

Divalent Dirhodium Imido Complexes: Formation, Structure, and Alkyne Cycloaddition Reactivity

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Supporting Information

All manipulations were performed under nitrogen using standard Schlenk techniques. Complex $[\text{Cp}^*\text{RhCl}_2]_2$ was prepared according to the literature.¹ Aniline was degassed and stored over activated 4A molecular sieves in the dark. Anhydrous solvents (toluene, hexanes, THF, and diethyl ether) were purchased from Kanto Chemical Co., Inc. and degassed before use. Deuterated solvents were obtained from Cambridge Isotope Laboratories, Inc., degassed, and stored over activated 4A molecular sieves. Other reagents were purchased from commercial vendors and used without further purification unless otherwise noted. ^1H and $^{31}\text{P}\{^1\text{H}\}$ NMR spectra were recorded on a JEOL ECP500 spectrometer at the field strength of 500.16 MHz and 202.48 MHz, respectively. $^{13}\text{C}\{^1\text{H}\}$ NMR spectra were obtained on a Varian VNMR400 spectrometer at the field strength of 100.55 MHz. Infrared spectra were obtained using a JASCO FT-IR 4100 spectrometer. FAB MS spectra were recorded on a JEOL JMS700 spectrometer. Elemental analyses were performed on a Perkin-Elmer 2400 Series II CHNS/O analyzer.

$[\text{Cp}^*\text{Rh}(\mu_2\text{-Cl})_2]$ (1). The original report by Sharp, Hoard, and Barnes involves sodium amalgam reduction of $[\text{Cp}^*\text{RhCl}_2]_2$ in dichloromethane.² We use zinc as a reducing agent and THF as a solvent. A suspension of $[\text{Cp}^*\text{RhCl}_2]_2$ (1.0 g, 1.6 mmol) and zinc dust (1.06 g, 16.2 mmol) in 15 mL of THF was stirred at room temperature until a deep blue gray suspension was obtained (typically this is achieved

within 3 h). The reaction mixture was filtered and the remaining solid was extracted with THF (10 mL \times 3). The combined filtrate was evaporated to dryness and the residue was extracted with toluene (15 + 10 mL). Removal of toluene in vacuo gave **1** as a dark blue crystalline solid. Yield 434 mg (50%). ^1H NMR (C_6D_6): δ 1.64 (s, 30H, Cp*).

[Cp*Rh(μ_2 -NHPH)]₂ (2). To a solution of aniline (121 μL , 1.32 mmol) in THF (15 mL) was added *n*-BuLi (2.73 M in *n*-hexane, 485 μL , 1.32 mmol) at -80°C and the solution was allowed to warm slowly to room temperature over 1 h. The pale yellow LiNHPH solution was then added to a solution of **1** (362 mg, 0.662 mmol) in THF (15 mL) at -80°C . The mixture was allowed to warm slowly to room temperature and stirred for 14 h. The solvent was removed in vacuo and the residue was extracted with hexanes (25 mL). Removal of hexanes afforded **2** as a dark violet crystalline solid (324 g, 74%), which was pure as judged by ^1H NMR spectroscopy. Analytically pure sample of **2** was obtained after recrystallization from hexanes at -30°C . Anal. Calcd for $\text{C}_{32}\text{H}_{42}\text{N}_2\text{Rh}_2$: C, 58.19; H, 6.41; N, 4.24. Found: C, 58.28; H, 6.42; N, 3.97. ^1H NMR (C_6D_6 , 60°C): δ 7.12 (t, 4H, Ph), 6.89 (br d, 4H, Ph), 6.82 (t, 2H, Ph), 1.55 (s, 30H, Cp*), 1.47 (br s, 2H, NH). At room temperature, the doublet phenyl signal is almost invisible due to significant line broadening. $^{13}\text{C}\{^1\text{H}\}$ NMR (C_6D_6 , 60°C): δ 158.5, 127.9, 120.0 (Ph), 88.9 (dd, $J_{\text{RhC}} = 2.2$ and 4.4 Hz, C_5Me_5), 10.0 (C_5Me_5). IR (nujol): 3583 (w), 3387 (w), 2723 (w), 1594(m), 1375(s), 1238(m), 998(w), 759(m) cm^{-1} .

