

(I) Synthesis of New Compounds

(1) Synthesis of Tungsten- η^1 -alkynol. Synthesis of Compound 2. To enyne **1** (0.50 g, 5.20 mmol) in diethyl amine (15 mL) was added $\text{CpW}(\text{CO})_3\text{Cl}$ (2.0 g, 5.43 mmol) and CuI (103 mg, 0.54 mmol) at 0 °C, and the solution was stirred for 2 h before the it was concentrated. The resulting dark red residue were eluted through a silica column (diethyl ether/hexane=1/1) to give a dark red band that yielded tungsten-alkynol **2** as a dark orange solid (1.81 g, 4.20 mmol, 78%).

(2) Synthesis of Tetranuclear Cyclopropane Derivatives. Synthesis of Compound 3. To compound **2** (100 mg, 0.24 mmol) in THF (10 mL) was added $\text{Co}_2(\text{CO})_8$ (87 mg, 0.25 mmol) at 23 °C, and the mixture was stirred for 8 h. The solution was concentrated, eluted through a silica column to yield a dark black band that yielded **3** as a dark black solid (135 mg, 0.20 mmol, 85 %).

(3) Insertion of XH bond into Carbenoid: Synthesis of Compounds 5 and 6. To a THF solution (15 mL) of compound **4** (485 mg, 1.13 mmol) was added $\text{Co}_2(\text{CO})_6$ (426 mg, 1.25 mmol) at 23 °C, and the mixture was stirred for 8 h before it was concentrated. Elution of the dark red residue through a silica column (diethyl ether/hexane=1/1) produced two black bands which yield compound **5** ($R_f=0.70$, 280 mg, 0.41 mmol, 36%) and **6** ($R_f = 0.75$, 217 mg, 0.32 mmol, 28%) respectively.

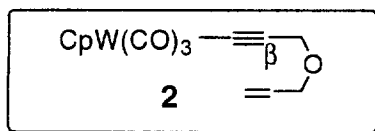
(4) Cyclocarbonylation Reaction. Synthesis of Compound 10. Synthesis of this compound was conducted similarly from tungsten alkynyl compound **9** (450 mg, 0.94 mmol) and $\text{Co}_2(\text{CO})_8$ (354 mg, 1.04 mmol); the yield of compound **10** (554 mg, 0.72 mmol) was 77%.

(5) Synthesis of Tungsten- η^1 -cyclopentenone. Synthesis of Compound

25. To a benzene solution (15 mL) of compound **18** (500 mg, 0.90 mmol) was added $\text{Co}_2(\text{CO})_8$ (338 mg, 0.99 mmol) at 25°C and the mixture was stirred for 2 h. The solution was heated at 50 °C for 7 h, and then cooled to 23°C before it was concentrated. The residues were chromatographed on a silica column (diethyl ether/hexane=1/1) to yield compound **25** as a yellow solid (320 mg, 0.55 mmol 61%).

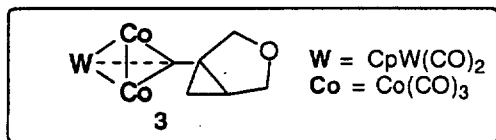
(6) Conversion of Cyclopropane to Tungsten- η^1 -cyclopentenone 29.

Synthesis of Compound 29. To a benzene solution (15 mL) of cyclopropane **3** (450 mg, 0.66 mmol) was added $\text{P}(\text{OPh})_3$ (244 mg, 0.79 mmol) and $\text{Co}_2(\text{CO})_8$ (244 mg, 0.72 mmol), and the mixtures were heated in benzene for 3 h. The residues were chromatographed (diethyl ether/hexane=1/1) through a silica column to yield a yellow band that afforded **29** (203 mg, 0.45 mmol, 68%) as yellow solid.

(II) Spectral Data for New Compounds**Compound 2**

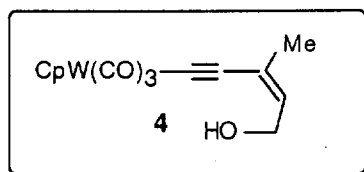
IR(neat, cm^{-1}): $\nu(\text{CO})$ 2046(s), 1956(s), $\nu(\text{C} \equiv \text{C})$ 2125(w), $\nu(\text{C}=\text{C})$ 1654(w). ^1H NMR (300MHz, CDCl_3): δ 5.91 (1H, m), 5.58 (5H, s), 5.28 (1H, dd, $J = 18.0, 2.0$ Hz), 5.14 (1H, dd, $J = 10.4, 2.0$ Hz), 4.23 (2H, s), 4.02 (2H, d, $J = 4.9$ Hz). ^{13}C NMR (100 MHz, CDCl_3): δ 229.2, 211.7, 134.7, 124.4, 117.1, 91.4, 69.9, 67.7, 50.6. MS (EI, 75 eV, m/z) 428 (M^+). Anal. Calcd. for $\text{C}_{14}\text{H}_{12}\text{WO}_4$: C, 39.25; H, 2.83. Found: C, 39.13; H, 2.99.

Compound 3



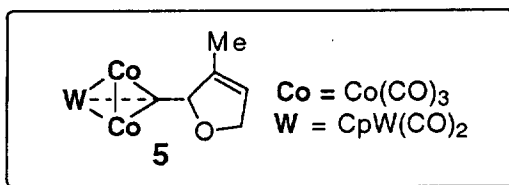
IR(neat, cm⁻¹): ν (CO) 2061(s), 2024(s), 2006(s), 1998(s), 1938(s), 1872 (m). ¹H NMR (300MHz, dg-toluene): δ 4.65 (5H, s, Cp), 4.57 (1H, dd, J = 7.8, 2.0 Hz), 4.27 (1H, dd, J = 7.8, 1.6 Hz), 4.14 (1H, ABq, J = 8.1 Hz), 4.00 (1H, ABq, J = 8.1 Hz), 1.96 (1H, m), 1.83 (1H, dd, J = 4.8, 3.6 Hz), 1.83 (1H, dd, J = 8.6, 4.8 Hz). ¹³C NMR (100 MHz, CDCl₃): δ 271.0, 203.5, 88.9, 77.1, 69.7, 56.2, 36.6, 25.5. MS (EI, 75 eV, m/z): 686 (M⁺). Anal. Calcd. for C₁₉H₁₂WCo₂O₉: C, 33.24; H, 1.76. Found: C, 33.30; H, 1.88.

Compound 4



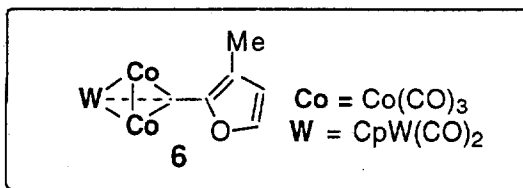
IR(neat, cm⁻¹): ν (OH) 3350 (bs vs), ν (CO) 2046(s), 1956(s), ν (C $\equiv$ C) 2125(w), ν (C=C) 1644(w). ¹H NMR (400 MHz, CDCl₃): δ 5.57 (5H, s), 5.50 (1H, t, J = 5.6 Hz), 4.23 (2H, d, J = 5.6 Hz), 1.75 (3H, s). ¹³C NMR (100 MHz, CDCl₃): δ 228.3, 211.4, 131.0, 127.6, 123.7, 91.5, 78.9, 61.3, 23.9. MS (EI, 75 eV, m/z): 428 (M⁺). Anal. Calcd. for C₁₄H₁₂WO₄: C, 39.28; H, 2.83. Found: C, 39.33; H, 2.90.

Compound 5



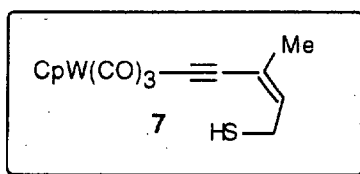
IR(neat, cm⁻¹): 2061(s), 2024(s), 2006(s), 1998(s), 1938(s), 1872 (m), ν (C=C) 1644(w). ¹H NMR (400 MHz, d₆-benzene): δ 6.53 (1H, br s), 5.43 (1H, s), 4.80 (5H, s), 4.65 (1H, dd, J = 12.2, 2.0 Hz), 4.48 (1H, dd, J = 12.2, 1.8 Hz), 1.75 (3H, s). ¹³C NMR (100 MHz, CDCl₃): δ 203.8, 199.4, 138.5, 123.7, 104.9, 88.4, 73.7, 14.7. MS (EI, 75 eV, m/z): 686 (M⁺). Anal. Calcd. for C₁₉H₁₂WCo₂O₉: C, 33.27; H, 1.76. Found: C, 32.98; H, 1.73.

Compound 6



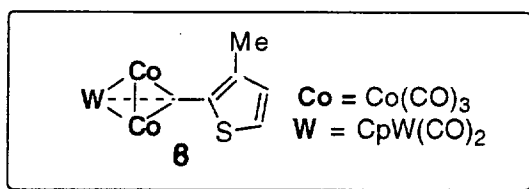
IR(neat, cm⁻¹): 2061(s), 2024(s), 2006(s), 1998(s), 1938(s), 1872 (m), $\nu(\text{C}=\text{C})$ 1644(w). ¹H NMR (400 MHz, d₆-benzene): δ 6.82 (1H, br s), 5.93 (1H, br s), 4.40 (5H, s), 2.12 (3H, s). ¹³C NMR (100 MHz, CDCl₃): δ 237.8, 203.4, 167.9, 139.3, 114.3, 112.8, 90.7, 12.6. MS (EI, 75 eV, m/z): 684 (M⁺). Anal. Calcd. for C₁₉H₁₀WCo₂O₉: C, 33.36; H, 1.47. Found: C, 33.30; H, 1.70.

Compound 7



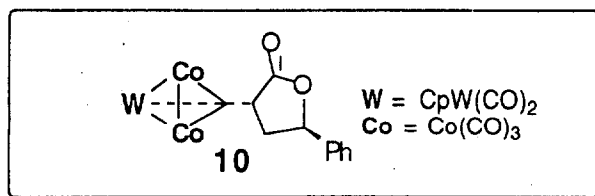
IR(neat, cm⁻¹): $\nu(\text{CO})$ 2046(s), 1956(s), $\nu(\text{C} \equiv \text{C})$ 2125(w), $\nu(\text{C}=\text{C})$ 1644(w). ¹H NMR (300 MHz, CDCl₃): δ 5.58 (5H, s), 5.39(1H, t, $J = 7.0$ Hz), 3.98 (2H, d, $J = 7.0$ Hz), 2.76 (3H, s). ¹³C NMR (75 MHz, CDCl₃): δ 228.9, 211.5, 128.9, 128.2, 123.3, 91.5, 31.3, 23.9. MS (EI, 75 eV, m/z): 443 (M⁺). Anal. Calcd. for C₁₄H₁₂WCO₃: C, 37.86; H, 2.72. Found: C, 37.73; H, 2.70.

Compound 8



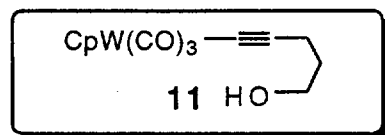
IR(neat, cm⁻¹): 2061(s), 2023(s), 2008(s), 1996(s), 1936(s), 1870(m), $\nu(\text{C} \equiv \text{C})$ 2125(w). ¹H NMR (400 MHz, d₆-benzene): δ 6.46 (1H, s), 6.42 (1H, s), 4.47 (5H, s), 2.20 (3H, s). ¹³C NMR (100 MHz, CDCl₃): δ 241.0, 202.1, 199.5, 162.5, 131.3, 129.1, 121.3, 91.0, 29.7. MS (EI, 75 eV, m/z): 699 (M⁺). Anal. Calcd. for C₁₉H₁₀-WCO₂SO₃: C, 32.60; H, 1.44. Found: C, 32.33; H, 1.49.

Compound 10



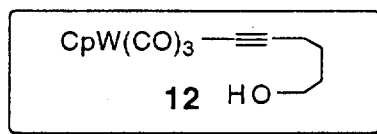
IR(neat, cm^{-1}): 2060(s), 2023(s), 2008(s), 1996(s), 1936(s), 1870(m), $\nu(\text{C} \equiv \text{C})$ 2125(w). ^1H NMR (300 MHz, dg-toluene): δ 7.45–7.22 (5H, m), 5.13 (1H, br t, $J = 6.8$ Hz), 4.87 (5H, s), 3.05 (1H, m), 2.55 (1H, dd, $J = 8.2, 6.8$ Hz). ^{13}C NMR (100 MHz, dg-toluene): δ 244.0, 204.1, 174.0, 162.5, 141.3, 90.5, 78.3, 62.1, 49.1. MS (EI, 75 eV, m/z) 764 (M^+). Anal. Calcd. for $\text{C}_{24}\text{H}_{14}\text{WCo}_2\text{O}_{10}$: C, 37.73; H, 1.85. Found: C, 37.66; H, 2.01.

Compound 11



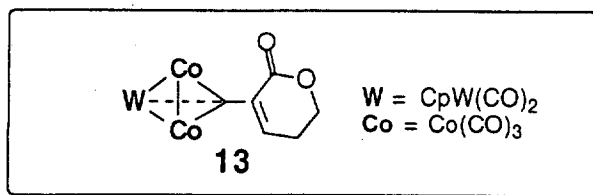
IR(neat, cm^{-1}): $\nu(\text{CO})$ 2040(s), 1955(s), $\nu(\text{C} \equiv \text{C})$ 2125(w). ^1H NMR (300 MHz, CDCl_3): δ 5.54 (5H, s), 3.72 (2H, br t, $J = 6.3$ Hz), 2.48 (2H, br t, $J = 6.3$ Hz), 1.68 (2H, m). ^{13}C NMR (100 MHz, CDCl_3): δ 229.6, 211.8, 128.9, 91.4, 63.2, 57.8, 32.1, 19.5. MS (EI, 75 eV, m/z) 402 (M^+). Anal. Calcd. for $\text{C}_{12}\text{H}_{10}\text{WO}_4$: C, 35.85; H, 2.51. Found: C, 35.80; H, 2.44.

