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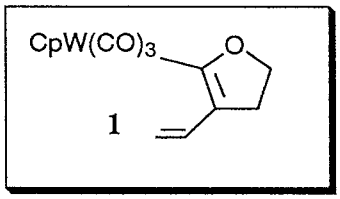
(1) Synthesis of Tungsten-η^1-Diene. Synthesis of Compound 1. To tungsten-η^1-butyn-4-ol (0.47 g, 1.16 mmol) in diethyl ether (25 mL) was added acetaldehyde (0.16 g, 3.66 mmol) and $BF_3.Et_2O$ (0.15 mL, 1.22 mmol) at - 78 ^{0}C, and the solution was stirred for 1 h before the temperature was brought to 23 ^{0}C. The resulting dark orange precipitates were collected by filtration and washed twice with diethyl ether. The precipitates were redissolved in CH_2Cl_2 (20 mL) at 23 ^{0}C, and added with Et_3N (0.24 mL, 1.74 mmol). The solution was stirred for one hour, and evaporated to dryness. Elution the residues through a short alumina column afforded **1** (diethyl ether/hexane = 1/1, R_f= 0.85) as an orange solid (0.44 g, 1.02 mmol, 88%).

Synthesis of Furopyridines. Synthesis of Compound 6. To compound **1** (0.44 g, 1.02 mmol) was dissolved in isobuyyronitrile (2 mL) and $Me_3NO{\cdot}H_2O$ (567 mg, 5.1 mmol), and the mixture was stirred for 8 h at 23 ^{0}C. The solution was concentrated, added with water (2 mL) and CH_2Cl_2 (10 mL). The organic layer was separated, concentrated and chromatographed through a preparative silica TLC to yield **6** (diethyl ether/hexane = 1/1, R_f= 0.40) as a colorless oil (95.5 mg, 0.59 mmol, 58%).

Synthesis of Tungsten-π-azaallyl Compound. Synthesis of Compound 13. compound **12** (506 mg, 0.95 mmol) was dissolved in CH_3CN (2 mL) and the solution was photolyzed at 23 ^{0}C by a Mercury lamp (Rayonet RPR 100, λ = 300 nm) for 70 h . The solution was concentrated, added with water (2 mL) and CH_2Cl_2 (10 mL). The organic layer was separated, concentrated and chromatographed through a cold florisil column (-20 ^{0}C) to yield **13** (yellow solid, 136 mg, 0.25 mmol, 27 %) and **14** (colorless oil, 118 mg, 0.52 mmol, 55 %) respectively.

(2) Spectral Data for Compounds 1-18.

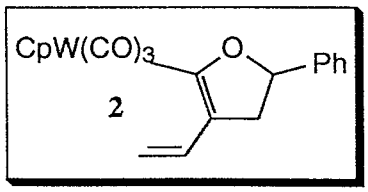
Compound 1



IR(neat, cm^{-1}): υ(CO) 2020 (s), 1916 (vs) ; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.36 (1H, dd, J = 11.2, 16.8 Hz, =CH), 5.54 (5H, s, Cp), 4.73 (1H, dd, J = 11.2, 2.4 Hz

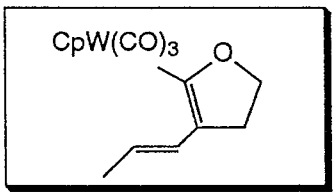
=CH*H'*), 4.65 (1H, dd, *J* = 2.4, 16.8 Hz, =C*H* H'), 4.20 (2H, t, *J* = 5.6 Hz, OCH_2), 2.56 (2H, t, *J* = 5.6 Hz, CH_2), ^{13}C NMR (75 MHz, $CDCl_3$): δ 227.4, 215.6, 151.2, 134.6, 130.3, 106.4, 91.2, 71.0, 30.5 ; MS (EI, 75eV, *m/e*) 428 (M^+). Anal. Calcd. for $C_{14}H_{12}WO_4$: C, 39.28; H, 2.83. Found: C, 39.33; H, 2.90