[Cp*Rh(μ_2 -NHPH)(μ_2 -Cl)RhCp*] (3). To a solution of **1** (224 mg, 0.409 mmol) in THF (15 mL) was added aniline (112 μL , 1.23 mmol), and the mixture was stirred at 60°C for 2 days. Volatiles were removed under reduced pressure, and the residue was extracted with hexanes (15 mL \times 3). Removal of hexanes afforded **3** as a dark red crystalline solid (176 mg, 71%), which was pure as judged by ^1H NMR spectroscopy. Analytically pure sample of **3** was obtained after recrystallization from hexanes at -30°C . Anal. Calcd for $\text{C}_{26}\text{H}_{36}\text{NClRh}_2$: C, 51.72; H, 6.01; N, 2.32. Found: C, 51.57; H, 6.26; N, 2.28. ^1H NMR (C_6D_6): δ 7.05 (t, 2H, Ph), 6.80 (t, 1H, Ph), 6.56 (d, 2H, Ph), 3.53 (br, 1H, NH), 1.64 (s, 30H, Cp*).

$^{13}\text{C}\{^1\text{H}\}$ NMR (C_6D_6): δ 157.9, 127.9, 122.4, 120.1 (Ph), 88.9 (dd, $J_{\text{RhC}} = 2.2$ and 7.4 Hz, C_5Me_5), 10.4 (C_5Me_5). IR (nujol): 3376(m), 1595(s), 1244(m), 1121(w), 941(w), 764(w) cm^{-1} .

[Cp* $\text{Rh}(\mu_2\text{-NHPH})(\mu_2\text{-OMe})\text{RhCp}^*$] (4). To a stirred solution of **3** (126 mg, 0.209 mmol) in THF (15 mL) was added NaOMe (113 mg, 2.09 mmol) at -80°C . The mixture was allowed to warm slowly to room temperature and stirred overnight. Volatiles were removed under reduced pressure, and the remaining solid was extracted with hexanes (20 mL). Removal of hexanes afforded **3** as a dark red crystalline solid (92 mg, 72%), which was pure as judged by ^1H NMR spectroscopy. Analytically pure sample of **4** was obtained after recrystallization from hexanes at -30°C . Anal. Calcd for $\text{C}_{27}\text{H}_{39}\text{NORh}_2$: C, 54.10; H, 6.56; N, 2.34. Found: C, 53.70; H, 6.62; N, 2.05. ^1H NMR (C_6D_6): δ 7.08 (t, 2H, Ph), 6.81 (t, 1H, Ph), 6.68 (d, 2H, Ph), 3.59 (s, 3H, OMe), 1.99 (br s, 1H, NH), 1.67 (s, 30H, Cp*). $^{13}\text{C}\{^1\text{H}\}$ NMR (C_6D_6): δ 157.5, 128.0, 122.6, 119.6 (Ph), 87.2 (dd, $J_{\text{RhC}} = 2.0$ and 5.5 Hz, C_5Me_5), 67.7 (OMe), 10.4 (C_5Me_5).

[Cp* $\text{Rh}(\text{PMe}_3)(\mu_2\text{-NPh})\text{RhCp}^*$] (5). To a solution of **3** (113 mg, 0.195 mmol) in THF (15 mL) were added successively PMe_3 (1.0 M in THF, 195 μL , 0.195 mmol) and $\text{NaN}(\text{SiMe}_3)_2$ (1.0 M in THF, 234 μL , 0.234 mmol) at -80°C . The mixture was allowed to warm slowly to room temperature and stirred for 14 h. The dark green solution was evaporated to dryness and the residue was extracted with hexanes (25 mL). Removal of hexanes afforded **5** as a dark green crystalline solid (100 mg, 80%), which was pure as judged by ^1H NMR spectroscopy. Analytically pure sample of **5** was obtained after recrystallization from hexanes at -30°C . Anal. Calcd for $\text{C}_{29}\text{H}_{44}\text{NPRh}_2$: C, 54.13; H, 6.89; N, 2.18. Found: C, 53.75; H, 6.66; N, 1.91. ^1H NMR (C_6D_6): δ 7.90 (d, 2H, Ph), 7.25 (t, 2H, Ph), 7.03 (t, 1H, Ph), 2.00 (s, 15H, Cp*), 1.85 (d, $^4J_{\text{PH}} = 2.3$ Hz, 15H, Cp*), 0.97 (d, $^2J_{\text{PH}} = 8.3$ Hz, 9H, PMe_3). $^{13}\text{C}\{^1\text{H}\}$ NMR (C_6D_6): δ 165.6, 129.2, 127.9, 123.9, 119.2 (Ph), 94.6 (t, $J = 4.4$ Hz, C_5Me_5), 88.3 (d, $^1J_{\text{RhC}} = 6.7$ Hz, C_5Me_5), 19.6 (dd, $^1J_{\text{PC}} = 26.4$ Hz, $^2J_{\text{RhC}} = 1.5$ Hz, PMe_3), 11.7 (C_5Me_5), 11.6 (C_5Me_5). $^{31}\text{P}\{^1\text{H}\}$ NMR (C_6D_6): δ -1.25 (d, $^1J_{\text{RHP}} = 204$ Hz). IR (nujol): 2723(w), 1574(w), 1145(m), 1021(w), 988(w), 943(m), 902(w) cm^{-1} .

