temperature a brown solution from which compound **5** was quantitatively precipitated by addition of *n*-pentane (see Scheme I). Analytical data indicated that two alkyne units have been incorporated per palladium atom. The ^1H NMR spectrum of **5** displayed moreover characteristic resonance patterns for the four protons of the cyclohexadienyl unit η^3-bonded to Pd.[6] A related spirocyclic organopalladium compound has been fully characterized recently in our laboratory.[4] The molecule of water in **5** (detected by NMR, IR, and microanalysis) comes most probably from the silver triflate used, a feature that has already been observed in similar cationic compounds.[4]


1


2

When the cationic compound **4** (synthesized through addition of $AgBF_4$ to **2** in a CH_2Cl_2/MeCN solution[3]) was heated with a slight excess of diphenylacetylene (PhC≡CPh:**4** = 2.1) in chlorobenzene at 90 °C, it afforded a red solution after ca. 10 min. Filtration of this solution through a Celite column, removal of the chlorobenzene under reduced pressure, and washing the solid thus obtained with pentane afforded deep red **6** in 86% yield. The ^1H NMR spectrum of this compound did not allow the detection of any other organometallic species; thus the formation of **6** was indeed quantitative. The molecular structure of this compound was ascertained by an X-ray diffraction study[7] since its NMR spectra were of little help in order to determine its geometry. Crystals of **6** suitable for the X-ray analysis (which contain four molecules of acetone of crystallization) were obtained from an acetone/diethyl ether solution. The cationic part of the molecule is shown in Figure 1. It reveals that the former palladated aryl ring in **4** has been annelated by two diphenylacetylene reagents to form a pentasubstituted naphthalene derivative.[8] Two aryl rings of this latter ligand are both η^4-coordinated to a dipalladium unit. This gives rise to a so-called sandwich

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(1) Reactivity of Cyclopalladated Compounds. 20. Part 19, see: van Koten, G.; Maassarani, F.; Pfeffer, M. *Organometallics* **1989**, *8*, 871.

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(4) Maassarani, F.; Pfeffer, M.; Le Borgne, G. *Organometallics* **1987**, *6*, 2043.

(5) See, for example: Hiraki, K.; Fuchita, Y.; Takechi, K. *Inorg. Chem.* **1981**, *20*, 4316.

(6) All new compounds gave satisfactory elemental analyses. Selected spectroscopic data are as follows: ^1H NMR ($CDCl_3$, 293 K, 200.13 MHz): **5**, δ 8.62–6.31 (m, 24 H, aromatic), 6.13 (d, 1 H, H^1), 6.01 (dd, 1 H, H^3, ^3J (H^2H^3) = 5.7 Hz), 5.57 (d, 1 H, H^4, ^3J (H^3H^4) = 8.0 Hz), 4.80 (t, 1 H, H^2, ^3J (H^1H^2) = 6.1 Hz), 2.78 and 1.49 (2s, 6 H, NMe_2), 1.75 (s, 2 H, H_2O) (H^1–H^4 represent the protons of the hexadienyl unit in **5**); **6**, δ 7.80–5.52 (28 H, aromatic), 2.29 (s, 3 H, SMe); **7**, δ 7.62–6.58 (m, 28 H, aromatic + NH), 2.29 (s, 6 H, NMe_2); **8**, δ 7.71–6.60 (m, 27 H, aromatic), 2.24 (s, 3 H, SMe). IR (KBr pellets): **7**, 3040 cm^{-1} (ν(NH)). MS (FAB): **7**, *m/z* 553 (552, $M^+ - H$); **8**, *m/z* 554 (554, M^+), 539 (M – Me), 507 (M – SMe).