Compound 2



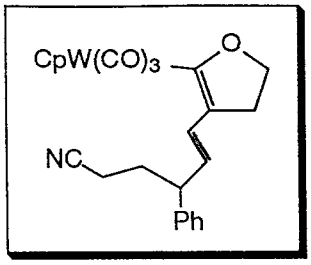
IR(neat, cm^{-1}): υ(CO) 1953 (vs), 2035 (s) ; ^1H NMR (400 MHz, $CDCl_3$): δ 7.34-7.25 (5H, m, Ph), 6.37 (1H, dd, *J* = 17.0, 10.4 Hz, =CH), 5.60 (5H, s, Cp), 5.46 (1H, dd, *J* =7.2, 10.4 Hz, CHO), 4.75 (1H, dd, *J* = 1.6, 10.4, =CH*H'*), 4.65 (1H, dd, *J* = 1.6, 17.0 Hz, =C*H* H'), 3.12 (1H, dd, *J* = 10.4, 14.0 Hz, C*H* H'), 2.59 (1H, dd, *J* = 7.2, 14.0 Hz, CH*H*), ^{13}C NMR (75 MHz, $CDCl_3$): δ 227.5, 215.6, 151.0, 144.4, 134.6, 129.3, 128.3, 127.1, 125.4, 106.7, 91.7, 83.9, 39.4 ; MS (EI, 75eV, *m/e*): 516 (M^+). Anal. Calcd. for $C_{20}H_{16}WO_4$: C, 47.64; H, 3.20. Found: C, 47.81; H, 3.13.

Compound 3



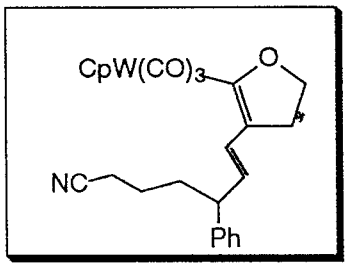
IR(neat, cm^{-1}): υ(CO) 1952 (vs), 2036 (s) ; ^1H NMR (300 MHz, $CDCl_3$): δ 6.03 (1H, d, J =14.2 Hz, =CH), 5.54 (5H, s, Cp), 5.16 (1H, m, =CH), 4.16 (2H, t, *J* = 9.3, OCH_2), 2.55 (2H, t, *J* = 9.3 Hz, CH_2), 1.80 (3H, dd, *J* = 1.5, 5.2 Hz, CH_3), ^{13}C NMR (75 MHz, $CDCl_3$): δ 227.8, 215.7, 145.3,130.2, 128.9,119.1, 91.5, 70.6, 31.5, 18.8 ; MS (EI, 75eV, *m/e*): 442 (M^+). Anal. Calcd. for $C_{15}H_{14}WO_4$: C, 40.72; H, 3.19. Found: C, 40.71; H, 3.13.

Compound 4



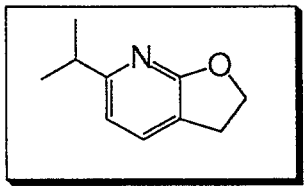
IR(neat, cm^{-1}): υ(CN) 2239(w), υ(CO) 2024(s), 1939(s), υ(C=C) 1668(s). ^1H NMR (300 MHz, $CDCl_3$): δ 7.38–7.23 (5H,.m, Ph), 6.05 (1H, d,.*J* = 15.2 Hz, HC=C), 5.42 (5H, s, Cp), 5.22 (1H, dd, *J* = 7.3, 15.2 Hz, C=CH), 4.23 (2H, m, CH_2O), 3.53 (2H, m, *J* = 7.3 Hz, CHPh), 2.58 (2H, m, CH_2), 2.33 (2H, t, *J* = 6.6 Hz, CH_2CN) 2.08 (2H, m, *J* = 6.6 Hz CH_2CH_2CN). ^{13}C NMR (100 MHz, $CDCl_3$): δ 227.2, 215.7, 150.4, 143.1, 129.7, 129.8, 128.5, 127.5, 126.4, 124.6, 119.6, 91.4, 70.9, 47.8, 31.4, 31.0, 15.3. MS (EI, 75eV, *m/e*): 571 (M^+). Anal. Calcd. for $C_{24}H_{21}WO_4N$: C, 50.46; H, 3.71. Found: C, 50.55; H, 3.88.

Compound 5



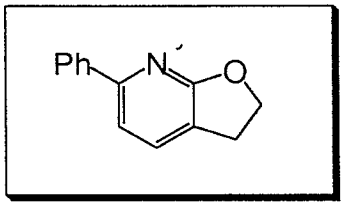
IR(neat, cm^{-1}): υ(CN) 2248(w), υ(CO) 2027, 1938(s), υ(C=C) 1650(w). ^1H NMR (400 MHz, $CDCl_3$): δ 7.34-7.21(5H, m, Ph), 5.92 (1H, d, *J* = 15.2 Hz, HC=C), 5.42 (5H, s, Cp), 5.27(1H, dd, *J* = 15.2 Hz, 6.8 Hz, C=CH), 4.19 (2H, m, CH_2O), 3.38 (1H, m, CHPh), 2.55(2H, m, CH_2CH_2O), 2.31 (2H, t, J = 7.6Hz, CH_2CN), 1.88 (2H, m, CH_2CHPh), 1.64 (2H, m, CH_2CH_2CN). ^{13}C NMR (100 MHz, $CDCl_3$): δ 227.8, 215.8, 149.2, 144.5, 129.8, 128.9, 128.5, 127.8, 126.6, 126.2, 119.7, 91.5, 70.9, 48.3, 35.0, 31.1, 23.4, 16.9. MS (EI, 75eV, *m/e*): 585 (M^+). Anal. Calcd. for $C_{25}H_{23}WO_4N$: C, 51.30; H, 3.93. Found: C, 51.42; H, 3.96.