Compound 12



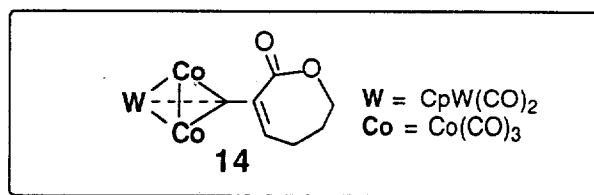
IR(neat, cm^{-1}): $\nu(\text{CO})$ 2041(s), 1959(s), $\nu(\text{C} \equiv \text{C})$ 2150(w). ^1H NMR (300 MHz, CDCl_3): δ 5.54 (5H, s), 3.62 (2H, t, $J = 6.1$ Hz), 2.38 (2H, t, $J = 6.3$ Hz), 1.62 (2H, m), 1.51 (2H, m). ^{13}C NMR (100 MHz, CDCl_3): δ 230.0, 211.9, 129.2, 62.9, 55.9, 31.9, 26.5, 22.0. MS (EI, 75 eV, m/z) 416 (M^+). Anal. Calcd. for $\text{C}_{13}\text{H}_{12}\text{WO}_4$: C, 35.53; H, 2.91. Found: C, 35.88; H, 2.80.

Compound 13



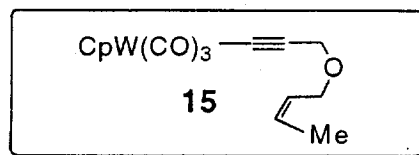
IR(neat, cm⁻¹): ν (CO) 2066(s), 2022(s), 2006(s), 1998(s), 1938(s), 1872 (m), 1730(s), ¹H NMR (300 MHz, CDCl₃): δ 6.92 (1H, br s), 5.43 (5H, s), 4.35 (2H, br s), 2.67 (2H, br s). ¹³C NMR (75.0 MHz, CDCl₃): δ 250.6, 203.6, 162.6, 151.4, 135.6, 90.0, 63.3, 24.9. MS (EI, 75 eV, m/z) 700 (M⁺). Anal. Calcd. for C₁₉H₁₀WCo₂O₁₀: C, 32.58; H, 1.44. Found: C, 32.33; H, 1.55.

Compound 14



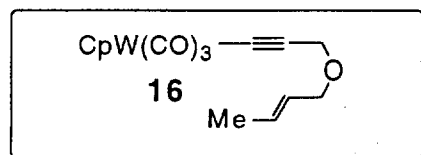
IR(neat, cm⁻¹): ν (CO) 2076 (s), 2026(s), 1989(s), 1931(s), 1874 (m), 1717(s), ¹H NMR (300 MHz, CDCl₃): δ 6.27 (1H, br s), 4.88 (5H, s), 4.25 (2H, br s), 2.13 (2H, br s), 1.54 (2H, br s). ¹³C NMR (75.0 MHz, CDCl₃): δ 252.6, 202.2, 170.6, 157.5, 127.4, 91.0, 65.5, 26.7, 22.22. MS (EI, 75 eV, m/z) 714 (M⁺). Anal. Calcd. for C₂₀H₁₂WCo₂O₁₀: C, 33.60; H, 1.70. Found: C, 33.58; H, 1.80.

Compound 15



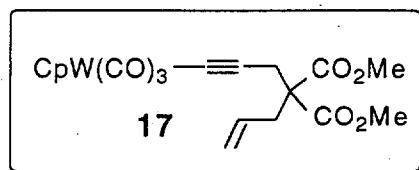
IR(neat, cm⁻¹): ν (CO), 2046(s), 19540(s), ν (C $\equiv$ C) 2129(w), ν (C=C) 1647 (s), ¹H NMR (300 MHz, CDCl₃): δ 5.56-5.70 (2H, m), 5.56 (5H, s), 4.17 (2H, s), 4.08 (2H, d, J = 7.2 Hz), 1.63 (3H, d, J = 6.2 Hz). ¹³C NMR (75.0 MHz, CDCl₃): δ 228.9, 211.7, 129.6, 127.4, 124.6, 91.4, 69.3, 63.8, 59.3, 13.0. MS (EI, 75 eV, m/z) 442 (M⁺). Anal. Calcd. for C₁₅H₁₄WO₄: C, 40.72; H, 3.19. Found: C, 40.81; H, 3.31

Compound 16



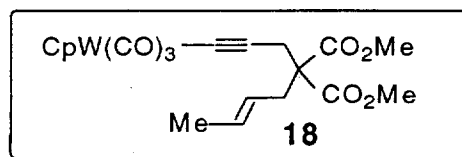
IR(neat, cm^{-1}): $\nu(\text{CO})$, 2054(s), 1953(s), $\nu(\text{C} \equiv \text{C})$ 2124(w), $\nu(\text{C}=\text{C})$ 1648 (s), ^1H NMR (300 MHz, CDCl_3): δ 5.52-5.72 (2H, m), 5.56 (5H, s), 4.21 (2H, s), 3.95 (2H, d, $J = 7.2$ Hz), 1.63 (3H, d, $J = 6.2$ Hz). ^{13}C NMR (75.0 MHz, CDCl_3): δ 229.0, 211.7, 129.7, 127.4, 124.7, 91.4, 69.4, 59.2, 17.7. MS (EI, 75 eV, m/z) 442 (M^+). Anal. Calcd. for $\text{C}_{15}\text{H}_{14}\text{WO}_4$: C, 40.72; H, 3.19. Found: C, 40.66; H, 3.11.

Compound 17



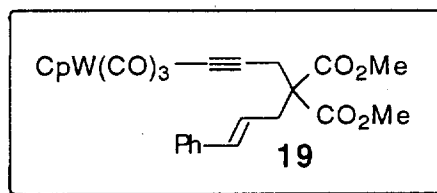
IR(neat, cm^{-1}): $\nu(\text{CO})$ 2053 (s), 1960 (s), 1710(s), $\nu(\text{C} \equiv \text{C})$ 2145(w), $\nu(\text{C}=\text{C})$ 1657(s), ^1H NMR (300 MHz, CDCl_3): δ 5.65 (1H, m), 5.53 (5H, s), 5.12 (1H, dd, $J = 16.8, 2.0$ Hz), 5.07 (1H, dd, $J = 10.8, 1.9$ Hz), 3.68 (6H, s), 2.93 (2H, s), 2.73 (1H, d, $J = 8.0$ Hz). ^{13}C NMR (75.0 MHz, CDCl_3): δ 229.6, 211.8, 170.6, 132.3, 122.3, 119.0, 91.3, 61.1, 58.1, 52.2, 36.5, 26.2. MS (EI, 75 eV, m/z) 542 (M^+). Anal. Calcd. for $\text{C}_{19}\text{H}_{18}\text{WO}_7$: C, 42.09; H 3.35, . Found: C, 41.66; H, 3.18.

Compound 18



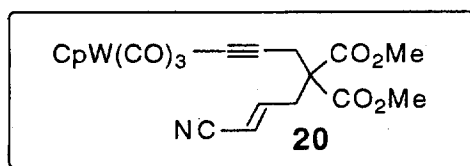
IR(neat, cm^{-1}): $\nu(\text{CO})$ 2053 (s), 1950 (s), 1720(s), $\nu(\text{C} \equiv \text{C})$ 2131(w), $\nu(\text{C}=\text{C})$ 1647(s), ^1H NMR (400 MHz, CDCl_3): δ 5.57 (1H, m), 5.52 (5H, s), 5.20 (1H, m), 3.65 (6 H, s), 2.92 (2H, s), 2.68 (2H, d, $J = 6.8$ Hz), 1.67 (3H, t, $J = 6.0$ Hz); ^{13}C NMR (100.0 MHz, CDCl_3): δ 229.8, 211.7, 170.9, 129.9, 122.7, 121.4, 91.4, 71.2, 58.4, 52.3, 35.4, 26.2, 18.0. MS (EI, 75 eV, m/z) 556 (M^+). Anal. Calcd. for $\text{C}_{20}\text{H}_{20}\text{WO}_7$: C, 43.16; H, 3.62. Found: C, 43.11; H, 3.58.

Compound 19



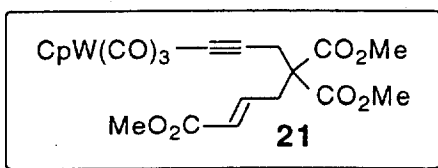
IR(neat, cm^{-1}): $\nu(\text{CO})$ 2050 (s), 1956 (s), 1720(s), $\nu(\text{C} \equiv \text{C})$ 2143(w), $\nu(\text{C}=\text{C})$ 1643(s), ^1H NMR (400 MHz, CDCl_3): δ 6.48 (1H, d, $J = 15.6$ Hz), 6.04 (1H, m), 5.54 (5H, s), 3.73 (6 H, s), 2.99 (2H, s), 2.92 (2H, d, $J = 6.8$ Hz); ^{13}C NMR (100.0 MHz, CDCl_3): δ 229.6, 211.8, 170.8, 137.2, 134.0, 128.4, 127.2, 126.2, 123.9, 122.5, 91.4, 61.4, 58.5, 52.4, 36.0, 26.6. MS (EI, 75 eV, m/z) 618 (M^+). Anal. Calcd. for $\text{C}_{25}\text{H}_{22}\text{WO}_7$: C, 48.54; H, 3.59. Found: C, 48.51; H, 3.75.

Compound 20



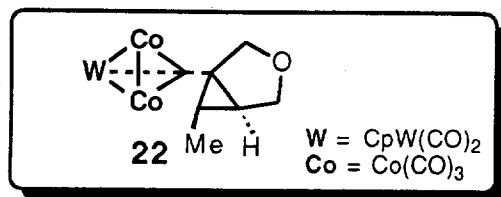
IR(neat, cm^{-1}): $\nu(\text{CO})$ 2050 (s), 1955 (s), 1721(s), $\nu(\text{C} \equiv \text{N})$ 2125(w), $\nu(\text{C}=\text{C})$ 1643(s), ^1H NMR (400 MHz, CDCl_3): trans isomer δ 6.60 (1H, dt, $J = 15.6, 7.0$ Hz), 5.51 (5H, s), 5.45 (1H, d, $J = 15.6$ Hz), 3.66 (6H, s), 2.90 (2 H, s), 2.86 (2H, d, $J = 7.0$ Hz), cis isomer 6.48 (1H, dt, $J = 10.6, 6.6$ Hz), 5.55 (5H, s), 5.45 (1H, d, $J = 10.6$ Hz), 3.70 (6H, s), 3.12 (2H, d, $J = 6.6$ Hz), 2.95 (2H, s); ^{13}C NMR (100.0 MHz, CDCl_3): δ trans isomer, 228.7, 212.0, 169.6, 149.9, 120.8, 116.7, 102.9, 91.3, 57.8, 52.5, 36.2, 26.7; cis isomer 229.3, 212.2, 169.8, 149.1, 120.7, 115.3, 102.1, 91.3, 63.3, 57.5, 34.7, 27.1. MS (EI, 75 eV, m/z) 567 (M^+). Anal. Calcd. for $\text{C}_{20}\text{H}_{17}\text{WNO}_7$: C, 42.35; H, 3.02. Found: C, 42.33; H, 3.09.

Compound 21



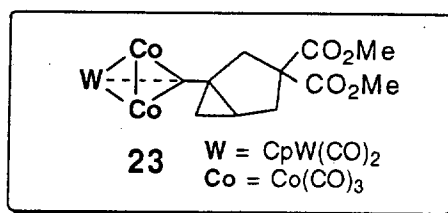
IR(neat, cm^{-1}): $\nu(\text{CO})$ 2053 (s), 1950 (s), 1720(s), 1627(s), ^1H NMR (400 MHz, CDCl_3): δ 6.77 (1H, dt, $J = 16.2, 7.8$ Hz), 5.90 (1H, d, $J = 16.2$ Hz), 5.51 (5H, s), 3.75 (6 H, br s), 2.93 (3H, s), 3.68 (6H, s), 2.90 (1H, d, $J = 7.8$ Hz); ^{13}C NMR (100.0 MHz, CDCl_3): δ 229.2, 211.8, 170.0, 166.1, 142.7, 124.5, 121.4, 91.3, 62.4, 57.7, 52.4, 51.2, 35.0, 26.6. MS (EI, 75 eV, m/z): 600 (M^+). Anal. Calcd. for $\text{C}_{21}\text{H}_{20}\text{WO}_9$: C, 41.00; H, 3.36. Found: C, 40.59; H, 3.28.

Compound 22



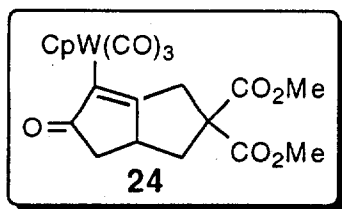
IR(neat, cm^{-1}): $\nu(\text{CO})$ 2058(s), 2025(s), 2006(s), 2001(s), 1936(s), 1871(m). ^1H NMR (300MHz, dg-toluene): δ 4.64 (5H, s), 4.61 (1H, ABq, $J = 11.2$ Hz), 4.46 (1H, ABq, $J = 11.2$ Hz), 4.35 (1H, br d, $J = 9.2$ Hz), 4.07 (1H, br d, $J = 9.2$ Hz), 2.07 (1H, br s), 1.56 (2H, br s). ^{13}C NMR (100 MHz, CDCl_3): δ 274.2, 203.8, 88.6, 75.0, 67.8, 60.4, 41.6, 33.5, 7.5. MS (EI, 75 eV, m/z): 700 (M^+). Anal. Calcd. for $\text{C}_{20}\text{H}_{14}\text{WCo}_2\text{O}_9$: C, 34.29; H, 2.02. Found: C, 34.23; H, 2.12.