(7) Crystal structure of **6** $((CH_3)_2CO)_4$: triclinic; space group $P\bar{1}$; a = 15.776 (6) Å, b = 19.786 (6) Å, c = 15.486 (6) Å, α = 91.79 (2)°, β = 114.88 (2)°, γ = 106.79 (2)°; V = 4132 Å^3, Z = 2; ρ_{calcd} = 1.234 g cm^{-3}; F_{000} = 1564; λ = 1.5418 Å; μ = 45.446 cm^{-1}; 8199 independent ±*hkl* reflections measured (Philips PW 1100/16 diffractometer, graphite-monochromated radiation, $\theta/2\theta$ flying step scan, $3° < \theta < 50°$ at –100 °C). The structure was solved by using Patterson and Fourier methods and refined by full-matrix least squares by using 6918 observed reflections ($I > 3\sigma(I)$). Hydrogen atoms were introduced in structure factor calculations at their computed coordinates (C–H = 0.95 Å, B(H) = 1.3B_{eqv} (C)) but not refined. The fluorine atoms of one of the BF_4^- moieties are disordered over two positions in the ratio 1/1 (difference Fourier relative peak heights). Empirical absorption corrections were applied. $R(F)$ = 0.067; $R_w(F)$ = 0.110. Further details of the structure investigation can be obtained from the authors.

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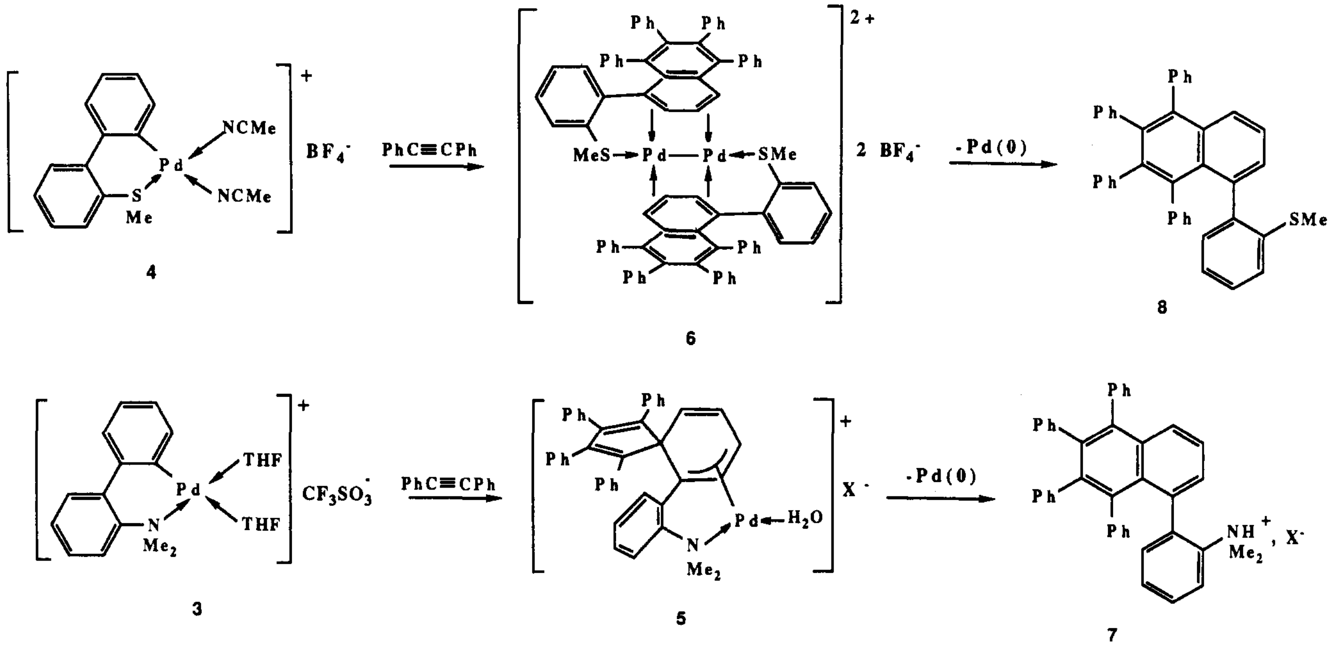
Scheme I. Reaction Path for the Annelation Reaction of the Palladated Aryl Units in Compounds 3 and 4