Compound 6



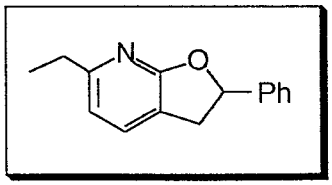
IR(neat, cm^{-1}): υ 2964 (m), 1644 (w), 1604 (s), 1561 (w), 1449 (m), 1420 (s) ; ^1H NMR (300MHz, $CDCl_3$): δ 7.26 (1H, d, J = 7.3 Hz, =CH), 6.53 (1H, d, J = 7.3 Hz, =CH), 4.47 (2H, t, J = 8.5 Hz, OCH_2), 3.08 (2H, t, J = 8.5 Hz, CH_2), 2.82 (1H, m, CH), 1.30 (6H, d, J = 7.0 Hz, CH_3), ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.0, 164.9, 133.5, 116.0, 112.5, 68.7, 35.5, 27.6, 22.3, MS (EI, 75eV, *m/e*) 163.(M^+). HRMS calcd for $C_{10}H_{13}ON$: 163.2212 ; found: 163.2202.

Compound 7



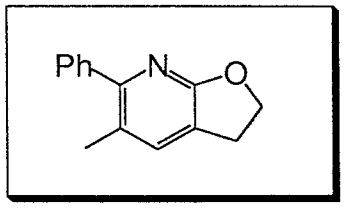
IR(neat, cm^{-1}): υ 3030 (w), 1751 (m), 1607 (m), 1450 (m), 1416 (m), 1217 (m) ; ^1H NMR (400MHz, $CDCl_3$): δ 7.96 (2H, d ,J =7.4 Hz, , Ph), 7.50 (1H, d, J = 7.4 Hz, Ph), 7.42 (1H, t, J = 7.4 Hz, Ph), 7.33 (1H, d, J = 7.4 Hz, Ph), 7.26 (1H, d, J = 7.4 Hz, Ph), 4.65 (2H, t, J = 8.8 Hz, $-CH_2O$), 3.24 (2H, t, J =8.8 Hz, CH_2) ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.7, 155.1, 136.6, 133.9, 128.6, 128.5, 126.7, 118.5, 113.0, 69.0, 27.9; MS (EI, 75eV, *m/e*) 197 (M^+). HRMS calcd for $C_{13}H_{11}ON$: 197.0841; found: 197.0837.

Compound 8



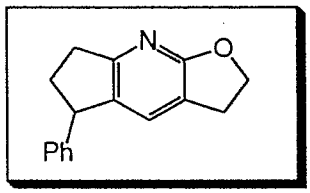
IR (neat, cm^{-1}): υ 2966 (w), 1639 (w), 1605 (m), 1450 (m), 1418 (s), 1219 (m); ^1H NMR (300MHz, $CDCl_3$): δ 7.40-7.28 (6H, m, Ph+HC=), 6.65 (1H, d, *J* = 7.3 Hz, =CH), 5.78 (1H, dd, *J* = 9.6, 7.8 Hz, CHO), 3.59 (1H, dd, *J* = 9.6, 15.7 Hz, CH*H'*), 3.17 (1H, dd, *J* = 7.8, 15.7 Hz, C*H* H'), 2.70 (2H, q, *J* =7.6 Hz, $-CH_2$), 1.26 (3H, t, *J* = 7.6 Hz, CH_3). ^{13}C NMR (75 MHz, $CDCl_3$): δ 167.7, 161.6, 141.5, 133.7, 128.5, 127.9, 125.4, 115.9, 114.5, 81.6, 36.6, 30.8, 13.8. MS (EI, 75eV, *m/e*): 225 (M^+). HRMS: calcd for $C_{15}H_{15}ON$: 225.2929; found: 225.2946.

