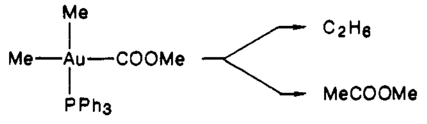
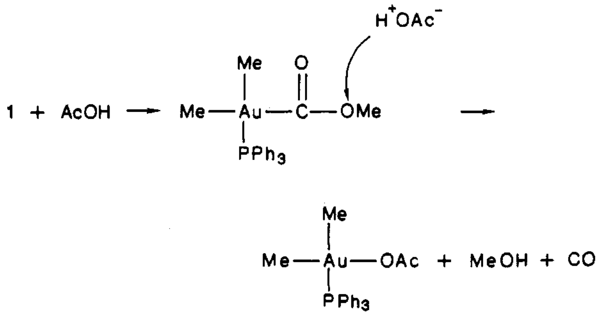
Thermolysis of 1 in C_6D_6 at 80 °C for 80 min resulted in the reductive elimination of both methyl acetate and ethane in ca. 1:1 ratio in the initial stages of the reaction (7% decomposition). Gold products are considered to be Au(I)Me(PPh_3) and Au(I)(COOMe)(PPh_3). Formation of the former was confirmed by ^1H NMR, but the latter seems to be unstable under the thermolysis conditions, since carbon monoxide and methanol were detected after the thermolysis. A prolonged thermolysis of 1 at 80 °C for 6 h gave trimethyl(triphenylphosphine)gold(III) (ca. 20%/1) accompanied by evolution of carbon monoxide in addition to the reductive elimination products, probably due to the concomitant decarbonylation of 1 giving an unstable dimethylmethoxogold(III) species (vide infra). The observed comparable rates of two possible reductive eliminations are noteworthy, since the electron-withdrawing methoxycarbonyl group is expected to be much more stable toward reductive elimination than methyl.[9] Enhanced reductive elimination in a metal–C(sp^2) bonded group has been recently invoked in reactions of organonickel(II),[10] -gold(III),[4c] and -ruthenium(II)[11] complexes. pπ-Orbitals of the sp^2 carbon might assist the reductive elimination process.[4c]



1

(3)

Acidolysis of 1 (0.027 mmol) with a stoichiometric amount of acetic acid (0.027 mmol) in C_6D_6 took place smoothly at room temperature to give *cis*-dimethyl(acetato)(triphenylphosphine)gold(III)[12] in quantitative yield with liberation of methanol and carbon monoxide.



(4)

Electrophilic attack of the acid at the methoxy group in 1 to liberate methanol, followed by decarbonylation from the resulting labile dimethylcarbonylgold(III) intermediate would give the reaction products. A mechanistic investigation of the insertion reaction is now in progress.

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Activation of a Fluorinated Carbon–Carbon Bond by Oxidative Addition of Tetrafluorocyclopropene to Platinum(0). The First Example of a Perfluorometallacyclobutene

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Summary: Carbon–carbon bond activation of perfluorocyclopropene (**3**) by $[Pt(C_2H_4)(PR_3)_2]$ (R = Ph, Me) affords perfluorometallacyclobutene products $[Pt(C_3F_4)(PR_3)_2]$ (**4a**, R = Ph; **4b**, R = Me), characterized in solution by a combination of $^{19}F\{^{31}P\}$, $^{19}F\{^{19}F\}$, and $^{31}P\{^{1}H\}$ NMR spectroscopic methods. No intermediate olefin complex could be detected spectroscopically. The triphenylphosphine complex **4a** has also been characterized in the solid state by a single-crystal X-ray diffraction study.

The transition-metal chemistry of cyclopropenes is extensive.[2] Both catalytic reactions to give organic products[3-7] and stoichiometric reactions to give organometallic complexes[8-24] are known. Metal-promoted activation of a carbon–carbon bond of the cyclopropene to give an intermediate metallacyclobutene (1), or its valence tautomeric vinylcarbene relative (2), has been proposed as a key step in many of these reactions. However, no examples of the direct formation of an isolable metallacyclobutene

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(1) (a) Dartmouth College. (b) University of Delaware.