Compound 23



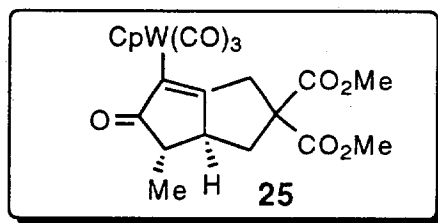
IR(neat, cm^{-1}): $\nu(\text{CO})$ 2059(s), 2023(s), 2002(s), 2000(s), 1941(s), 1876 (m). ^1H NMR (300MHz, dg-toluene): δ 4.75 (s, 5H), 3.73 (1H, ABq, $J = 14.2$ Hz), 3.56 (3H, s), 3.49 (3H, s), 3.41 (1H, ABq, $J = 14.2$ Hz), 3.13 (1H, d, $J = 15.2$ Hz), 3.02 (1H, dd, $J = 15.2, 2.8$ Hz), 2.02 (1H, m), 1.65 (2H, m). ^{13}C NMR (100 MHz, CDCl_3): δ 280.5, 203.6, 173.2, 172.0, 89.1, 59.7, 56.4, 53.0, 46.4, 37.6, 36.0, 27.7. MS (EI, 75 eV, m/z): 800 (M^+). Anal. Calcd. for $\text{C}_{24}\text{H}_{18}\text{WCo}_2\text{O}_{12}$: C, 36.00; H, 2.27. Found: C, 35.97; H, 2.25.

Compound 24



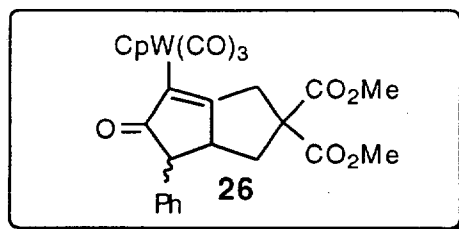
IR(neat, cm^{-1}): $\nu(\text{CO})$ 2050(s), 1948(s), 1756(s), 1720 (s). ^1H NMR (300 MHz, CDCl_3): δ 5.51 (s, 5 H), 3.74 (s, 3H), 3.69 (s, 3H), 3.29 (1H, ABq, $J = 18.6$ Hz), 3.05 (1H, ABq, $J = 18.6$ Hz), 3.04 (1H, m), 2.92 (1H, dd, $J = 12.6, 7.8$ Hz), 2.58 (1H, dd, $J = 12.6, 6.3$ Hz), 1.98 (1H, ABq, $J = 17.4$ Hz), 1.48 (1H, t, $J = 12.6$ Hz). ^{13}C NMR (100 MHz, CDCl_3): δ 227.9, 216.6, 215.7, 215.2, 194.9, 172.3, 172.1, 124.5, 90.9, 60.0, 52.9, 52.8, 46.8, 41.5, 39.8. MS (EI, 75 eV, m/z) 570 (M^+). Anal. Calcd. for $\text{C}_{20}\text{H}_{18}\text{WO}_8$: C, 42.10; H, 3.18. Found: C, 42.12; H, 3.16.

Compound 25



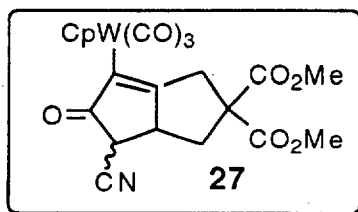
IR(neat, cm^{-1}): $\nu(\text{CO})$ 2052(s), 1951(s), 1752(s), 1720(s). ^1H NMR (300 MHz, CDCl_3): δ 5.53 (s, 5 H), 3.74 (s, 3H), 3.70 (s, 3H), 3.32 (1H, ABq, $J = 18.0$ Hz), 3.05 (1H, ABq, $J = 18.0$ Hz), 2.79 (1H, dd, $J = 12.0, 7.0$ Hz), 2.68 (1H, m), 1.95 (1H, m), 1.58 (1H, t, $J = 12.0$ Hz), 1.15 (3H, d, $J = 6.8$ Hz), cis-isomer, selected peaks, 3.01 (1H, ABq, $J = 18.0$ Hz), 2.52 (1H, dd, $J = 12.0, 6.0$ Hz), 1.62 (1H, t, $J = 12.0$ Hz), 0.93 (3H, d, $J = 6.3$ Hz), the remaining peaks are masked with those of the *trans*-isomer. ^{13}C NMR (100 MHz, CDCl_3): *trans* isomer, 227.9, 217.8, 216.8, 215.7, 192.3, 123.0, 90.9, 60.1, 55.2, 53.2, 53.0, 50.9, 49.2, 39.6, 39.114.7, *cis* isomer, 227.9, 218.9, 217.0, 215.7, 193.5, 172.3, 124.6, 90.9, 59.6, 52.9, 52.8, 51.0, 42.8, 40.3, 34.4, 29.6, 13.5. MS (EI, 75 eV, m/z) 584 (M^+). Anal. Calcd. for $\text{C}_{21}\text{H}_{20}\text{WO}_8$: C, 43.17; H, 3.45. Found: C, 43.02; H, 3.55.

Compound 26



IR(neat, cm⁻¹): ν (CO) 2051(s), 1951(s), 1753 (m), 1712 (s). ¹H NMR (300 MHz, CDCl₃): *trans* isomer, δ 7.08-7.31 (m, 5H), 5.57 (s, 5H), 3.73 (3H, s), 3.72 (3H, s), 3.47 (1H, ABq, J = 18.0 Hz), 3.25 (1H, m), 3.17 (1H, br s), 3.08 (1H, ABq, J = 18.0 Hz), 2.83 (1H, dd, J = Hz), 2.85 (1H, dd, J = 12.0, 8.1 Hz), 1.80 (1H, t, J = 12.0 Hz); *cis* isomer (selected peaks), 7.08-7.31 (m, 5H), 5.63 (s, 5H), 1.96 (1H, t, J = 12.0 Hz), the remaining peaks are masked with those of the *trans*-isomer. ¹³C NMR (100 MHz, CDCl₃): *trans* isomer δ 227.7, 216.8, 214.6, 172.1, 171.9, 139.6, 128.6, 128.2, 126.6, 123.4, 90.9, 60.6, 60.3, 55.7, 52.8, 52.9, 50.4, 39.5, 38.5. *cis* isomer, selected peaks, 208.0, 182.8, 171.0, 170.9, 138.4, 128.4, 127.0, 124.4, 65.7, 53.9, 38.5, 35.1. MS (EI, 75 eV, *m/z*) 646 (M⁺). Anal. Calcd. for C₂₆H₂₂WO₈: C, 48.29; H, 3.43. Found: C, 48.29; H, 3.43.

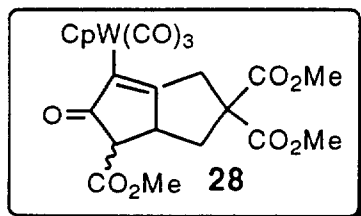
Compound 27



IR(neat, cm⁻¹): ν (CO) 2059(s), 1950(s), 1753 (m), 1712 (s). ¹H NMR (400 MHz, d₆-benzene): *trans* isomer, δ 4.74 (s, 5H), 3.58 (1H, ABq, J = 15.6 Hz), 3.25 (3H, s), 3.22 (3H, s), 3.10 (1H, m), 2.55 (1H, dd, J = 12.0, 8.6 Hz), 2.42 (1H, d, J = 5.0 Hz), 1.44 (1H, t, J = 12.0 Hz); *cis* isomer (selected peaks), 4.67 (s, 5H), 3.51 (1H, ABq, J = 13.0 Hz), 3.49 (1H, ABq, J = 13.0 Hz), 2.95 (1H, dd, J = 12.0, 8.6 Hz), 2.89 (1H, d, J = 7.4 Hz), 2.70 (1H, m), 2.00 (1H, t, J = 12.0 Hz), the remaining peaks are masked with those of the *trans*-isomer. ¹³C NMR (100 MHz, CDCl₃): *trans* isomer δ 226.5, 216.5, 215.4, 204.4, 191.6, 171.3, 123.3, 117.3, 91.1, 59.1, 53.1, 50.8, 42.5, 39.4, 38.5. *cis* isomer, selected peaks, 225.8, 214.5, 204.3, 194.1, 171.4, 110.4, 91.1, 65.4, 58.7, 52.8, 47.8, 39.6, 36.2, the remaining peaks are overlapped with those of the *trans*-isomer. MS (EI, 75

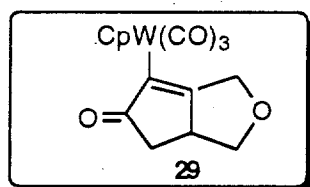
eV, m/z) 595 (M^+). Anal. Calcd. for $C_{21}H_{17}WNO_8$: C, 42.38; H, 2.88. Found: C, 42.44; H, 2.75.

Compound 28



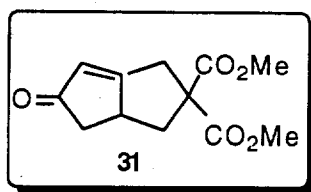
IR(neat, cm^{-1}): $\nu(CO)$ 2058(s), 1959(s), 1752 (m), 1710 (s). 1H NMR (300 MHz, $CDCl_3$): *trans* isomer, δ 5.51 (s, 5H), 3.71 (3H, s), 3.65 (3H, s), 3.59 (3H, s), 3.51 (1H, m), 3.44 (1H, m), 3.35 (1H, ABq, $J = 15.2$ Hz), 2.98 (1H, br s), 2.97 (ABq, $J = 15.2$ Hz), 2.78 (1H, dd, $J = 12.8, 8.1$ Hz), 1.63 (1H, t, $J = 11.6$ Hz); *cis* isomer (selected peaks), 5.45 (s, 5H), 3.62 (s, 3H), 3.30 (1H, AB q, $J = 14.8$ Hz), 3.12 (1H, AB q, $J = 14.8$ Hz), 2.63 (1H, dd, $J = 12.0, 7.8$ Hz), the remaining peaks are masked with those of the *trans*-isomer. ^{13}C NMR (100 MHz, $CDCl_3$): *trans* isomer δ 227.5, 216.5, 215.4, 208.9, 193.2, 171.7, 170.4, 122.2, 90.8, 59.6, 59.1, 52.9, 52.8, 52.1, 50.4, 39.4, 38.4. MS (EI, 75 eV, m/z) 628 (M^+). Anal. Calcd. for $C_{22}H_{20}WO_{10}$: C, 42.03; H, 3.21. Found: C, 43.13; H, 3.20.

Compound 29



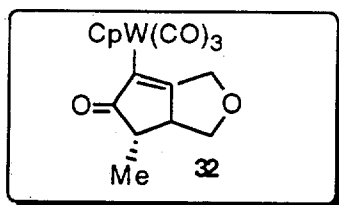
IR(neat, cm^{-1}): $\nu(CO)$ 2054(s), 1958(s), 1752 (s). 1H NMR (300 MHz, $CDCl_3$): δ 5.58 (5H, s, Cp), 4.72 (1H, ABq, $J = 16.8$ Hz), 4.28 (1H, t, $J = 7.8$ Hz), 4.20 (1H, ABq, $J = 16.8$ Hz), 3.23 (1H, m), 3.07 (1H, dd, $J = 14.6, 7.8$ Hz), 2.63 (1H, dd, $J = 12.6, 6.8$ Hz), 2.00 (1H, dd, $J = 12.6, 3.2$ Hz). ^{13}C NMR (75.0 MHz, $CDCl_3$): δ 222.9, 216.6, 216.1, 215.8, 193.8, 110.2, 90.9, 72.4, 69.0, 47.8, 38.8. MS (EI, 75 eV, m/z) 456 (M^+). Anal. Calcd. for $C_{15}H_{12}WO_5$: C, 39.50; H, 2.65. Found: C, 39.47; H, 2.75.

Compound 31



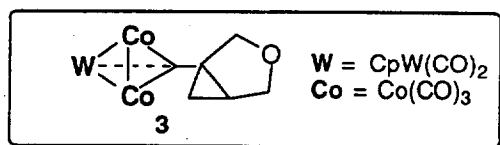
IR(neat, cm^{-1}): $\nu(\text{CO})$ 1750 (s), 1718(s). ^1H NMR (300 MHz, CDCl_3): δ 5.87 (1H, s), 3.73 (3H, s), 3.68 (3H, s), 3.23 (2H, ABq, $J = 16.2$ Hz), 3.04 (1H, m), 2.73 (1H, dd, $J = 12.6, 8.3$ Hz), 2.58 (1H, dd, $J = 17.6, 6.0$ Hz), 2.05 (1H, dd, $J = 17.6, 2.8$ Hz), 1.75 (1H, t, $J = 12.6$ Hz) ^{13}C NMR (75.0 MHz, CDCl_3): δ 209.2, 216.6, 185.1, 171.7, 171.1, 115.5, 90.8, 60.6, 53.1, 52.9, 44.9, 42.0, 38.9, 35.1. HRMS calcd. for $\text{C}_{11}\text{H}_{14}\text{O}_5$: 226.0841; found: 226.08439.

