

Supporting Information

General methods. Reactions were performed under a nitrogen atmosphere employing standard Schlenk and glovebox techniques unless specified otherwise. NMR spectra were obtained on a Varian spectrometer operating at 400 MHz for ^1H NMR, 101 MHz for ^{13}C NMR, and 161 MHz for ^{31}P NMR in CD_2Cl_2 at room temperature. Methylene chloride and diethylether were purified by passage through columns of activated alumina under nitrogen. CD_2Cl_2 was dried over CaH_2 prior to use. Chromium hexacarbonyl, bromocyclopropane, *tert*-butyllithium, $[\text{P}(t\text{-Bu})_2o\text{-biphenyl}]\text{AuCl}$, AgSbF_6 , hexanes, and pentane were purchased from major chemical suppliers and used as received.

$(\text{CO})_5\text{CrC}(\text{OMe})(\text{c-Pr})$ (2). Chromium complex **2** was synthesized using a modification^{S1} of a published procedure.^{S2} *tert*-Butyllithium (1.2 mL of a 1.7 M solution in hexane, 2.0 mmol) was added dropwise to a stirred solution of cyclopropylbromide (121 mg, 1.00 mmol) in Et_2O (2 mL) at $-78\text{ }^\circ\text{C}$, the resulting mixture was stirred for 30 min and transferred via canulae over 10 min to a suspension of chromium hexacarbonyl (220 mg, 1.00 mmol) in Et_2O (4 mL) at $0\text{ }^\circ\text{C}$. The resulting brown solution was warmed to room temperature and stirred for 2 h. The crude reaction mixture was concentrated under vacuum and the residue was dissolved in ice-cold deionized water. The reaction mixture was cooled at $0\text{ }^\circ\text{C}$ and treated with trimethyloxonium tetrafluoroborate (0.30 g, 2.0 mmol) and the resultant solution was warmed to room temperature over 15 min. The crude reaction mixture was extracted with pentane ($4 \times 30\text{ mL}$), and the combined organic fractions were dried (Na_2SO_4), filtered, and concentrated to give a yellow solid that was chromatographed (SiO_2 , pentane) to give **2** (158 mg, 57%) as a yellow solid.

$\{[\text{P}(t\text{-Bu})_2o\text{-biphenyl}]\text{AuC}(\text{OMe})(\text{c-Pr})\}^+ \text{SbF}_6^-$ (1). A suspension of $[\text{P}(t\text{-Bu})_2o\text{-biphenyl}]\text{AuCl}$ (33 mg, 6.2×10^{-2} mmol) and AgSbF_6 (21 mg, 6.2×10^{-2} mmol) in CH_2Cl_2 (1 mL) was added to a stirred solution of **2** (51 mg, 0.19 mmol) in CH_2Cl_2 (1 mL). The resulting solution was stirred 1 h and filtered through Celite, eluting with CH_2Cl_2 . The filtrate was concentrated and dried to give a solid yellow residue that was triturated with pentane to give

1 (51 mg, 66%) as a white solid. ^1H NMR: δ 7.91 (dt, $J = 2.4, 7.2$ 1 H), 7.54 - 7.56 (m, 2 H), 7.53 (t, $J = 7.6$ Hz, 2 H), 7.37-7.25 (m, 4 H), 4.53 (s, 3 H), 2.46 (tdd, $J = 4.0, 8.4, 11.6$ Hz, 1 H), 1.89 - 1.81 (m, 2 H), 1.80 - 1.76 (m, 2 H), 1.42 (d, $J = 15.6$ Hz, 18 H). $^{13}\text{C}\{^1\text{H}\}$ NMR: δ 303.9 (d, $J = 99.0$ Hz), 149.1 (d, $J = 14.6$ Hz), 143.9 (d, $J = 5.8$ Hz), 134.9, 133.5 (d, $J = 7.5$ Hz), 131.8, 130.1, 129.8, 128.2 (d, $J = 6.3$ Hz), 128.0, 125.5 (d, $J = 42.3$ Hz), 71.5, 38.3 (d, $J = 22.4$ Hz), 35.6 (d, $J = 5.6$ Hz), 31.1 (d, $J = 4.8$ Hz), 19.2. $^{31}\text{P}\{^1\text{H}\}$ NMR: δ 62.2. Anal. Calcd (found) for $\text{C}_{25}\text{H}_{35}\text{AuF}_6\text{PSb}$: H, 4.32 (4.41); C, 37.46 (37.57).

REFERENCES

S1) Semmelhack, M. F.; Lee, G. R. *Organometallics* **1987**, *6*, 1839.

S2) Turner, S. U.; Herndon, J. W.; McMullen, L. *J. Am. Chem. Soc.* **1992**, *114*, 8394.