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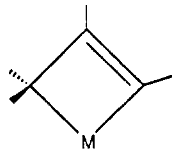
Table I. Atomic Coordinates ($\times 10^4$) and Isotropic Thermal Parameters ($Å^2 \times 10^3$) for 4a

	X	*Y*	*Z*	U^a
Pt	1281.3 (2)	6402.5 (1)	6857.2 (2)	34.6 (1)
P(1)	814 (2)	5730 (1)	8048 (1)	36 (1)
P(2)	−245 (2)	7143 (1)	6626 (2)	36 (1)
C(1)	2058 (7)	6847 (4)	5867 (6)	49 (3)
C(2)	2859 (6)	6371 (4)	5878 (6)	50 (3)
C(3)	2669 (6)	5873 (4)	6606 (5)	42 (3)
F(1)	1940 (4)	7399 (3)	5319 (4)	78 (2)
F(2)	3686 (4)	6325 (3)	5352 (4)	77 (2)
F(4)	3608 (4)	5760 (3)	7300 (3)	60 (2)
F(5)	2451 (4)	5219 (2)	6195 (3)	57 (2)
C(11)	2503 (4)	4749 (3)	8424 (3)	52 (3)
C12)	3382	4392	8992	62 (4)
C(13)	3718	4550	9968	61 (4)
C(14)	3175	5065	10375	65 (4)
C(15)	2296	5422	9806	48 (3)
C(16)	1960	5264	8831	38 (3)
C(21)	−435 (4)	4532 (3)	8176 (3)	50 (3)
C(22)	−1144	4404	7781	58 (3)
C(23)	−1575	3987	6791	63 (4)
C(24)	−1295	4497	6197	71 (4)
C(25)	−585	5025	6592	54 (3)
C(26)	−155	5043	7581	38 (3)
C(31)	−744 (4)	6030 (3)	9253 (4)	51 (3)
C(32)	−1123	6430	9936	67 (4)
C(33)	−542	7018	10290	72 (4)
C(34)	417	7207	9959	61 (4)
C(35)	795	6807	9276	49 (3)
C(36)	215	6218	8922	40 (3)
C(41)	−2263 (5)	6429 (3)	6179 (4)	65 (4)
C(42)	−3288	6193	6337	86 (5)
C(43)	−3669	6392	7161	84 (5)
C(44)	−3024	6827	7828	75 (4)
C(45)	−1998	7063	7670	57 (3)
C(46)	−1617	6864	6846	47 (3)
C(51)	−827 (3)	8424 (3)	7304 (4)	53 (3)
C(52)	−589	9019	7843	67 (4)
C(53)	496	9146	8343	71 (4)
C(54)	1344	8678	8304	66 (4)
C(55)	1107	8082	7765	50 (3)
C(56)	22	7956	7265	44 (3)
C(61)	−520 (4)	6834 (2)	4675 (4)	54 (3)
C(62)	−743	6975	3689	68 (4)
C(63)	−1017	7636	3365	69 (4)
C(64)	−1068	8156	4027	65 (4)
C(65)	−845	8014	5012	54 (3)
C(66)	−571	7353	5336	40 (3)

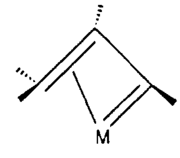
[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized U_{ij} tensor.

complex from the reaction of a simple cyclopropene with a transition-metal center have been reported, although this ligand framework has been constructed indirectly by the coupling of alkylidene ligands with alkynes.[25-27] We now report the first example of such a reaction, from platinum-induced activation of a fluorinated carbon–carbon bond.

Perfluorocyclopropene (**3**) was prepared by using a modification of the literature method,[28] by ultrasonically assisted dechlorination[29] of 1,2-dichloro-1,2,3,3-tetrafluorocyclopropane with Zn powder, and was stored and used as a dilute toluene solution.[30] The reaction of **3** with $[Pt(C_2H_4)(PR_3)_2]$ (R = Ph,[31] Me[32]) occurred rapidly in toluene at −78 °C to afford the tetrafluoroplatinacyclobutene complexes **4** in high yield.[33] Observation of the evolving reaction solution using low-temperature ^{19}F NMR spectroscopy showed smooth disappearance of the resonances of **3** with concomitant appearance of those of **4**. No intermediate species could be detected at −78 °C. The PPh_3 complex **4a** is air-stable in the solid state and in solution, but its PMe_3 analogue **4b** decomposes in solution on exposure to air. Complex **4b** can also be prepared by treatment of **4a** with excess PMe_3.