Compound 32



IR(neat, cm^{-1}): $\nu(\text{CO})$ 2055(s), 1955(s), 1750 (s). ^1H NMR (300 MHz, CDCl_3): δ 5.58 (5H, s, Cp), 4.72 (1H, ABq, $J = 16.8$ Hz), 4.22 (1H, t, $J = 7.8$ Hz), 4.20 (1H, ABq, $J = 15.6$ Hz), 3.37 (2H, m), 2.64 (1H, m), 0.96 (3H, d, $J = 6.8$ Hz), ^{13}C NMR (75.0 MHz, CDCl_3): δ 227.0, 219.3, 216.3, 192.7, 90.9, 69.4, 67.9, 51.8, 42.0, 13.5. MS (EI, 75 eV, m/z) 470 (M^+). Anal. Calcd. for $\text{C}_{16}\text{H}_{14}\text{WO}_5$: C, 40.88; H, 3.00. Found: C, 40.47; H, 3.08.

Tables of crystal data, bond distances and angles and ORTEP drawings of Compound **3**



Summary of Crystal Data and Intensity Collection 98JL09 (980138)

Empirical Formula	C ₁₉ H ₁₂ Co ₂ O ₉ W
Color; Habit	Black; Columnar
Crystal Size (mm)	0.10 x 0.10 x 0.30
Space Group	P2 ₁ /n; Monoclinic
Unit Cell Dimensions	$a = 8.1458(1) \text{ \AA}$ $b = 16.1177(1) \text{ \AA}; \beta = 95.437(1)^\circ$ $c = 15.6961(2) \text{ \AA}$
Volume	2051.5(6) $\text{\AA}^3$
Z	4
Formula Weight	686.0
Density(calc.)	2.221 Mg/m ³
Absorption Coefficient	7.241 mm ⁻¹
F(000)	1304
Diffractionmeter Used	Siemens Smart CCD
Radiation	MoK α ($\lambda = 0.71073 \text{ \AA}$)
Temperature (K)	295
Monochromator	Highly oriented graphite crystal
2 θ Range	2.5 to 56.0 $^\circ$
Scan Speed	20.00 seconds/frame
Scan Range (ω)	0.30 $^\circ$ / frame
Index Ranges	$-10 \leq h \leq 10, -12 \leq k \leq 21, -20 \leq l \leq 18$
Reflections Collected	12202(9878 $\geq$ 3.0 σ (I))
Independent Reflections	4468(3018 $\geq$ 3.0 σ (I))(R _{int} =4.08%)
System Used	Siemens SHELXTL PLUS (VMS)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Extinction Correction	$\chi = 0.00019(4)$, where $F^* = F [1 + 0.002\chi F^2 / \sin(2\theta)]^{-1/4}$
Hydrogen Atoms	Located on difference map
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0013F^2$
Number of Parameters Refined	281
Final R Indices (obs. data)	R = 0.0368, R _w = 0.0385
Goodness-of-Fit	0.91
Largest and Mean Δ/σ	0.002, 0.000
Data-to-Parameter Ratio	10.7:1
Largest Difference Peak/Hole	1.59/-1.97 e $\text{\AA}^{-3}$

A crystal of dimensions 0.10 x 0.10 x 0.30 mm for $C_{19}H_{12}Co_2O_9W$ was selected for indexing and intensity data collection on a Siemens Smart-CCD diffractometer equipped with a normal focus, 3 KW sealed tube X-ray source. Intensity data were collected in 1271 frames with increasing ω (width of 0.3 deg per frame). Unit cell dimensions were determined by a least-squares fit of 7626 reflections with $5 < 2\theta < 50$ deg. Absorption correction was based on 8757 symmetry-equivalent reflections using the SHELXTL-PC program package ($T_{min,max} = 0.468, 0.915$). On the basis of systematic absences, statistics of intensity distribution, and successful solution and refinement of the structure, the space group was determined to be $P2_1/n$ (No. 14).

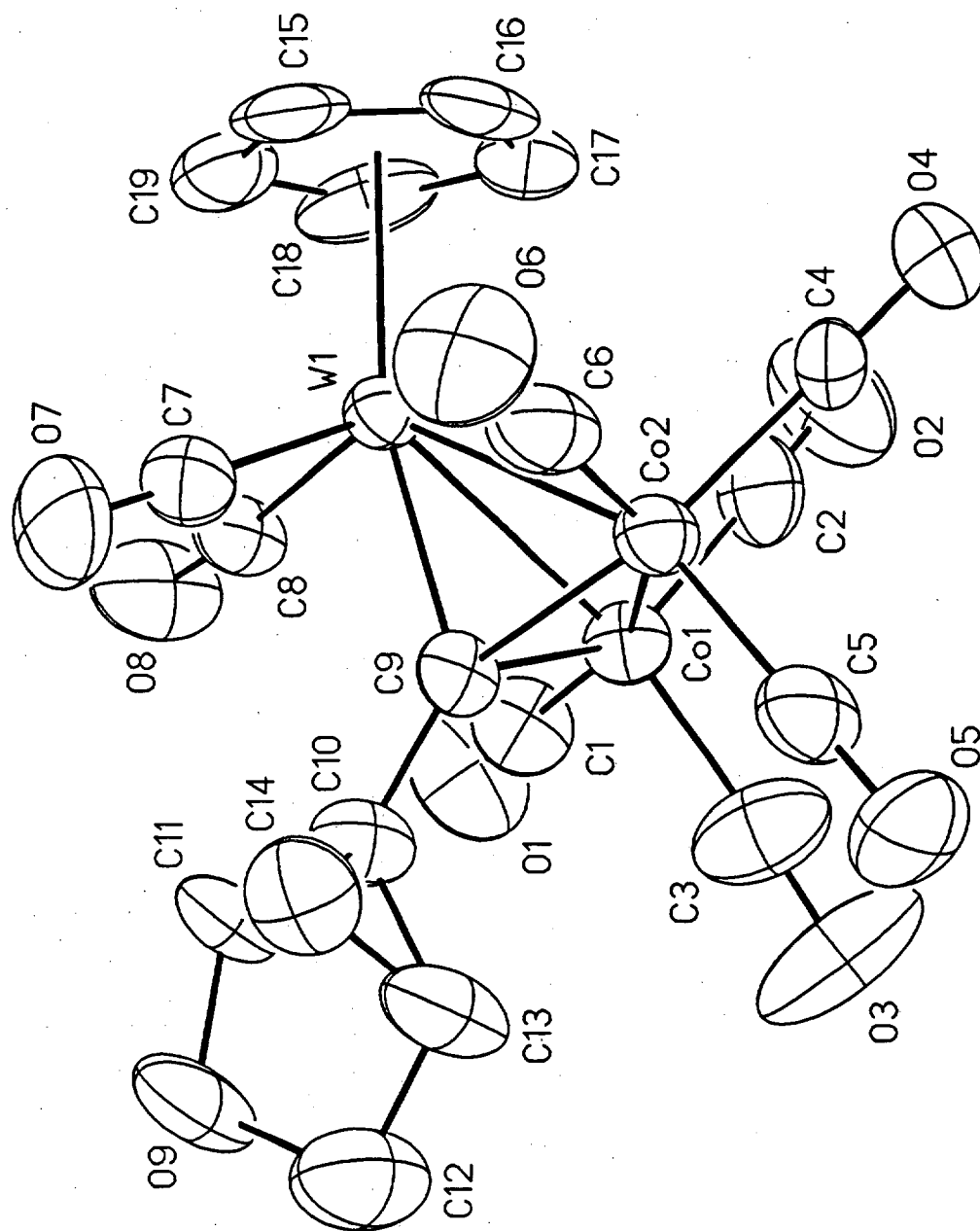


Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	x	y	z	U(eq)
W(1)	12659(1)	3586(1)	11476(1)	34(1)
Co(1)	15030(1)	3071(1)	10503(1)	42(1)
Co(2)	14963(1)	2478(1)	11968(1)	37(1)
O(1)	13688(12)	3535(6)	8779(5)	105(4)
O(2)	17285(10)	4468(6)	10822(5)	103(4)
O(3)	16914(13)	1670(6)	9925(6)	123(5)
O(4)	17550(8)	3607(4)	12715(5)	70(3)
O(5)	16834(10)	922(5)	11948(5)	86(3)
O(6)	13102(9)	2099(5)	13426(4)	74(3)
O(7)	9817(8)	2406(5)	11895(5)	84(3)
O(8)	10505(10)	3898(5)	9743(4)	84(3)
O(9)	10901(9)	1012(4)	9465(4)	72(3)
C(1)	14175(13)	3352(6)	9467(6)	64(4)
C(2)	16413(13)	3933(7)	10723(6)	66(4)
C(3)	16231(14)	2235(7)	10154(7)	75(4)
C(4)	16544(10)	3158(5)	12436(5)	46(3)
C(5)	16078(12)	1516(6)	11928(6)	55(3)
C(6)	13829(11)	2252(5)	12857(5)	50(3)
C(7)	10859(10)	2841(6)	11733(6)	50(3)
C(8)	11335(11)	3763(5)	10357(6)	54(3)
C(9)	13304(8)	2434(4)	10978(4)	33(2)
C(10)	12421(10)	1713(5)	10599(5)	43(2)
C(11)	11265(11)	1830(6)	9787(5)	52(3)
C(12)	12353(16)	496(7)	9682(7)	81(4)
C(13)	13073(13)	842(5)	10536(7)	68(4)
C(14)	11857(12)	1009(6)	11151(6)	60(3)
C(15)	11814(12)	4322(6)	12645(6)	61(3)
C(16)	13517(12)	4360(5)	12724(5)	57(3)
C(17)	14024(11)	4790(5)	12023(6)	58(3)
C(18)	12601(13)	5022(5)	11501(6)	63(4)
C(19)	11231(11)	4738(6)	11876(6)	62(3)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2. Bond lengths (Å)

W(1)-Co(1)	2.704 (1)	W(1)-Co(2)	2.652 (1)
W(1)-C(7)	1.966 (9)	W(1)-C(8)	1.993 (9)
W(1)-C(9)	2.101 (7)	W(1)-C(15)	2.343 (10)
W(1)-C(16)	2.371 (8)	W(1)-C(17)	2.357 (8)
W(1)-C(18)	2.315 (9)	W(1)-C(19)	2.309 (10)
Co(1)-Co(2)	2.494 (1)	Co(1)-C(1)	1.767 (10)
Co(1)-C(2)	1.803 (11)	Co(1)-C(3)	1.781 (12)
Co(1)-C(9)	1.945 (7)	Co(2)-C(4)	1.794 (8)
Co(2)-C(5)	1.801 (9)	Co(2)-C(6)	1.782 (9)
Co(2)-C(9)	1.961 (6)	O(1)-C(1)	1.153 (12)
O(2)-C(2)	1.119 (14)	O(3)-C(3)	1.143 (15)
O(4)-C(4)	1.148 (10)	O(5)-C(5)	1.137 (12)
O(6)-C(6)	1.144 (11)	O(7)-C(7)	1.148 (11)
O(8)-C(8)	1.144 (11)	O(9)-C(11)	1.433 (11)
O(9)-C(12)	1.460 (14)	C(9)-C(10)	1.463 (10)
C(10)-C(11)	1.523 (11)	C(10)-C(13)	1.507 (12)
C(10)-C(14)	1.525 (12)	C(12)-C(13)	1.517 (15)
C(13)-C(14)	1.472 (15)	C(15)-C(16)	1.383 (14)
C(15)-C(19)	1.422 (13)	C(16)-C(17)	1.395 (13)
C(17)-C(18)	1.405 (13)	C(18)-C(19)	1.388 (14)

Table 3. Bond angles ($^{\circ}$)