[Cp*Rh(μ_2 - η^2 : η^3 -PhCCPhNPh)] (6a). Complex **3** (83 mg, 0.14 mmol) and diphenylacetylene (25 mg, 0.14 mmol) were dissolved in THF (10 mL) and the solution was cooled to $-80\text{ }^{\circ}\text{C}$. To this solution was added 1.0 M THF solution of NaN(SiMe₃)₂ (165 μL , 0.165 mmol) and the mixture was allowed to warm slowly to room temperature with stirring over 14 h. The dark red solution was evaporated to dryness and the residue was extracted with hexanes (15 mL). Removal of hexanes afforded **6a** as a dark red crystalline solid (80 mg, 78%), which was pure as judged by ¹H NMR spectroscopy. Analytically pure crystals of **6a** was obtained by recrystallization from diethyl ether at $-30\text{ }^{\circ}\text{C}$. Anal. Calcd for C₄₀H₄₅NRh₂: C, 64.43; H, 6.08; N, 1.88. Found: C, 64.25; H, 6.31; N, 1.65. ¹H NMR (C₆D₆, 20 $^{\circ}\text{C}$): δ 6.82–7.27 (m, 15H, Ph), 1.73 (s, 30H, Cp*). ¹H NMR (THF-*d*₈, $-90\text{ }^{\circ}\text{C}$): δ 7.12–7.07 (m, 7H, Ph), 6.90 (m, 4H, Ph), 6.73 (m, 2H, Ph), 6.56 (m, 2H, Ph), 1.68, 1.63 (s, 15H each, Cp*). ¹³C{¹H} NMR (C₆D₆, 20 $^{\circ}\text{C}$): δ 150.6, 145.1, 136.5, 128.9, 128.8, 127.6, 126.1, 124.1, 123.4, 122.3 (Ph), 121.6 (br t, ¹J_{RhC} = 26.5 Hz, PhCCPhNPh), 108.2 (PhCCPhNPh), 91.9 (C₅Me₅), 10.5 (C₅Me₅). IR (nujol): 1665(w), 1589(s), 1498(m), 1312(m), 1299(m), 1150(m), 1067(s), 950(m), 757(s), 698(s) cm⁻¹.

[Cp*Rh(μ_2 - η^2 : η^3 -HCC(*t*-Bu)NPh)] (6b). To a solution of **3** (95 mg, 0.16 mmol) in THF (6 mL) were added successively *tert*-butylacetylene (18 μL , 0.16 mmol) and NaN(SiMe₃)₂ (1.0 M in THF, 190 μL , 0.190 mmol) at $-80\text{ }^{\circ}\text{C}$. The solution was allowed to warm slowly to room temperature and stirred overnight. The dark red solution was evaporated to dryness and the residue was extracted with hexanes (15 mL). Removal of hexanes afforded **6b** as a dark red crystalline solid (87 mg, 85%), which was pure as judged by ¹H NMR spectroscopy. Analytically pure crystals of **6b** was obtained by recrystallization from hexanes at $-80\text{ }^{\circ}\text{C}$. Anal. Calcd for C₃₂H₄₅NRh₂: C, 59.17; H, 6.98; N, 2.16. Found: C, 58.71; H, 7.21; N, 1.99. ¹H NMR (C₆D₆, 20 $^{\circ}\text{C}$): δ 7.12 (t, 2H, Ph), 7.06 (d, 2H, Ph), 6.89 (t, 1H, Ph), 5.11 (s, 1H, HCC(*t*-Bu)NPh), 1.94 (s, 30H, Cp*), 1.14 (s, 9H, CMe₃). ¹H NMR (THF-*d*₈, $-70\text{ }^{\circ}\text{C}$): δ 7.12 (m, 2H, Ph), 6.86 (m, 3H, Ph), 4.79 (s, 1H, HCC(*t*-Bu)NPh), 2.05, 1.60 (s, 15H each, Cp*). ¹³C{¹H} NMR (C₆D₆, 20 $^{\circ}\text{C}$): δ 152.2, 124.9, 122.5, 128.2 (Ph), 129.4 (HCC(*t*-Bu)NPh), 101.1 (t, ¹J_{RhC} = 26.5 Hz,