1



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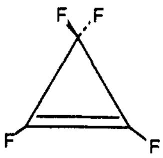
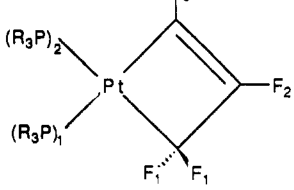
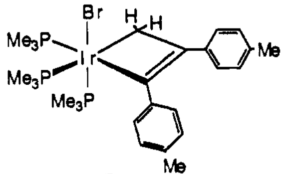


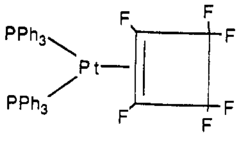
3



4 a. R = Ph
b. R = Me



5



6

Single crystals of **4a** were subjected to an X-ray diffraction study, which confirmed the proposed structure.[34]

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(30) **3** can be stored as a dilute toluene solution under dinitrogen at −78 °C for several months without loss or decomposition. *WARNING: Reference 28 states that tetrafluorocyclopropene is explosive and dangerously reactive toward some nucleophiles.* We have experienced no such difficulties with dilute solutions of **3**.

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(33) **4a** (81%): mp 175–178 °C dec; ^{19}F NMR (282 MHz; acetone-d_6) δ (upfield from internal $CFCl_3$) 88.1 (m, F_1), 98.4 (m, F_2), 121.7 (m, F_3), $J_{1,2} = 8$, $J_{1,3} = 23$, $J_{2,3} = 11$, $J_{Pt-F_1} = 410$, $J_{Pt-F_2} = 540$, $J_{Pt-F_3} = 517$, $J_{P_1-F_2} = 15$, $J_{P_1-F_3} = 68$, $J_{P_2-F_1} = 44$, $J_{P_2-F_2} = 23$ Hz; ^1H NMR (300 MHz, acetone-d_6) δ 7.24–7.52 (30 H, Ph); $^{31}P\{^1H\}$ NMR (121 MHz, acetone-d_6) δ 21.8 (m, P_1), 16.8 (m, P_2), $J_{P_1-F_2} = 15$, $J_{P_1-F_3} = 68$, $J_{P_1-P_2} = 15$, $J_{P_2-F_1} = 44$, $J_{P_2-F_2} = 23$, $J_{Pt-P_1} = 2710$, $J_{Pt-P_2} = 2019$ Hz; MS, *m/e* 830 ($M^+ - H$). Anal. Calcd for $C_{39}H_{30}F_4P_2Pt$: C, 56.29; H, 3.60. Found: C, 55.89; H, 3.20. **4b** (75%): mp 125–127 °C; ^{19}F NMR (C_6D_6) δ 90.0 (m, F_1), 99.4 (m, F_2), 125.1 (m, F_3), $J_{1,2} = 9$, $J_{1,3} = 23$, $J_{2,3} = 11$, $J_{Pt-F_1} = 403$, $J_{Pt-F_2} = 466$, $J_{Pt-F_3} = 540$, $J_{P_1-F_1} = 3$, $J_{P_1-F_2} = 17$, $J_{P_1-F_3} = 74$, $J_{P_2-F_1} = 47$, $J_{P_2-F_2} = 26$, $J_{P_2-F_3} = 4$ Hz; ^1H NMR (C_6D_6) δ 0.86 (d, P_2Me_3), 1.03 (d, P_1Me_3), $J_{P_1-H_1} = 9.6$, $J_{P_2-H_2} = 9.4$, $J_{Pt-H_1} = 25.9$, $J_{Pt-H_2} = 20.5$ Hz; $^{31}P\{^1H\}$ NMR (C_6D_6) δ 22.3 (m, P_2), 20.2 (m, P_1), $J_{P_1-F_1} = 3$, $J_{P_1-F_2} = 17$, $J_{P_1-F_3} = 74$, $J_{P_1-P_2} = 23$, $J_{P_2-F_1} = 47$, $J_{P_2-F_2} = 26$, $J_{P_2-F_3} = 4$, $J_{Pt-P_1} = 2504$, $J_{Pt-P_2} = 1989$ Hz. Anal. Calcd for $C_9H_{18}F_4P_2Pt$: C, 23.52; H, 3.95. Found: C, 23.41; H, 4.04.