Co(1)-W(1)-Co(2)	55.5(1)	Co(1)-W(1)-C(7)	121.0(3)
Co(2)-W(1)-C(7)	92.9(2)	Co(1)-W(1)-C(8)	84.4(3)
Co(2)-W(1)-C(8)	131.5(3)	C(7)-W(1)-C(8)	85.2(4)
Co(1)-W(1)-C(9)	45.6(2)	Co(2)-W(1)-C(9)	47.0(2)
C(7)-W(1)-C(9)	75.8(3)	C(8)-W(1)-C(9)	86.1(3)
Co(1)-W(1)-C(15)	151.2(2)	Co(2)-W(1)-C(15)	111.4(2)
C(7)-W(1)-C(15)	82.4(3)	C(8)-W(1)-C(15)	116.3(3)
C(9)-W(1)-C(15)	147.3(3)	Co(1)-W(1)-C(16)	117.2(2)
Co(2)-W(1)-C(16)	88.2(2)	C(7)-W(1)-C(16)	108.6(3)
C(8)-W(1)-C(16)	138.2(3)	C(9)-W(1)-C(16)	135.1(3)
C(15)-W(1)-C(16)	34.1(3)	Co(1)-W(1)-C(17)	96.9(2)
Co(2)-W(1)-C(17)	98.7(2)	C(7)-W(1)-C(17)	139.8(4)
C(8)-W(1)-C(17)	113.6(3)	C(9)-W(1)-C(17)	137.5(3)
C(15)-W(1)-C(17)	57.4(3)	C(16)-W(1)-C(17)	34.3(3)
Co(1)-W(1)-C(18)	109.5(3)	Co(2)-W(1)-C(18)	133.1(3)
C(7)-W(1)-C(18)	126.2(4)	C(8)-W(1)-C(18)	82.0(3)
C(9)-W(1)-C(18)	153.6(3)	C(15)-W(1)-C(18)	58.2(3)
C(16)-W(1)-C(18)	57.7(3)	C(17)-W(1)-C(18)	35.0(3)
Co(1)-W(1)-C(19)	143.7(2)	Co(2)-W(1)-C(19)	145.4(2)
C(7)-W(1)-C(19)	91.7(3)	C(8)-W(1)-C(19)	83.1(3)
C(9)-W(1)-C(19)	164.2(3)	C(15)-W(1)-C(19)	35.6(3)
C(16)-W(1)-C(19)	57.9(3)	C(17)-W(1)-C(19)	58.1(3)
C(18)-W(1)-C(19)	34.9(4)	W(1)-Co(1)-Co(2)	61.2(1)
W(1)-Co(1)-C(1)	101.6(3)	Co(2)-Co(1)-C(1)	154.9(3)
W(1)-Co(1)-C(2)	97.0(3)	Co(2)-Co(1)-C(2)	100.7(3)
C(1)-Co(1)-C(2)	99.3(5)	W(1)-Co(1)-C(3)	148.1(4)
Co(2)-Co(1)-C(3)	93.1(3)	C(1)-Co(1)-C(3)	95.2(5)
C(2)-Co(1)-C(3)	106.9(5)	W(1)-Co(1)-C(9)	50.6(2)
Co(2)-Co(1)-C(9)	50.6(2)	C(1)-Co(1)-C(9)	104.6(4)
C(2)-Co(1)-C(9)	142.6(4)	C(3)-Co(1)-C(9)	99.1(4)
W(1)-Co(2)-Co(1)	63.3(1)	W(1)-Co(2)-C(4)	99.7(3)
Co(1)-Co(2)-C(4)	93.8(3)	W(1)-Co(2)-C(5)	156.2(3)
Co(1)-Co(2)-C(5)	104.0(3)	C(4)-Co(2)-C(5)	101.3(4)
W(1)-Co(2)-C(6)	87.7(3)	Co(1)-Co(2)-C(6)	149.1(3)
C(4)-Co(2)-C(6)	102.1(4)	C(5)-Co(2)-C(6)	98.7(4)
W(1)-Co(2)-C(9)	51.6(2)	Co(1)-Co(2)-C(9)	50.0(2)
C(4)-Co(2)-C(9)	139.5(3)	C(5)-Co(2)-C(9)	104.7(3)
C(6)-Co(2)-C(9)	104.1(3)	C(11)-O(9)-C(12)	107.8(7)
Co(1)-C(1)-O(1)	176.9(10)	Co(1)-C(2)-O(2)	176.8(10)
Co(1)-C(3)-O(3)	175.7(11)	Co(2)-C(4)-O(4)	178.0(7)
Co(2)-C(5)-O(5)	175.8(8)	Co(2)-C(6)-O(6)	179.3(8)
W(1)-C(7)-O(7)	179.0(8)	W(1)-C(8)-O(8)	175.4(8)
W(1)-C(9)-Co(1)	83.8(3)	W(1)-C(9)-Co(2)	81.4(2)
Co(1)-C(9)-Co(2)	79.4(3)	W(1)-C(9)-C(10)	136.3(5)
Co(1)-C(9)-C(10)	127.5(5)	Co(2)-C(9)-C(10)	128.8(5)
C(9)-C(10)-C(11)	118.9(7)	C(9)-C(10)-C(13)	127.1(7)
C(11)-C(10)-C(13)	104.7(7)	C(9)-C(10)-C(14)	121.5(7)
C(11)-C(10)-C(14)	111.9(7)	C(13)-C(10)-C(14)	58.1(6)
O(9)-C(11)-C(10)	105.8(7)	O(9)-C(12)-C(13)	103.6(8)
C(10)-C(13)-C(12)	106.9(8)	C(10)-C(13)-C(14)	61.6(6)
C(12)-C(13)-C(14)	114.7(9)	C(10)-C(14)-C(13)	60.3(6)
W(1)-C(15)-C(16)	74.1(5)	W(1)-C(15)-C(19)	70.9(6)
C(16)-C(15)-C(19)	107.8(8)	W(1)-C(16)-C(15)	71.8(5)
W(1)-C(16)-C(17)	72.3(5)	C(15)-C(16)-C(17)	108.7(8)
W(1)-C(17)-C(16)	73.4(5)	W(1)-C(17)-C(18)	70.9(5)
C(16)-C(17)-C(18)	107.6(8)	W(1)-C(18)-C(17)	74.1(5)

W(1)-C(18)-C(19)	72.3(5)	C(17)-C(18)-C(19)	108.4(8)
W(1)-C(19)-C(15)	73.5(6)	W(1)-C(19)-C(18)	72.8(5)
C(15)-C(19)-C(18)	107.4(8)		

Table 4. Anisotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
W(1)	37(1)	32(1)	34(1)	3(1)	-2(1)	-2(1)
Co(1)	46(1)	45(1)	37(1)	-1(1)	8(1)	1(1)
Co(2)	39(1)	37(1)	33(1)	4(1)	-3(1)	-1(1)
O(1)	125(7)	127(8)	57(5)	-29(5)	-18(5)	33(5)
O(2)	100(6)	114(7)	96(6)	-65(6)	20(5)	-8(5)
O(3)	146(9)	105(7)	127(8)	62(7)	71(7)	13(6)
O(4)	46(4)	87(5)	76(4)	-11(3)	-3(3)	-18(4)
O(5)	94(5)	60(5)	98(5)	34(4)	-17(4)	-14(4)
O(6)	87(5)	90(6)	48(4)	-2(4)	19(3)	14(4)
O(7)	57(4)	73(5)	128(7)	-15(4)	36(4)	-12(5)
O(8)	106(6)	78(5)	58(4)	29(4)	-40(4)	-1(4)
O(9)	92(5)	50(4)	67(4)	-15(4)	-34(4)	-8(3)
C(1)	74(7)	65(6)	51(6)	-16(5)	5(5)	7(5)
C(2)	62(6)	81(7)	58(6)	-14(6)	15(5)	4(5)
C(3)	91(8)	69(7)	71(7)	7(6)	40(6)	7(6)
C(4)	38(4)	56(5)	44(4)	8(4)	-3(3)	-1(4)
C(5)	60(5)	54(6)	48(5)	7(4)	-9(4)	-4(4)
C(6)	59(5)	46(5)	43(5)	7(4)	-5(4)	-5(4)
C(7)	45(5)	49(5)	57(5)	0(4)	14(4)	-6(4)
C(8)	61(5)	42(5)	57(5)	11(4)	-7(4)	-8(4)
C(9)	38(4)	33(4)	28(3)	1(3)	-3(3)	2(3)
C(10)	54(5)	38(4)	34(4)	-2(3)	-9(3)	-1(3)
C(11)	63(6)	44(5)	45(4)	-6(4)	-12(4)	-10(4)
C(12)	112(9)	54(6)	72(7)	-3(6)	-16(6)	-11(5)
C(13)	90(7)	33(5)	74(6)	10(5)	-33(6)	-6(5)
C(14)	70(6)	46(5)	61(6)	-5(5)	2(5)	11(5)
C(15)	68(6)	53(5)	64(6)	0(5)	15(5)	-34(5)
C(16)	83(7)	39(5)	46(5)	14(4)	-16(5)	-22(4)
C(17)	67(6)	33(5)	72(6)	-5(4)	2(5)	-27(5)
C(18)	106(9)	29(4)	53(5)	20(5)	-1(5)	-2(4)
C(19)	50(5)	63(6)	70(6)	21(5)	-7(4)	-25(5)

The anisotropic displacement factor exponent takes the form:

$$-2\pi^2(h^2a^{*2}U_{11} + \dots + 2hka^*b^*U_{12})$$

Table 5. H-Atom coordinates ($\times 10^4$) and isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	x	y	z	U
H(11A)	11941	2226	9402	80
H(11B)	10333	2133	9994	80
H(12A)	12881	777	9162	80
H(12B)	11993	-202	9776	80
H(13A)	14319	612	10780	80
H(14A)	10425	968	11006	80
H(14B)	12060	1022	11892	80
H(15A)	10947	4152	13083	80
H(16A)	13828	3946	13330	80
H(17A)	15154	4749	11913	80
H(18A)	12216	5394	11017	80
H(19A)	9911	4841	11737	80

Tables of crystal data, bond distances and angles and ORTEP drawings of Compound 6

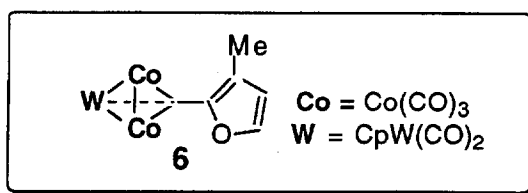


Table. Crystal Data and Conditions for Crystallographic Data Collection and Structure Refinement

TITLE	*** IC5946 ***
Formula	C19 H10 O9 Co2 W
Formular wt	683.99
Diffractionmeter used	Nonius
Space Group	Monoclinic P 21/n
a (angstrom)	8.6526(20)
b (angstrom)	15.313(3)
c (angstrom)	15.372(4)
beta (deg.)	95.96(3)
V (A**3)	2025.8(8)
Z	4
Dcalc. (g.cm**-3)	2.243
lambda (Angstrom)	0.71073
F(000)	1295.
Unit cell detn: #;(2theta range)	25;(18.62 - 26.84 deg.)
Scan type	theta/2theta
Scan width (deg.)	2(0.85+0.35tan(theta))
Scan Speed (deg./min)	2.06-8.24
(2Theta)max.	50.0
h k l ranges	(-10; 10)(0; 18)(0; 18)
mu (cm**-1)	74.534
Crystal size (mm)	0.60 x 0.55 x 0.45
Transmission	0.062; 0.098
Temperature (K)	295.
# of meas. reflns.	3553
# of obsd reflns. (I>2.0sig(I))	3048
# of unique reflns.	3553
Rf;Rw	0.031;0.038
GoF	1.81
Refinement program	NRCVAX
# of atoms	41
# of refined params.	281 (3048 out of 3553 reflns.)
Minimize function	Sum(w Fo-Fc **2)
Weights scheme	1/[sigma**2(Fo)]
The weight modifier K in KFo**2 is	0.000200
g (2nd. ext. coeff.) x 10E4	0.119(7)
(delta/sigma)max.	0.0299
Residual in final D-map (e/A**3)	-1.260; 1.370

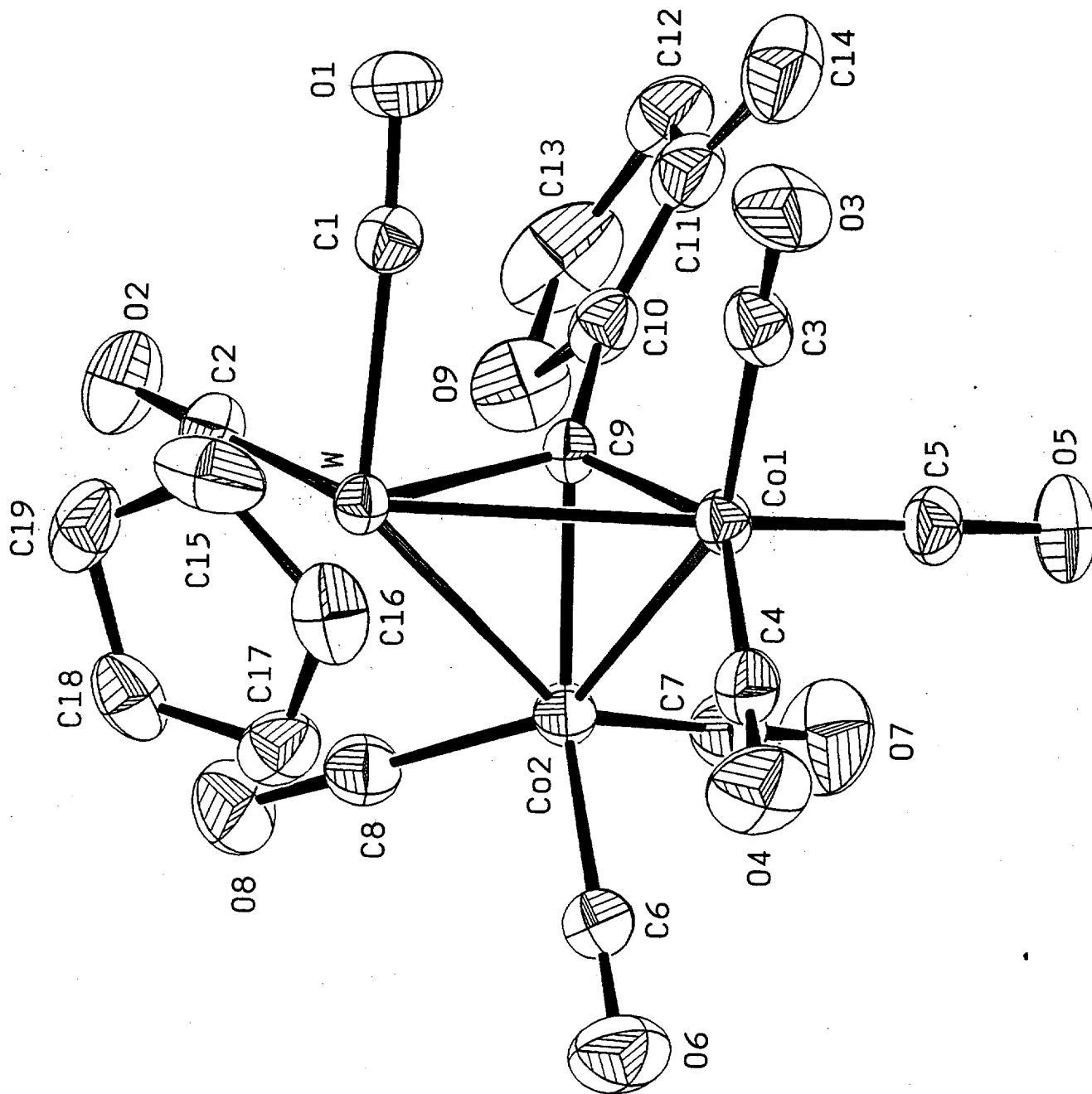
NOTE :

$$R_f = \text{Sum}(F_o - F_c) / \text{Sum}(F_o)$$

$$R_w = \text{Sqrt}[\text{Sum}(w(F_o - F_c)^2) / \text{Sum}(wF_o^2)]$$

$$\text{GoF} = \text{Sqrt}[\text{Sum}(w(F_o - F_c)^2) / (\text{No. of reflns} - \text{No. of params.})]$$