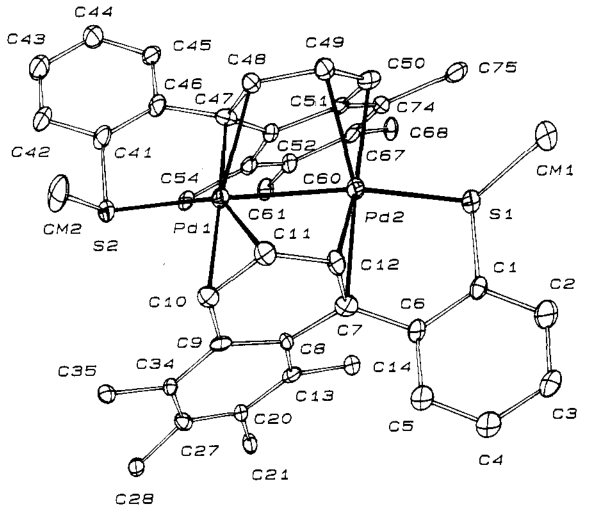


Figure 1. ORTEP view of the cationic part of 6: only the ipso carbon atoms of the peripheral phenyl rings are represented; hydrogen atoms are omitted. Ellipsoids are scaled to enclose 50% of the electron density. selected bond distances (Å): Pd1–Pd2, 2.603 (1); Pd1–S2; 2.339 (2); Pd2–S1, 2.333 (2); Pd1–C10, 2.25 (2); Pd1–C11, 2.34 (2); Pd1–C47, 2.26 (2); Pd1–C48, 2.17 (2); Pd2–C7, 2.25 (2); Pd2–C12, 2.20 (2); Pd2–C49, 2.32 (1); Pd2–C50, 2.22 (2); S1–CM1, 1.79 (1); S2–CM2, 1.83 (1); S(1)C(1), 1.76 (2), S2–C41, 1.78 (1). Bond angles (deg): Pd1–Pd2–S1, 166.34 (5); Pd2–Pd1–S2, 167.31 (6).

coordination around the palladium atoms. The coordination sphere of each metal is completed by coordination of a lone pair of the sulfur atoms. Thus each of the two annelated ligands are six-electron donors. Since the organodipalladium unit bears a 2+ charge, the two Pd must be in the +1 oxidation state. This is confirmed by the Pd(1)–Pd(2) distance (2.603 (1) Å) which is characteristic of a molecule containing a dipalladium(I) moiety.[9] Structurally characterized compounds in which the palladium is in π-interaction with the arenes are still very rare.[10] It is however worthwhile to mention the compound obtained more than 20 years ago by Allegra et al.,[11] during the reduction of $PdCl_2$ by aluminum in the presence of $AlCl_3$ in benzene. This compound is structurally related to compound 6 since it contains two benzene rings both η^4-coordinated to a dipalladium(I) unit. To the best of our knowledge no further example of palladium(I) compounds stabilized by coordination to neutral arene units have been published in the literature since then.

Compounds 5 and 6, though reasonably stable in the solid state, both evolve in solution above room temperature to afford the corresponding palladium-free ligands 7 and 8, respectively (see Scheme I). The structure of 8 was deduced from that of 6. Compound 7 was analyzed accordingly since the aromatic protons display an ^{1}H NMR spectrum very similar to that of 8. Moreover the IR spectrum of 7 showed an intense absorption at 3040 cm^{-1} (assigned to the ν(N–H) vibration) together with the absorptions characteristic of the triflate anion.

At this stage of our study it is tempting to anticipate that a concerted radical process would explain the formation of compound 6. Indeed a radical mechanism was proposed earlier for the annelation of aryl rings with different alkynes in presence of palladium acetate.[12] We have no direct proof as to decide whether the formation of 6 is preceded by an intermediate such as 5. However, we have observed that compound 5 can evolve at −20 °C to a deep red transient species, which might be analogous to 6. Although we have not been able to fully characterize this transient product yet, this observation is a good indication that rearrangement of the spiro complex 5 to the annelated product 7 might occur via a palladium(I) intermediate.

Work is in progress to demonstrate the generality of these findings to the reactions of related organopalladium compounds with alkynes.

Acknowledgment. The CNPq (Brazil) is thanked for a Fellowship (to J.D.) and the Commission of European

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Commities for financial support (Contract No. ST 2J-0090-1-F).