HCC(*t*-Bu)NPh), 91.5 (C_5Me_5), 37.5 (CMe_3), 29.2 (CMe_3), 11.5 (C_5Me_5). IR (nujol): 2724(w), 1589(s), 1385(s), 1307(m), 1150(w), 1066(w), 925(m), 764(w), 722(w), 695(w) cm^{-1} .

[Cp*Rh(μ_2 - η^2 : η^3 -HCC(C_6H_4 -*p*-Me)NPh)] (6c). To a solution of **3** (187 mg, 0.310 mmol) in THF (15 mL) were added successively *p*-tolylacetylene (39 μ L, 0.310 mmol) and NaN(SiMe₃)₂ (1.0 M in THF, 372 μ L, 0.372 mmol) at $-80^\circ C$. The solution was allowed to warm slowly to room temperature and stirred overnight. The dark red solution was evaporated to dryness and the residue was extracted with hexanes (15 mL). Removal of hexanes afforded **6c** as a dark red sticky solid (192 mg, 91%), which was pure as judged by ¹H NMR spectroscopy. We were unable to obtain satisfactory elemental analysis on this compound. ¹H NMR (C_6D_6 , $20^\circ C$): δ 7.12 (t, 2H, Ar), 7.02 (d, 2H, Ar), 6.93 (d, 2H, Ar), 6.89 (t, 1H, Ar), 6.82 (d, 2H, Ar), 5.63 (s, 1H, HCC(*p*-tol)NPh), 2.02 (s, 3H, C_6H_4Me), 1.83 (s, 30H, Cp*). ¹H NMR (THF-*d*₈, $-80^\circ C$): δ 7.12 (m, 2H, Ar), 7.02 (m, 2H, Ar), 6.93 (m, 2H, Ar), 6.88 (m, 1H, Ar), 6.71 (m, 2H, Ar), 5.25 (s, 1H, HCC(*p*-tol)NPh), 2.21 (s, 3H, C_6H_4Me), 1.72, 1.65 (s, 15H each, Cp*). ¹³C{¹H} NMR (C_6D_6 , $20^\circ C$): δ 151.7, 135.2, 134.6, 128.6, 125.9, 123.1, 121.8, 119.1 (Ar), 110.1 (HCC(*p*-tol)NPh), 105.1 (t, ¹*J*_{RhC} = 26.5 Hz, HCC(*p*-tol)NPh), 91.4 (C_5Me_5), 21.2 (C_6H_4Me), 10.9 (C_5Me_5). IR (nujol): 1662(m), 1592(s), 1069(s), 941(m), 723(w) cm^{-1} .

[Cp*Rh(μ_2 -NAr)RhCp*] (7; Ar = 2,6-diisopropylphenyl). To a solution of 2,6-diisopropylaniline (190 μ L, 1.00 mmol) in THF (10 mL) was added *n*-BuLi (2.77 M in *n*-hexane, 400 μ L, 1.11 mmol) at $-80^\circ C$ and the solution was allowed to warm slowly to room temperature over 1 h. The pale yellow solution was then added to a stirred solution of **1** (274 mg, 0.500 mmol) in THF (10 mL) at $-80^\circ C$. The mixture was allowed to warm slowly to room temperature and stirred overnight. The solvent was removed in vacuo and the residue was extracted with hexanes (15 mL). The extract was concentrated to 1.5 mL and stored overnight in a freezer to give **7** as dark brown crystals. Yield 208 mg, 64%. Anal. Calcd for $C_{32}H_{47}NRh_2$: C, 58.99; H, 7.27; N, 2.15. Found: C, 58.81; H, 7.64; N, 2.11. ¹H NMR (C_6D_6): δ 7.31 (m, 2H, Ar), 7.26 (m, 1H, Ar), 3.59 (sept, ³*J*_{HH} = 6.9 Hz, 2H, *CHMe*₂), 1.72 (s, 30H, Cp*), 1.28 (d,