(34) **4a**: colorless, monoclinic, $P2_1/n$, $a = 12.215$ (3), $b = 19.643$ (4), $c = 14.155$ (3) Å; $\beta = 101.51$ (2)°, $V = 3328$ (1) Å^3, $Z = 4$, D(calcd) = 1.660 g cm^{-3}, μ(Mo K_α) = 45.73 cm^{-1}, $T = 293$ K. Of 6312 absorption corrected reflections collected (Niclolet R3m diffractometer, $4° \le 2\theta \le 50°$), 5258 were independent ($R_{int} = 1.66\%$) and 3943 were observed ($\ge 5\sigma F_o$). The Pt atom was located by heavy-atom methods. Phenyl rings were constrained to rigid hexagons, and hydrogen atoms were idealized. With all non-hydrogen atoms isotropic: $R(F) = 3.61\%$, $R(wF) = 4.44\%$, GOF = 0.943, $\Delta/\sigma(\max) = 0.025$, $\Delta(\rho) = 0.78$ e Å^3, $N_o/N_v = 11.5$. All computations: SHELXTL(5.1), G. Sheldrick, Nicolet XRD, Madison, WI.

Organometallics 1988, *7*, 2241-2243

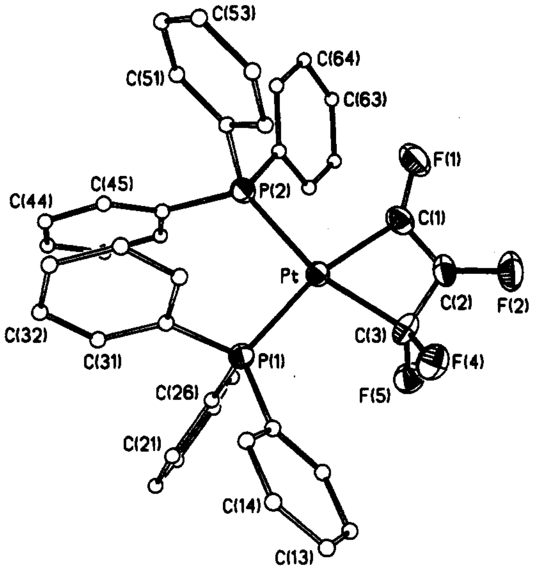

Figure 1. Molecular structure of complex **4a**. Carbon atoms in the phenyl rings are shown as spheres of arbitrary radius.

Table II. Selected Bond Lengths and Angles for Complex 4a

Selected Bond Lengths (Å)	
Pt-P(1) 2.302 (2)	Pt-P(2) 2.336 (2)
Pt-C(1) 2.039(9)	Pt-C(3) 2.079 (8)
C(1)-C(2) 1.351 (12)	C(2)-C(3) 1.474 (12)
C(1)-F(1) 1.325 (10)	C(2)-F(2) 1.373 (11)
C(3)-F(4) 1.371 (8)	C(3)-F(5) 1.414 (9)
Selected Bond Angles (deg)	
P(1)-Pt-P(2) 98.8 (1)	Pt-C(1)-C(2) 97.5 (6)
P(1)-Pt-C(1) 165.2 (2)	Pt-C(1)-F(1) 138.0 (6)
P(1)-Pt-C(3) 99.6 (2)	Pt-C(3)-C(2) 92.0 (5)
P(2)-Pt-C(1) 96.0 (2)	Pt-C(3)-F(4) 123.6 (5)
P(2)-Pt-C(3) 160.6 (2)	Pt-C(3)-F(5) 115.6 (5)
C(1)-Pt-C(3) 65.8 (3)	C(1)-C(2)-C(3) 104.7 (7)
F(1)-C(1)-C(2) 124.5 (8)	F(2)-C(2)-C(1) 130.5 (8)
F(2)-C(2)-C(3) 124.8 (7)	F(5)-C(3)-C(2) 111.0 (6)
F(4)-C(3)-C(2) 112.5 (6)	F(4)-C(3)-F(5) 102.1 (6)