IC5946



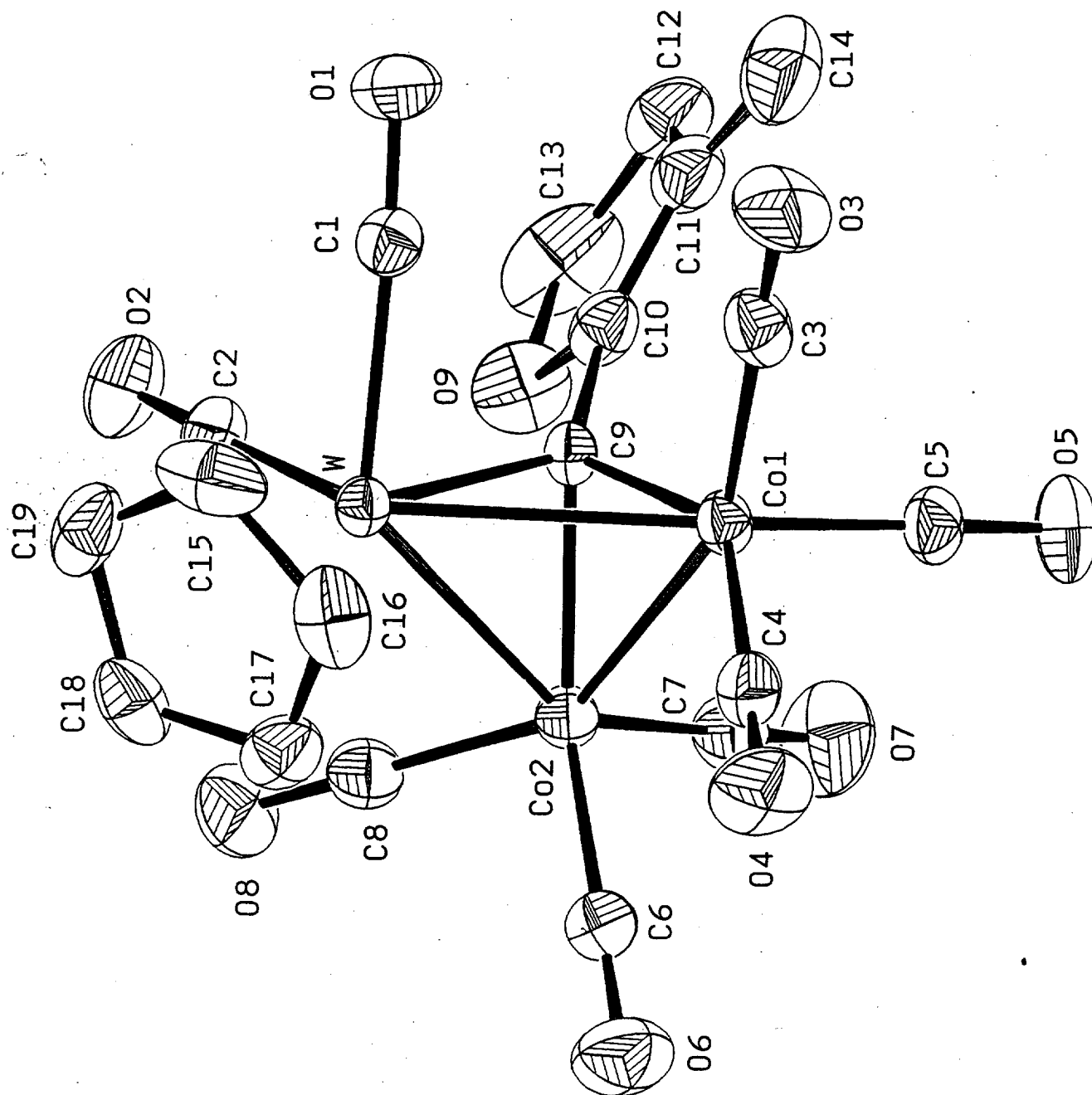


Table : Bond Distances and Bond Angles of IC5946

W-Co1	2.6597(9)	C2-O2	1.134(8)
W-Co2	2.6620(10)	C3-O3	1.109(9)
W-C1	1.964(7)	C4-O4	1.133(9)
W-C2	1.981(7)	C5-O5	1.133(9)
W-C9	2.091(6)	C6-O6	1.115(9)
W-C15	2.306(7)	C7-O7	1.118(10)
W-C16	2.342(7)	C8-O8	1.136(9)
W-C17	2.339(7)	C9-C10	1.454(9)
W-C18	2.315(7)	C10-C11	1.311(11)
W-C19	2.282(7)	C10-O9	1.468(9)
Co1-Co2	2.4637(14)	C11-C12	1.373(11)
Co1-C3	1.797(7)	C11-C14	1.391(13)
Co1-C4	1.793(7)	C12-C13	1.436(16)
Co1-C5	1.778(7)	C13-O9	1.309(12)
Co1-C9	1.922(6)	C15-C16	1.368(14)
Co2-C6	1.805(7)	C15-C19	1.374(14)
Co2-C7	1.793(8)	C16-C17	1.349(12)
Co2-C8	1.763(7)	C17-C18	1.343(11)
Co2-C9	1.957(6)	C18-C19	1.378(12)
C1-O1	1.143(8)		
Co1-W-Co2	55.16(3)	C4-Co1-C5	105.8(3)
Co1-W-C1	86.90(19)	C4-Co1-C9	142.6(3)
Co1-W-C2	126.00(19)	C5-Co1-C9	97.1(3)
Co1-W-C9	45.82(16)	W-Co2-Co1	62.38(3)
Co1-W-C15	117.0(3)	W-Co2-C6	106.60(24)
Co1-W-C16	92.71(20)	W-Co2-C7	147.5(3)
Co1-W-C17	101.33(19)	W-Co2-C8	87.34(23)
Co1-W-C18	133.35(20)	W-Co2-C9	51.10(17)
Co1-W-C19	149.68(22)	Co1-Co2-C6	95.42(24)
Co2-W-C1	125.77(19)	Co1-Co2-C7	102.25(24)
Co2-W-C2	90.69(19)	Co1-Co2-C8	149.05(23)
Co2-W-C9	46.74(17)	Co1-Co2-C9	49.95(17)
Co2-W-C15	146.25(24)	C6-Co2-C7	103.1(4)
Co2-W-C16	112.98(22)	C6-Co2-C8	99.6(3)
Co2-W-C17	91.35(19)	C6-Co2-C9	143.4(3)
Co2-W-C18	102.07(21)	C7-Co2-C8	100.5(3)
Co2-W-C19	136.33(24)	C7-Co2-C9	96.7(3)
C1-W-C2	82.1(3)	C8-Co2-C9	106.8(3)
C1-W-C9	79.1(3)	W-C1-O1	176.8(6)
C1-W-C15	82.4(3)	W-C2-O2	177.0(6)
C1-W-C16	105.1(3)	Co1-C3-O3	178.2(7)
C1-W-C17	137.2(3)	Co1-C4-O4	178.1(7)
C1-W-C18	131.4(3)	Co1-C5-O5	179.5(7)
C1-W-C19	96.5(3)	Co2-C6-O6	178.2(7)
C2-W-C9	80.2(3)	Co2-C7-O7	177.6(8)
C2-W-C15	113.6(3)	Co2-C8-O8	178.8(7)
C2-W-C16	141.2(3)	W-C9-Co1	82.90(22)
C2-W-C17	122.0(3)	W-C9-Co2	82.16(22)
C2-W-C18	89.9(3)	W-C9-C10	137.5(4)
C2-W-C19	84.3(3)	Co1-C9-Co2	78.85(23)
C9-W-C15	155.2(3)	Co1-C9-C10	130.6(5)
C9-W-C16	138.4(3)	Co2-C9-C10	124.4(5)
C9-W-C17	135.17(25)	C9-C10-C11	139.0(7)
C9-W-C18	146.5(3)	C9-C10-O9	113.9(6)

C9-W-C19	164.3(3)	C11-C10-O9	107.1(6)
C15-W-C16	34.2(3)	C10-C11-C12	112.1(8)
C15-W-C17	56.3(3)	C10-C11-C14	125.7(8)
C15-W-C18	57.3(3)	C12-C11-C14	122.2(8)
C15-W-C19	34.8(4)	C11-C12-C13	104.0(7)
C16-W-C17	33.5(3)	C12-C13-O9	110.9(8)
C16-W-C18	56.4(3)	W-C15-C16	74.3(4)
C16-W-C19	57.3(3)	W-C15-C19	71.6(4)
C17-W-C18	33.5(3)	C16-C15-C19	107.9(7)
C17-W-C19	56.7(3)	W-C16-C15	71.4(4)
C18-W-C19	34.9(3)	W-C16-C17	73.1(4)
W-Co1-Co2	62.47(3)	C15-C16-C17	107.5(7)
W-Co1-C3	96.66(23)	W-C17-C16	73.4(4)
W-Co1-C4	98.99(22)	W-C17-C18	72.3(4)
W-Co1-C5	147.62(22)	C16-C17-C18	109.7(7)
W-Co1-C9	51.28(17)	W-C18-C17	74.2(4)
Co2-Co1-C3	156.52(23)	W-C18-C19	71.3(4)
Co2-Co1-C4	97.48(24)	C17-C18-C19	107.7(7)
Co2-Co1-C5	93.47(24)	W-C19-C15	73.5(4)
Co2-Co1-C9	51.20(18)	W-C19-C18	73.9(4)
C3-Co1-C4	96.3(3)	C15-C19-C18	107.2(7)
C3-Co1-C5	100.9(3)	C10-O9-C13	106.0(7)
C3-Co1-C9	108.1(3)		

Table . Atomic Parameters x,y,z and Beq.
E.S.Ds. refer to the last digit printed.

	x	y	z	Beq
W	0.23478(3)	0.145288(16)	0.140495(15)	2.542(9)
Co1	0.22505(10)	0.28179 (5)	0.24679 (5)	2.85 (3)
Co2	-0.00272(10)	0.25587 (6)	0.13989 (6)	3.07 (3)
C1	0.3297 (7)	0.0802 (4)	0.2424 (4)	3.4 (3)
C2	0.0902 (8)	0.0446 (4)	0.1329 (4)	3.7 (3)
C3	0.3936 (8)	0.2533 (5)	0.3196 (5)	4.1 (3)
C4	0.3197 (9)	0.3607 (4)	0.1844 (5)	4.0 (3)
C5	0.1171 (9)	0.3430 (4)	0.3180 (5)	3.8 (3)
C6	0.0371 (9)	0.3448 (4)	0.0684 (5)	4.2 (3)
C7	-0.1588 (9)	0.2974 (5)	0.1960 (5)	4.6 (3)
C8	-0.1058 (8)	0.1861 (5)	0.0633 (5)	4.1 (3)
C9	0.0901 (7)	0.1818 (4)	0.2351 (4)	2.65 (23)
C10	0.0029 (8)	0.1388 (4)	0.2988 (5)	3.8 (3)
C11	0.0177 (10)	0.1199 (5)	0.3824 (5)	4.7 (4)
C12	-0.1093 (10)	0.0760 (5)	0.4068 (6)	5.3 (4)
C13	-0.2111 (11)	0.0690 (7)	0.3271 (9)	8.1 (6)
C14	0.1466 (12)	0.1385 (6)	0.4413 (7)	6.9 (5)
C15	0.4694 (9)	0.1156 (7)	0.0881 (5)	6.4 (5)
C16	0.4441 (9)	0.2029 (6)	0.0740 (5)	5.2 (4)
C17	0.3170 (9)	0.2110 (5)	0.0159 (5)	4.6 (3)
C18	0.2612 (9)	0.1316 (5)	-0.0072 (5)	4.7 (4)
C19	0.3551 (11)	0.0706 (5)	0.0381 (6)	5.6 (4)
O1	0.3863 (6)	0.0392 (3)	0.2992 (3)	5.11 (25)
O2	0.0127 (7)	-0.0153 (3)	0.1282 (4)	5.9 (3)
O3	0.4993 (6)	0.2376 (4)	0.3640 (4)	6.2 (3)
O4	0.3828 (7)	0.4090 (4)	0.1448 (4)	6.4 (3)
O5	0.0473 (7)	0.3819 (4)	0.3629 (4)	6.0 (3)
O6	0.0577 (7)	0.3999 (4)	0.0236 (4)	6.5 (3)
O7	-0.2586 (7)	0.3206 (5)	0.2306 (5)	7.6 (4)
O8	-0.1746 (7)	0.1416 (4)	0.0144 (4)	6.1 (3)
O9	-0.1494 (7)	0.1058 (4)	0.2621 (4)	6.4 (3)
H12	-0.128	0.056	0.464	6.0
H13	-0.313	0.040	0.324	8.0
H14a	0.225	0.169	0.414	7.4
H14b	0.118	0.174	0.489	7.4
H14c	0.192	0.086	0.466	7.4
H15	0.551	0.088	0.127	6.6
H16	0.507	0.250	0.101	5.9
H17	0.274	0.267	-0.005	5.3
H18	0.172	0.119	-0.049	5.6
H19	0.341	0.008	0.034	6.1