$^3J_{\text{HH}} = 6.9$ Hz, 12H, CHMe_2). $^{13}\text{C}\{^1\text{H}\}$ NMR (C_6D_6): δ 166.3, 136.8, 122.1, 121.9 (Ar), 91.3 (dd, $J_{\text{RhC}} = 1.8$ and 4.3 Hz, C_5Me_5), 27.1 (CHMe_2), 23.4 (CHMe_2), 11.4 (C_5Me_5).

[Cp* $\text{Rh}(t\text{-BuNC})(\mu_2\text{-NAr})\text{RhCp}^*$] (8**; Ar = 2,6-diisopropylphenyl).** To a solution of **7** (143 mg, 0.219 mmol) in hexanes (8 mL) was added $t\text{-BuNC}$ (25 μL , 0.220 mmol) at room temperature. The solution was concentrated to ca. 4 mL and stored overnight in a freezer (-30 $^\circ\text{C}$) to give **8** as dark reddish brown crystals. Yield 102 mg, 63%. Anal. Calcd for $\text{C}_{37}\text{H}_{56}\text{N}_2\text{Rh}_2$: C, 60.49; H, 7.68; N, 3.81. Found: C, 60.58; H, 7.67; N, 3.79. ^1H NMR (C_6D_6 , 20 $^\circ\text{C}$): δ 7.19 (pseudo t, 1H, Ar), 7.15 (dd, 1H, Ar), 7.10 (dd, 1H, Ar), 4.33, 3.78 (sept, $^3J_{\text{HH}} = 6.9$ Hz, 1H each, CHMe_2), 1.87 (s, 30H, Cp*), 1.51 (d, $^3J_{\text{HH}} = 6.9$ Hz, 6H, CHMe_2), 1.25 (br, 6H, CHMe_2), 1.20 (s, 9H, $t\text{-Bu}$). ^1H NMR ($\text{THF-}d_8$, -80 $^\circ\text{C}$): δ 6.83 (m, 2H, Ar), 6.75 (m, 1H, Ar), 4.15, 3.66 (m, 1H, CHMe_2), 1.91, 1.68 (s, 15H each, Cp*), 1.47 (s, 9H, $t\text{-Bu}$), 1.31, 1.22, 1.16, 0.81 (m, 3H each, CHMe_2). $^{13}\text{C}\{^1\text{H}\}$ NMR (C_6D_6 , 20 $^\circ\text{C}$): δ 161.1 (dd, $^1J_{\text{RhC}} \approx 40$ Hz, $^2J_{\text{RhC}} \approx 4$ Hz, $t\text{-BuNC}$), 161.6 (t, $^2J_{\text{RhC}} = 6.0$ Hz, Ar), 140.7, 137.8, 122.6, 122.5, 121.0 (Ar), 92.5 (br, C_5Me_5), 55.5 (CMe_3), 31.8 (CMe_3), 27.2, 26.5 (CHMe_2), 25.8, 23.4 (br, CHMe_2), 11.2 (C_5Me_5). IR (nujol): 2028 (vs), 1581 (m), 1409, (w), 1321 (w), 1259 (w), 1233 (m), 1205 (m), 1096 (w), 1023 (m), 907 (w), 796 (w), 759 (m), 723 (w) cm^{-1} .