Registry No. **1**, 119326-02-2; **2**, 119326-03-3; **3**($\mathrm{CF_3SO_3}$), 119326-05-5; **4**($\mathrm{BF_4}$), 119326-07-7; **5**($\mathrm{CF_3SO_3}$), 119326-09-9; **6**($\mathrm{BF_4}$)$_2$, 119326-11-3; **6**($\mathrm{BF_4}$)$_2$·($\mathrm{(CH_3)_2CO}$)$_4$, 119326-12-4; **7**, 119297-92-6; **8**, 119297-93-7; PhC≡CPh, 501-65-5.

Supplementary Material Available: Tables of bond lengths, bond angles, atomic coordinates, and thermal parameters for **6** (18 pages); a listing of observed and calculated structure factors for **6** (28 pages). Ordering information is given in any current masthead page.

σ,π Complexes of Benzene[1]

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Summary: A series of electron-rich polymetallic complexes of benzene, namely, $\mathrm{C_6H_{6-\mathit{n}}Fp_\mathit{n}}$ and $(\eta^6\text{-}\mathrm{C_6H_{6-\mathit{n}}Fp_\mathit{n}})\mathrm{Cr(CO)_3}$ (Fp = $(\eta^5\text{-}\mathrm{C_5H_5})\mathrm{Fe(CO)_2}$ and n = 2 or 3), have been prepared. The X-ray crystal structure of $(\eta^6\text{-}1,3,5\text{-}\mathrm{C_6H_3Fp_3})\mathrm{Cr(CO)_3}\cdot\mathrm{CH_2Cl_2}$ confirms that the arene ring is σ-bound to iron and π-bound to chromium.

Over the last several years there has been growing interest in the synthesis of polymetallic complexes whose metal centers are joined by polyfunctional organic ligands having delocalized π systems.[2] Although most of these ligands contain an aromatic core (e.g. pyrazine, diaminobenzene, dicyanobenzene, diethylnylbenzene, etc.), remarkably few contain direct metal to arene–carbon σ bonds.[3] I now wish to report the synthesis of a number of such polymetallic complexes.

During the course of our studies, we have found that for many organometallic fragments such as $(\eta^5\text{-}\mathrm{C_5H_5})\mathrm{Fe(CO)_2}$, Fp, direct metathesis reactions, i.e. eq 1 and 2, fail to

$$2\mathrm{NaFp} + 1,4\text{-}\mathrm{C_6H_4Br_2} \xrightarrow[-78\ ^\circ\mathrm{C}]{\mathrm{THF}} 1,4\text{-}\mathrm{C_6H_4Fp_2} + 2\mathrm{NaBr} \tag{1}$$

$$2\mathrm{FpI} + 1,4\text{-}\mathrm{C_6H_4Li_2} \xrightarrow[-78\ ^\circ\mathrm{C}]{\mathrm{Et_2O}} 1,4\text{-}\mathrm{C_6H_4Fp_2} + 2\mathrm{NaI} \tag{2}$$

produce the desired phenylene-bridged complexes in synthetically useful yields. Fortunately, they are readily preparable via acyl intermediates, i.e. eq 3 and 4. Thus,

$$2\mathrm{NaFp} + 1,4\text{-}\mathrm{C_6H_4(COCl)_2} \xrightarrow[-78\ ^\circ\mathrm{C}]{\mathrm{THF}} 1,4\text{-}\mathrm{C_6H_4(COFp)_2} + 2\mathrm{NaCl} \tag{3}$$

$$1,4\text{-}\mathrm{C_6H_4(COFp)_2} \xrightarrow[\mathrm{Bu_2O}]{\Delta} 1,4\text{-}\mathrm{C_6H_4Fp_2} + 2\mathrm{CO}\uparrow \tag{4}$$