Beq is the Mean of the Principal Axes of the Thermal Ellipsoid

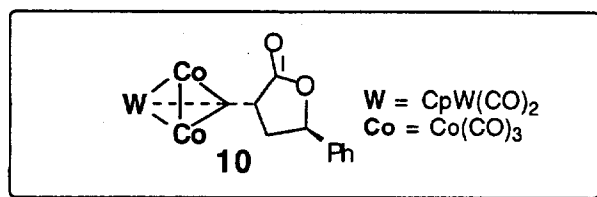
Table of u(i,j) or U values *100.
E.S.Ds. refer to the last digit printed

	u11(U)	u22	u33	u12	u13	u23
W	3.382(12)	3.106(12)	3.236(12)	-0.046(10)	0.662(10)	-0.291(11)
Co1	4.03 (5)	3.42 (5)	3.38 (4)	-0.54 (4)	0.37 (4)	-0.48 (4)
Co2	3.70 (5)	4.01 (5)	3.88 (5)	0.34 (4)	0.03 (4)	-0.17 (4)
C1	4.0 (4)	4.2 (4)	4.6 (4)	0.0 (3)	0.4 (3)	-0.2 (3)
C2	5.0 (4)	4.2 (4)	5.0 (4)	0.7 (3)	0.8 (3)	-0.4 (3)
C3	5.7 (4)	5.4 (4)	4.6 (4)	-1.1 (4)	0.3 (3)	-0.8 (3)
C4	5.7 (4)	4.3 (4)	5.1 (4)	-0.3 (3)	0.1 (3)	-0.8 (3)
C5	6.0 (4)	3.9 (4)	4.7 (4)	-1.0 (3)	0.4 (3)	-1.1 (3)
C6	6.1 (5)	4.6 (4)	5.1 (4)	0.3 (3)	-0.1 (4)	0.0 (3)
C7	5.4 (4)	6.2 (5)	5.7 (5)	1.0 (4)	-0.5 (4)	-0.4 (4)
C8	4.6 (4)	5.6 (4)	5.1 (4)	0.5 (4)	-0.3 (3)	0.1 (4)
C9	3.6 (3)	3.1 (3)	3.4 (3)	-0.3 (3)	0.6 (3)	-0.6 (3)
C10	5.1 (4)	3.9 (4)	5.8 (4)	0.3 (3)	2.0 (3)	0.1 (3)
C11	7.1 (5)	4.9 (4)	6.3 (5)	1.4 (4)	2.1 (4)	0.1 (4)
C12	7.7 (6)	5.5 (5)	7.6 (6)	0.7 (4)	3.8 (5)	1.4 (4)
C13	7.3 (6)	7.9 (7)	16.6 (11)	-1.2 (5)	5.7 (7)	2.9 (7)
C14	7.6 (6)	7.5 (7)	10.9 (8)	1.5 (5)	0.6 (6)	-2.6 (6)
C15	5.5 (5)	13.9 (8)	5.2 (5)	4.5 (5)	2.8 (4)	2.7 (5)
C16	5.3 (4)	7.8 (6)	7.0 (5)	-2.1 (4)	3.0 (4)	-1.9 (4)
C17	6.5 (5)	5.1 (4)	6.2 (5)	0.3 (4)	2.4 (4)	1.1 (4)
C18	6.3 (5)	8.0 (6)	3.9 (4)	-1.7 (4)	1.6 (4)	-0.9 (4)
C19	10.2 (7)	4.5 (4)	7.6 (6)	0.7 (4)	5.4 (5)	-0.2 (4)
O1	7.2 (4)	6.1 (3)	6.0 (3)	0.9 (3)	0.2 (3)	1.8 (3)
O2	7.5 (4)	4.6 (3)	10.4 (5)	-2.4 (3)	1.2 (3)	-1.5 (3)
O3	5.8 (3)	10.0 (5)	7.2 (4)	-0.2 (3)	-2.5 (3)	0.0 (3)
O4	8.8 (4)	6.5 (4)	9.2 (4)	-2.9 (3)	1.8 (4)	2.3 (3)
O5	8.2 (4)	7.6 (4)	7.3 (4)	0.3 (3)	2.5 (3)	-3.5 (3)
O6	9.8 (5)	6.6 (4)	8.4 (4)	-0.9 (4)	0.5 (4)	2.4 (3)
O7	6.5 (4)	11.1 (5)	11.6 (6)	2.4 (4)	2.7 (4)	-2.1 (5)
O8	6.8 (4)	8.1 (4)	7.5 (4)	-0.9 (3)	-2.5 (3)	-2.0 (3)
O9	7.1 (4)	8.1 (4)	9.2 (5)	-0.8 (3)	1.1 (3)	0.7 (4)
H12	7.7					
H13	10.1					
H14a	9.3					
H14b	9.3					
H14c	9.3					
H15	8.3					
H16	7.4					
H17	6.7					
H18	7.1					
H19	7.7					

Anisotropic Temperature Factors are of the form

$$\text{Temp} = -2\pi^2(h^2u_{11}^* + k^2u_{22}^* + l^2u_{33}^* + 2hku_{12}^* + 2hlu_{13}^* + 2kl u_{23}^*)$$

Tables of crystal data, bond distances and angles and ORTEP drawings of Compound 10



Summary of Crystal Data and Intensity Collection 98sp10 (980171)

Empirical Formula	C ₂₄ H ₁₂ Co ₂ O ₁₀ W
Color; Habit	black ; bladed
Crystal Size (mm)	0.25 x 0.125 x 0.025
Space Group	P2 ₁ /n; Monoclinic
Unit Cell Dimensions	$a = 9.0774(2) \text{ \AA}$ $b = 10.1899(2) \text{ \AA}; \beta = 93.307(1)^\circ$ $c = 26.6114(4) \text{ \AA}$
Volume	2457.4(7) $\text{\AA}^3$
Z	4
Formula Weight	762.0
Density(calc.)	2.060 Mg/m ³
Absorption Coefficient	6.060 mm ⁻¹
F(000)	1456
Diffractionmeter Used	Siemens Smart CCD
Radiation	MoK α ($\lambda = 0.71073 \text{ \AA}$)
Temperature (K)	296
2 θ Range	2.5 to 56.0°
Scan Speed	20.00 seconds/ frame
Scan Range (ω)	0.30°/ frame
Index Ranges	$-11 \leq h \leq 11, -13 \leq k \leq 13, -34 \leq l \leq 26$
Reflections Collected	14343(8677 $\geq$ 3.0 σ (1))
Independent Reflections	5324(2074 $\geq$ 3.0 σ (1))(R _{int} = 5.83%)
System Used	Siemens SHELXTL PLUS (VMS)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0014F^2$
Number of Parameters Refined	334
Final R Indices (obs. data)	R = 0.0448, R _w = 0.0465
Goodness-of-Fit	1.00
Largest and Mean Δ/σ	0.002, 0.000
Data-to-Parameter Ratio	6.2:1
Largest Difference Peak/Hole	1.47/-0.97 e $\text{\AA}^{-3}$

A crystal of dimensions 0.25 x 0.125 x 0.025 mm for $C_{24}H_{12}Co_2O_{10}W$ was selected for indexing and intensity data collection on a Siemens Smart-CCD diffractometer equipped with a normal focus, 3 KW sealed tube X-ray source. Intensity data were collected in 1271 frames with increasing ω (width of 0.3 deg per frame). Unit cell dimensions were determined by a least-squares fit of 4402 reflections with $5 < 2\theta < 50$ deg. Absorption correction was based on 6355 symmetry-equivalent reflections using the SHELXTL-PC program package ($T_{min,max} = 0.341740, 0.914526$). On the basis of systematic absences, statistics of intensity distribution, and successful solution and refinement of the structure, the space group was determined to be $P2_1/n$ (No. 14).

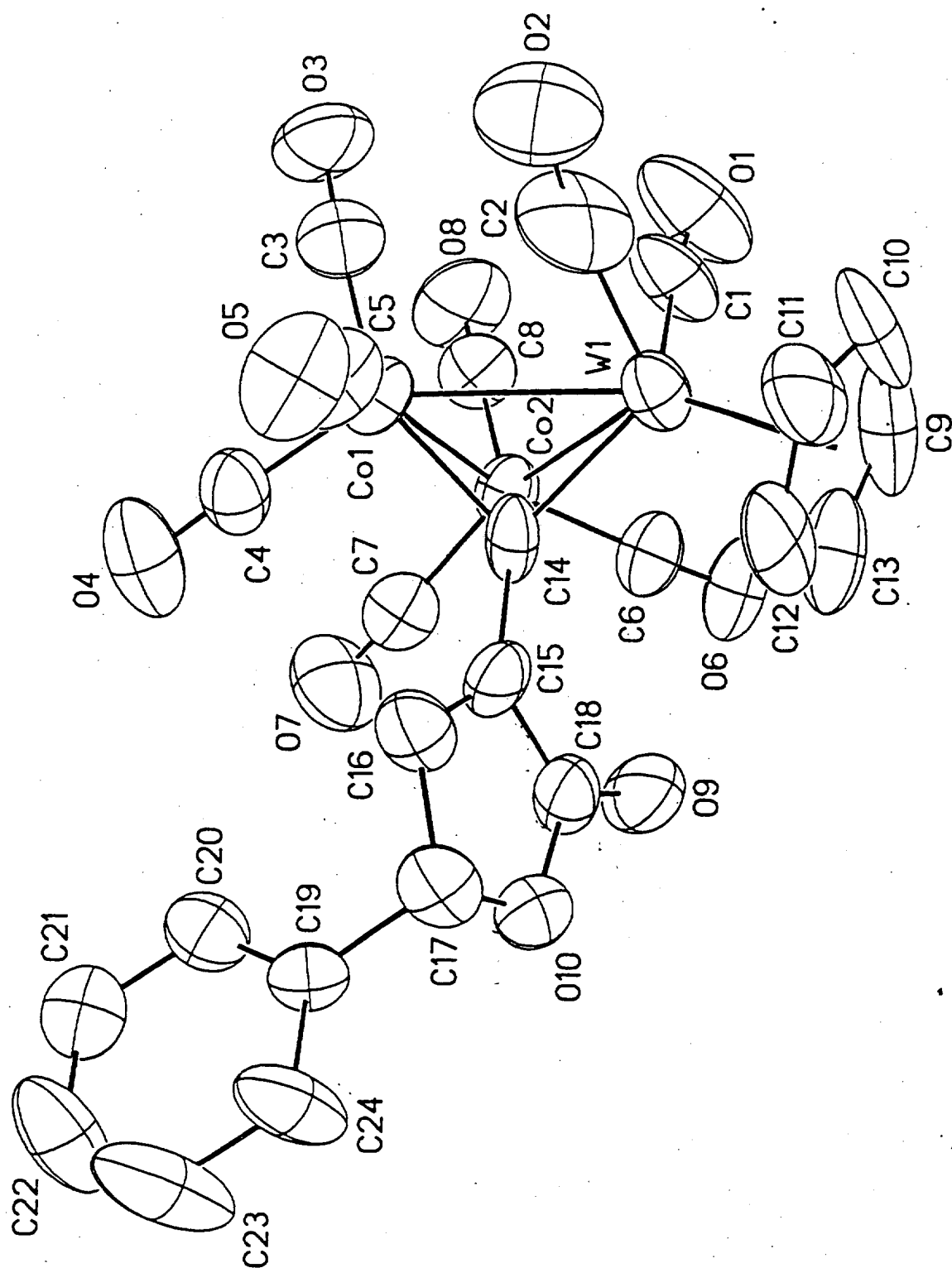


Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	x	y	z	U(eq)
W(1)	6491(1)	2475(1)	1071(1)	54(1)
Co(1)	3950(2)	3500(2)	1419(1)	54(1)
Co(2)	4018(2)	1075(2)	1263(1)	50(1)
O(1)	5252(16)	1628(17)	24(5)	130(7)
O(2)	6004(16)	5360(14)	651(6)	133(8)
O(3)	2265(15)	4233(13)	477(5)	100(6)
O(4)	1681(13)	3263(14)	2145(5)	104(6)
O(5)	5128(14)	5856(13)	1899(5)	102(6)
O(6)	5751(11)	-1321(10)	1104(4)	82(5)
O(7)	2125(14)	195(13)	2040(5)	111(6)
O(8)	2009(14)	868(14)	356(5)	105(6)
O(9)	6314(13)	-391(11)	2219(4)	87(5)
O(10)	6437(10)	734(10)	2929(4)	66(4)
C(1)	5610(19)	1916(19)	426(6)	77(7)
C(2)	6120(20)	4270(22)	820(8)	104(9)
C(3)	2895(18)	3935(15)	844(7)	67(7)
C(4)	2550(17)	3380(16)	1848(6)	67(6)
C(5)	4693(19)	4949(17)	1685(7)	82(8)
C(6)	5104(16)	-400(16)	1182(6)	64(6)
C(7)	2892(17)	561(15)	1754(6)	67(7)
C(8)	2779(19)	974(16)	703(7)	70(7)
C(9)	8534(20)	1347(31)	844(10)	125(12)
C(10)	8847(20)	2617(22)	797(8)	99(9)
C(11)	8860(17)	3242(20)	1251(9)	87(8)
C(12)	8619(15)	2380(25)	1623(7)	86(8)
C(13)	8456(17)	1196(23)	1363(12)	105(11)
C(14)	5299(14)	2159(13)	1685(5)	52(5)
C(15)	5721(14)	1955(15)	2224(6)	57(6)
C(16)	5771(15)	2776(15)	2620(6)	63(6)
C(17)	6301(16)	2122(15)	3088(6)	58(6)
C(18)	6162(16)	657(15)	2440(6)	59(6)
C(19)	5286(15)	2200(12)	3518(5)	47(5)
C(20)	3747(16)	2130(14)	3427(6)	64(7)
C(21)	2869(18)	2230(13)	3839(8)	69(7)
C(22)	3453(22)	2387(17)	4310(7)	96(9)
C(23)	4923(24)	2435(21)	4392(7)	134(12)
C(24)	5856(18)	2367(15)	4000(6)	82(7)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2. Bond lengths (Å)