General Procedure for X-ray Crystallographic Study. A crystal of each sample was mounted on a glass capillary with Paratone-N hydrocarbon oil and transferred to a Rigaku RAXIS Rapid diffractometer equipped with an imaging plate detector. The frame data were processed using the Rigaku PROCESS-AUTO program,³ and the reflection data were corrected for absorption with an ABSCOR program.⁴ The structures were solved by a direct method (SHELXS97) and refined on F^2 by full-matrix least-squares method by using SHELXL97.⁵ Unless otherwise noted, anisotropic refinement was applied to all non-hydrogen atoms and hydrogen atoms were computationally generated as riding models. Thermal ellipsoid plots were drawn with the ORTEP-3 program⁶ and presented at the 35% probability level.

[Cp*Rh(μ_2 -NHPH)]₂ (2). Crystals were grown from cold hexanes (−30 °C). Data were collected and processed as described above except that NH hydrogen atom was located in the difference electron density map and refined isotropically. Crystallographic data follow: C₃₂H₄₂N₂Rh₂, *M* = 660.50, orthorhombic, space group *Pnnm*, *a* = 12.601(5) Å, *b* = 14.722(6) Å, *c* = 16.085(6) Å, *V* = 2983.9(19) Å³, *Z* = 4, *T* = 296 K, $\mu(\text{Mo } K\alpha) = 1.128 \text{ mm}^{-1}$, 26254 reflections measured, 3543 unique (*R*_{int} = 0.0497), *R*₁ = 0.0485, *wR*₂ = 0.1038, *GOF* = 1.215.

[Cp*Rh(μ_2 - η^2 : η^3 -PhCCPhNPh)RhCp*] (6a). Crystals were grown from cold diethyl ether (−30 °C). Data were collected and processed as described in the general procedure section. There are two problems in the structure solution. Since the terminal N(1)Ph and C(2)Ph groups in the bridging ligand PhCCPhNPh were indistinguishable, they were treated as a disorder; N(1) and C(2) atoms were duplicated in each terminal with equal *U*_{ij} values and 50% occupancy. Additionally, the solved structure contains relatively high residual electron density peaks (4.848 e Å^{−3}) near rhodium atoms probably because of low quality of the crystals obtained. No special treatment was done for this problem. Crystallographic data follow: C₄₀H₄₅NRh₂, *M* = 745.59, monoclinic, space group *P2₁/a*, *a* = 12.763(4) Å, *b* = 16.265(7) Å, *c* = 16.571(6) Å, $\beta = 92.249(12)^\circ$, *V* = 3437(2) Å³, *Z* = 4, *T* = 296 K, $\mu(\text{Mo } K\alpha) = 0.988 \text{ mm}^{-1}$, 32560 reflections measured, 7864 unique (*R*_{int} = 0.1297), *R*₁ = 0.1018, *wR*₂ = 0.3140, *GOF* = 1.060.

[Cp*Rh(μ_2 -NAr)RhCp*] (7). Crystals were grown from cold hexanes (−30 °C). Data were collected and processed as described in the general procedure section. Crystallographic data follow: C₃₂H₄₇NRh₂, *M* = 651.53, monoclinic, space group *C2/c*, *a* = 16.831(7) Å, *b* = 16.775(5) Å, *c* = 23.043(7) Å, $\beta = 103.634(15)^\circ$, *V* = 6323(4) Å³, *Z* = 8, *T* = 296 K, $\mu(\text{Mo } K\alpha) = 1.062 \text{ mm}^{-1}$, 30583 reflections measured, 7244 unique (*R*_{int} = 0.0238), *R*₁ = 0.0305, *wR*₂ = 0.0845, *GOF* = 1.109.

[Cp*Rh(*t*-BuNC)(μ_2 -NAr)RhCp*] (8). Crystals were grown from cold hexanes (−30 °C). Data were collected and processed as described in the general procedure section. Crystallographic data follow: C₃₇H₅₆N₂Rh₂, *M* = 734.66, orthorhombic, space group *Pna2₁*, *a* = 18.111(4) Å, *b* = 11.438(7) Å,

$c = 17.523(4) \text{ \AA}$, $V = 3629.7(16) \text{ \AA}^3$, $Z = 4$, $T = 296 \text{ K}$, $\mu(\text{Mo } K\alpha) = 0.934 \text{ mm}^{-1}$, 33678 reflections measured, 8249 unique ($R_{\text{int}} = 0.0339$), $R1 = 0.0324$, $wR2 = 0.0597$, $\text{GOF} = 1.021$. The Flack parameter (0.45(2)) suggests that the crystal is a merohedral twin.

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