The molecular structure of **4a** is shown in Figure 1. Fractional atomic coordinates are presented in Table I, and selected bond lengths and angles are assembled in Table II. The metallacyclobutene ring is planar, with no atom deviating more than 0.015 Å from the plane. There is a significant deviation from planarity in the coordination sphere of platinum, the metallacyclobutene plane being canted by 7.6° from the P(1)-Pt-P(2) plane. Similar deviations from planarity within the coordination sphere of the metal have been noted in saturated platinacyclobutanes, though the metallacyclobutane rings themselves in these Pt(II) derivatives are usually found to be puckered.[35] The distance from Pt to the formally sp^2 C(1) is significantly shorter than that to the sp^3 C(3), but the higher trans-influence of the sp^3 carbon is implied by the significantly longer distance from Pt to P(2) compared to that to P(1). Identical trends in M-C and M-P distances are found in the octahedral iridium(III) metallacyclobutene complex **5**.[27] Complexes **4** represent the first examples of the smallest unsaturated perfluorinated metallacycle, although a fully saturated perfluorometallacyclobutane complex of iron has been prepared and crystallographically characterized.[37]

The solution structures of **4a** and **4b** were shown by ^{19}F{^{31}P}, ^{19}F{^{19}F}, and ^{31}P{^{1}H} NMR spectroscopic methods[33] to be analogous to that observed for **4a** in the solid state. Observation of three resonances of relative intensity 2:1:1 in the ^{19}F NMR spectrum of **4a**, even at -78 °C, illustrates that the metallacyclobutene structure is maintained in solution and that the solid-state distortion from planarity at Pt, which should result in different chemical shifts for the fluorines in the CF$_2$ group, cannot be frozen out in solution. The different trans influence of the metallacyclobutene carbon atoms results in significantly different values for $J_{\text{Pt-P}}$ in both **4a** and **4b**.

Finally we note that formation of complexes 4 via C-C bond cleavage demonstrates the high reactivity of perfluorocyclopropene compared to both its fluorinated homologues and its hydrocarbon analogues. While perfluorocyclobutene forms a simple η^2-olefin complex **6** with Pt(0),[36] we have been unable to observe formation of the cyclopropene analogue at low temperatures, even though hydrocarbon cyclopropenes form stable η^2-olefin complexes with Pt(0).[10,11] We cannot distinguish whether the metallacyclobutene compounds **4** are formed via an intermediate η^2-olefin complex or via direct attack of the metal at a C-C bond.

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Supplementary Material Available: Tables of bond distances and angles, anisotropic thermal parameters, and hydrogen-atom coordinates (4 pages); a listing of observed vs calculated structure factors (24 pages). Ordering information is given on any current masthead page.

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Clusters Containing Carbene Ligands. The Unusual Synthesis, Coordination, and Reactivity of a Bridging (Dimetalliomethyl)(diethylamino)carbene Ligand in the Cluster Complex $Os_3(CO)_9[\mu_3\text{-}HCC(NEt_2)](\mu\text{-}H)_2$

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Summary: The photoinduced decarbonylation of $Os_3(CO)_{10}[\mu\text{-}C(H)C(H)NEt_2](\mu\text{-}H)$ (**1**) yields $Os_3(CO)_9[\mu_3\text{-}CC(H)NEt_2](\mu\text{-}H)_2$ (**2**; 58% yield) and $Os_3(CO)_9[\mu_3\text{-}C(H)CNEt_2](\mu\text{-}H)_2$ (**3**; 21% yield). Compound **3** was also obtained from **2** in 70% yield by heating to 68 °C in hexane solvent. Both products were characterized by IR, ^{1}H NMR, and X-ray diffraction analyses. Compound **2** contains an η^1-triply bridging (diethylamino)alkenylidene ligand coordinated to a triangular triosmium cluster. Compound **3** contains a triply bridging HC_2NEt_2 ligand that is formulated to include two osmiums as a (dimetalliomethyl)(diethylamino)carbene ligand on the basis of structural and reactivity evidence. Compound **3** reacts with $NHPr^n_2$ to yield $Os_3(CO)_9[\mu_3\text{-}C(H)CNPr^n_2](\mu\text{-}H)_2$ (**4**) in 74% yield by replacement of the diethylamino group.

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