W(1)-Co(1)	2.742 (2)	W(1)-Co(2)	2.734 (2)
W(1)-C(1)	1.937 (17)	W(1)-C(2)	1.970 (22)
W(1)-C(9)	2.292 (24)	W(1)-C(10)	2.303 (19)
W(1)-C(11)	2.312 (16)	W(1)-C(12)	2.360 (16)
W(1)-C(13)	2.307 (20)	W(1)-C(14)	2.037 (14)
Co(1)-Co(2)	2.507 (3)	Co(1)-C(3)	1.811 (17)
Co(1)-C(4)	1.760 (16)	Co(1)-C(5)	1.756 (17)
Co(1)-C(14)	1.941 (13)	Co(2)-C(6)	1.818 (16)
Co(2)-C(7)	1.782 (17)	Co(2)-C(8)	1.818 (18)
Co(2)-C(14)	1.918 (13)	O(1)-C(1)	1.137 (21)
O(2)-C(2)	1.201 (26)	O(3)-C(3)	1.145 (21)
O(4)-C(4)	1.156 (20)	O(5)-C(5)	1.142 (22)
O(6)-C(6)	1.132 (19)	O(7)-C(7)	1.125 (22)
O(8)-C(8)	1.132 (21)	O(9)-C(18)	1.230 (19)
O(10)-C(17)	1.484 (18)	O(10)-C(18)	1.314 (18)
C(9)-C(10)	1.332 (38)	C(9)-C(13)	1.395 (41)
C(10)-C(11)	1.365 (33)	C(11)-C(12)	1.351 (32)
C(12)-C(13)	1.396 (35)	C(14)-C(15)	1.479 (22)
C(15)-C(16)	1.343 (22)	C(15)-C(18)	1.488 (22)
C(16)-C(17)	1.471 (21)	C(17)-C(19)	1.511 (20)
C(19)-C(20)	1.406 (20)	C(19)-C(24)	1.364 (20)
C(20)-C(21)	1.397 (25)	C(21)-C(22)	1.342 (27)
C(22)-C(23)	1.341 (30)	C(23)-C(24)	1.384 (26)

Table 3. Bond angles ($^{\circ}$)

Co(1)-W(1)-Co(2)	54.5(1)	Co(1)-W(1)-C(1)	95.7(5)
Co(2)-W(1)-C(1)	73.2(5)	Co(1)-W(1)-C(2)	68.3(6)
Co(2)-W(1)-C(2)	115.0(5)	C(1)-W(1)-C(2)	85.4(8)
Co(1)-W(1)-C(9)	171.6(7)	Co(2)-W(1)-C(9)	118.3(7)
C(1)-W(1)-C(9)	85.3(8)	C(2)-W(1)-C(9)	120.1(9)
Co(1)-W(1)-C(10)	153.8(6)	Co(2)-W(1)-C(10)	151.6(6)
C(1)-W(1)-C(10)	94.7(7)	C(2)-W(1)-C(10)	88.8(8)
C(9)-W(1)-C(10)	33.7(9)	Co(1)-W(1)-C(11)	126.3(5)
Co(2)-W(1)-C(11)	154.4(6)	C(1)-W(1)-C(11)	127.9(8)
C(2)-W(1)-C(11)	84.0(7)	C(9)-W(1)-C(11)	57.8(8)
C(10)-W(1)-C(11)	34.4(8)	Co(1)-W(1)-C(12)	118.6(5)
Co(2)-W(1)-C(12)	120.9(5)	C(1)-W(1)-C(12)	145.2(7)
C(2)-W(1)-C(12)	111.5(8)	C(9)-W(1)-C(12)	59.9(8)
C(10)-W(1)-C(12)	57.2(7)	C(11)-W(1)-C(12)	33.6(8)
Co(1)-W(1)-C(13)	138.6(7)	Co(2)-W(1)-C(13)	105.6(5)
C(1)-W(1)-C(13)	113.8(9)	C(2)-W(1)-C(13)	138.8(7)
C(9)-W(1)-C(13)	35.3(10)	C(10)-W(1)-C(13)	55.1(8)
C(11)-W(1)-C(13)	55.2(7)	C(12)-W(1)-C(13)	34.8(9)
Co(1)-W(1)-C(14)	45.0(4)	Co(2)-W(1)-C(14)	44.5(4)
C(1)-W(1)-C(14)	117.0(6)	C(2)-W(1)-C(14)	109.3(7)
C(9)-W(1)-C(14)	127.4(8)	C(10)-W(1)-C(14)	144.1(7)
C(11)-W(1)-C(14)	114.7(7)	C(12)-W(1)-C(14)	87.0(6)
C(13)-W(1)-C(14)	94.4(8)	W(1)-Co(1)-Co(2)	62.6(1)
W(1)-Co(1)-C(3)	102.8(5)	Co(2)-Co(1)-C(3)	96.8(5)
W(1)-Co(1)-C(4)	147.7(5)	Co(2)-Co(1)-C(4)	93.7(6)
C(3)-Co(1)-C(4)	101.4(7)	W(1)-Co(1)-C(5)	98.4(6)
Co(2)-Co(1)-C(5)	152.0(6)	C(3)-Co(1)-C(5)	107.9(8)
C(4)-Co(1)-C(5)	94.1(8)	W(1)-Co(1)-C(14)	47.9(4)
Co(2)-Co(1)-C(14)	49.1(4)	C(3)-Co(1)-C(14)	140.5(7)
C(4)-Co(1)-C(14)	100.3(7)	C(5)-Co(1)-C(14)	103.0(7)
W(1)-Co(2)-Co(1)	62.9(1)	W(1)-Co(2)-C(6)	87.3(5)
Co(1)-Co(2)-C(6)	148.6(5)	W(1)-Co(2)-C(7)	143.4(5)
Co(1)-Co(2)-C(7)	98.5(5)	C(6)-Co(2)-C(7)	100.5(7)
W(1)-Co(2)-C(8)	110.6(6)	Co(1)-Co(2)-C(8)	99.9(5)
C(6)-Co(2)-C(8)	99.8(7)	C(7)-Co(2)-C(8)	103.3(8)
W(1)-Co(2)-C(14)	48.1(4)	Co(1)-Co(2)-C(14)	49.9(4)
C(6)-Co(2)-C(14)	103.3(6)	C(7)-Co(2)-C(14)	95.5(7)
C(8)-Co(2)-C(14)	146.9(7)	C(17)-O(10)-C(18)	109.1(11)
W(1)-C(1)-O(1)	171.7(16)	W(1)-C(2)-O(2)	174.6(16)
Co(1)-C(3)-O(3)	177.8(16)	Co(1)-C(4)-O(4)	176.4(14)
Co(1)-C(5)-O(5)	173.8(16)	Co(2)-C(6)-O(6)	176.1(13)
Co(2)-C(7)-O(7)	175.4(14)	Co(2)-C(8)-O(8)	177.8(16)
W(1)-C(9)-C(10)	73.6(14)	W(1)-C(9)-C(13)	72.9(12)
C(10)-C(9)-C(13)	102.9(23)	W(1)-C(10)-C(9)	72.7(12)
W(1)-C(10)-C(11)	73.2(11)	C(9)-C(10)-C(11)	111.2(21)
W(1)-C(11)-C(10)	72.4(11)	W(1)-C(11)-C(12)	75.2(10)
C(10)-C(11)-C(12)	110.6(19)	W(1)-C(12)-C(11)	71.3(10)
W(1)-C(12)-C(13)	70.5(11)	C(11)-C(12)-C(13)	102.4(20)
W(1)-C(13)-C(9)	71.7(14)	W(1)-C(13)-C(12)	74.7(11)
C(9)-C(13)-C(12)	112.7(23)	W(1)-C(14)-Co(1)	87.1(6)
W(1)-C(14)-Co(2)	87.4(6)	Co(1)-C(14)-Co(2)	81.0(5)
W(1)-C(14)-C(15)	132.9(9)	Co(1)-C(14)-C(15)	125.1(10)
Co(2)-C(14)-C(15)	127.0(10)	C(14)-C(15)-C(16)	132.0(14)
C(14)-C(15)-C(18)	123.2(13)	C(16)-C(15)-C(18)	104.8(14)
C(15)-C(16)-C(17)	112.1(13)	O(10)-C(17)-C(16)	102.6(11)
O(10)-C(17)-C(19)	109.3(11)	C(16)-C(17)-C(19)	115.7(12)

O(9)-C(18)-O(10)	120.3(14)	O(9)-C(18)-C(15)	128.6(14)
O(10)-C(18)-C(15)	111.1(13)	C(17)-C(19)-C(20)	120.6(13)
C(17)-C(19)-C(24)	120.2(13)	C(20)-C(19)-C(24)	119.2(14)
C(19)-C(20)-C(21)	117.8(14)	C(20)-C(21)-C(22)	122.0(16)
C(21)-C(22)-C(23)	119.5(19)	C(22)-C(23)-C(24)	121.5(17)
C(19)-C(24)-C(23)	120.0(16)		

Table 4. Anisotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
W(1)	46(1)	70(1)	48(1)	-7(1)	6(1)	-8(1)
Co(1)	48(1)	51(1)	63(1)	5(1)	-8(1)	1(1)
Co(2)	42(1)	51(1)	57(1)	2(1)	-2(1)	-4(1)
O(1)	121(12)	214(16)	56(8)	-66(11)	19(8)	-32(10)
O(2)	136(13)	96(11)	169(15)	-21(9)	31(10)	55(10)
O(3)	119(11)	114(10)	64(8)	22(8)	-31(8)	14(8)
O(4)	70(8)	142(11)	103(10)	-18(8)	24(7)	-57(9)
O(5)	84(9)	91(9)	129(12)	-1(8)	-19(8)	-14(9)
O(6)	59(7)	62(8)	126(10)	21(6)	6(6)	-33(7)
O(7)	102(10)	114(11)	122(12)	-32(8)	37(9)	8(9)
O(8)	90(10)	139(12)	80(9)	5(8)	-45(8)	-9(8)
O(9)	112(10)	81(8)	69(8)	41(7)	-1(7)	8(7)
O(10)	65(7)	78(8)	56(7)	15(6)	1(5)	4(6)
C(1)	73(12)	119(14)	39(10)	-29(10)	10(9)	-24(9)
C(2)	82(14)	106(16)	128(17)	-44(13)	29(12)	24(14)
C(3)	74(12)	55(11)	71(12)	-17(9)	-3(10)	-5(9)
C(4)	51(9)	84(12)	65(11)	9(9)	5(8)	-14(10)
C(5)	74(13)	61(12)	107(15)	-10(10)	-24(10)	-25(11)
C(6)	48(10)	71(11)	71(11)	0(8)	-5(8)	0(9)
C(7)	62(11)	65(11)	73(12)	-5(9)	-4(9)	-2(9)
C(8)	66(12)	73(12)	68(12)	-7(9)	-4(9)	-18(10)
C(9)	66(14)	200(29)	111(18)	21(15)	11(12)	-121(20)
C(10)	78(12)	122(18)	104(16)	-38(13)	58(11)	-43(16)
C(11)	58(11)	95(14)	109(16)	-8(11)	19(11)	-10(15)
C(12)	26(7)	155(20)	77(12)	-20(12)	9(7)	1(16)
C(13)	29(10)	90(16)	196(28)	1(10)	-1(13)	5(17)
C(14)	38(8)	68(11)	51(9)	6(6)	2(7)	-31(7)
C(15)	33(8)	65(10)	71(12)	6(7)	-7(7)	-2(9)
C(16)	62(9)	63(11)	64(10)	1(8)	-7(8)	-1(9)
C(17)	47(9)	79(12)	48(10)	-11(7)	-1(7)	-8(8)
C(18)	64(10)	64(11)	47(10)	25(8)	-5(8)	-20(8)
C(19)	53(9)	43(9)	43(8)	-12(6)	-18(7)	0(6)
C(20)	55(10)	74(12)	63(11)	-6(7)	3(9)	-8(7)
C(21)	50(9)	58(12)	99(15)	-13(7)	2(10)	-3(9)
C(22)	92(15)	123(17)	75(13)	-26(12)	26(11)	-45(12)
C(23)	107(16)	251(30)	43(10)	-100(18)	6(11)	1(14)
C(24)	71(11)	126(16)	48(10)	-40(11)	-7(8)	-1(10)

The anisotropic displacement factor exponent takes the form:

$$-2\pi^2(h^2a^2U_{11} + \dots + 2hka*b*U_{12})$$