addition of terephthaloyl chloride (1.10 g, 5.50 mmol) to a solution of NaFp (11.30 mmol)[4] in THF (60 mL) at −78 °C produces a tan precipitate. This solid may be collected by filtration in air, washed with $\mathrm{Et_2O}$ (50 mL) and $\mathrm{H_2O}$ (100 mL), and dried in vacuo to give 1,4-$\mathrm{C_6H_4(COFp)_2}$ (**1a**)[5] in 94% yield. Gentle reflux of a suspension of this diacyl complex (0.51 mmol) in $\mathrm{Bu_2O}$ (60 mL) for 5.5 h, removal of solvent in vacuo, and recrystallization of the resultant product from $\mathrm{CH_2Cl_2}$/hexanes results in the isolation of the desired complex 1,4-$\mathrm{C_6H_4Fp_2}$ (**2a**)[6] in 68% yield. The related complexes 1,3-$\mathrm{C_6H_4Fp_2}$ (**2b**) and 1,3,5-$\mathrm{C_6H_3Fp_3}$ (**2c**) may be prepared in a similar fashion from 1,3-$\mathrm{C_6H_4(COCl)_2}$ and 1,3,5-$\mathrm{C_6H_3(COCl)_3}$, respectively.[7] These new phenylene-bridged complexes are tan crystalline solids exhibiting remarkable thermal and air stability. Their spectroscopic characteristics[6,7] are analogous to those reported for related iron aryls[8] and indicate that they retain the substitution geometry of the parent aroyl chlorides.

Although these phenylene complexes are very sterically crowded, they retain the ability of free arenes to form π complexes with $\mathrm{Cr(CO)_3}$,[9] i.e. eq 5.

$$\mathrm{C_6H_4Fp_2} + \text{excess } \mathrm{Cr(CO)_6} \xrightarrow[\Delta]{\mathrm{Bu_2O}} (\eta^6\text{-}\mathrm{C_6H_4Fp_2})\mathrm{Cr(CO)_3} + \mathrm{CO}\uparrow \tag{5}$$

Thus, when a suspension of 1,4-$\mathrm{C_6H_4(COFp)_2}$ (0.50 g, 1.02 mol)[10] is refluxed with excess $\mathrm{Cr(CO)_6}$ (1.00 g, 4.54 mmol) in $\mathrm{Bu_2O}$ (50 mL) for 14 h, a yellow microcrystalline precipitate forms upon cooling to room temperature. This may be fractionally recrystallized from $\mathrm{CH_2Cl_2}$/hexanes to give the new po-

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(5) **1a**: IR ($\mathrm{CH_2Cl_2}$) ν_{CO} 2030 (m), 1962 (m), 1606 (w, br) cm^{-1}; fast atom bombardment mass spectrum, m/z 487 ($\mathrm{P^+}$ + H). Anal. Calcd for $\mathrm{C_{22}H_{14}O_6Fe_2}$: C, 54.37; H, 2.90; O, 19.75. Found: C, 54.51; H, 2.79; O, 20.03.

(6) **2a**: IR ($\mathrm{CH_2Cl_2}$) ν_{CO} 2014 (s), 1959 (s) cm^{-1}; high-resolution mass spectrum, m/z calcd 429.9591, found 429.9603; ^{1}H NMR ($\mathrm{(CD_3)_2SO}$) δ 6.95 (s, 4 H, $\mathrm{C_6\mathit{H}_4}$), 5.07 (s, 5 H, $\mathrm{C_5\mathit{H}_5}$) ppm. Anal. Calcd for $\mathrm{C_{20}H_{14}O_4Fe_2}$: C, 55.86; H, 3.28. Found: C, 56.30; H, 3.58.

(7) Yields: **1b**, 88%; **1c**, 80%; **2b**, 51%; **2c**, 30%; **3b**, 35%; **3c**, 15%. Analytical and Spectroscopic data for these products are presented in the supplementary material.

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(9) A wide variety of (arene)$\mathrm{Cr(CO)_3}$ compounds have been prepared and characterized, see for example: (a) Collman, J. P.; Hegedus, L. S.; Norton, J. R.; Finke, R. G. *Principles and Applications of Organotransition Metal Chemistry*; University Science Books: Mill Valley, CA, 1987. (b) Sneedon, R. P. A. *Organochromium Compounds*; Academic Press: New York, 1975.

(10) These $\mathrm{Cr(CO)_3}$ adducts are best prepared from the phenylene complexes (i.e. 1,4-$\mathrm{C_6H_4Fp_2}$) generated in situ from the acyl intermediates (i.e. 1,4-$\mathrm{C_6H_4(COFp)_2}$).

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