Compound 9



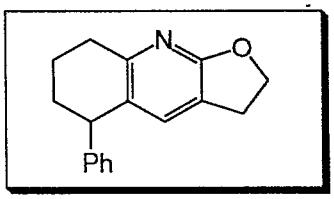
IR(neat, cm^{-1}): υ 3001 (w), 1655 (m), 1605 (m), 1440 (m), 1418 (m), 1233 (m) ; ^1H NMR (400MHz, $CDCl_3$): δ 7.48-7.32 (6H, m, Ph+HC=), 4.60 (2H, t, *J* = 8.4 Hz, CH_2O), 3.23 (2H, t, *J* = 8.4 Hz, CH_2), 2.24 (3H, s, $-CH_3$); ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.7, 154.8, 140.4, 136.4, 129.1, 127.9, 127.7, 122.6, 118.0, 69.0, 27.9, 19.3; MS (EI, 75eV, *m/e*) 211 (M^+). HRMS calcd for $C_{14}H_{13}ON$: 211.2658; found: 211.2650.

Compound 10



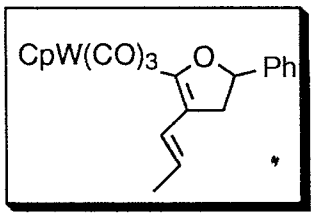
IR(neat, cm^{-1}): υ 3003(w), 1611(w), 1667(w), 1556(m), 1440(m), 1211(m) ; ^1H NMR(300MHz, $CDCl_3$): δ 7.35-7.13 (5H, m, Ph), 7.02 (1H, s, CH), 4.58 (2H, t , *J* = 8.6 Hz, CH_2O), 4.22 (2H, t , *J* = 8.1 Hz, CHPh), 3.09 (2H, t , J = 8.6 Hz, CH_2CH_2O), 2.95 (2H, m, CH_2), 2.58 (1H, m, C*H*H'CHPh), 2.04 (1H, m, CH*H'*CHPh). ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.1, 162.1, 145.4, 132.1, 130.8, 128.5, 127.7, 126.4, 117.2, 69.5, 48.8, 34.6, 27.9. MS (EI, 75eV, *m/e*) 237 (M^+). HRMS: calcd for $C_{16}H_{15}NO$, 237.1154; found, 237.1134.

Compound 11



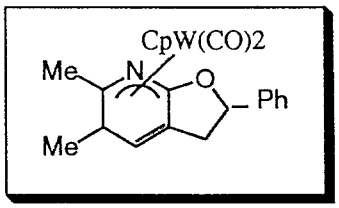
IR(neat, cm^{-1}): υ 2996(w), 1701(w), 1664(m), 1557(w), 1408(w), 1196(w) ; ^1H NMR(300MHz, $CDCl_3$): δ 7.24-7.05 (5H, m, Ph), 6.92 (1H, s, CH), 4.53 (2H, t, J = 8.6 Hz, CH_2O) , 3.99 (1H, t, J = 6.6 Hz, CHPh), 3.06 (2H, t, J = 8.6 Hz, CH_2CH_2O), 2.88 (2H, dd, J = 8.0, 14.8 Hz, CH_2), 2.11-1.71 (4H, m, CH_2CH_2CHPh). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.6, 154.0, 146.7, 135.7, 128.6, 128.4, 126.8, 117.3, 69.1, 44.8, 33.0, 32.4, 27.8, 20.7. MS (EI, 75eV, *m/e*) 251 (M^+). HRMS: calcd for $C_{17}H_{17}NO$, 251.1310; found, 251.1310.

Compound 12



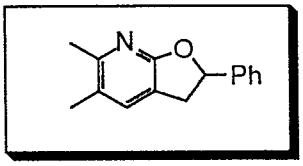
IR(neat, cm^{-1}): υ(CO) 1913 (vs) 2015 (s) ; ^1H NMR (300MHz, $CDCl_3$): δ 7.33-7.23 (5H, m, Ph), 6.06 (1H, d, J =15.0 Hz, =CH), 5.58 (5H, s, Cp), 5.43 (1H, dd, J = 7.2, 10.5, CH-O), 5.17 (1H, m, =CH), 3.10 (1H, dd, J = 10.5, 14.1 Hz, CH*H* '), 2.57 (1H, dd, J = 7.2, 14.1 Hz, C *H* H'), 1.82 (3H, d, J = 6.6 Hz, CH_3) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ 227.9, 215.7, 145.1, 144.8, 127.0, 125.4, 125.3, 119.3, 91.9, 83.4, 40.4, 18.8 ; MS (EI, 75eV, *m/e*) 530 (M^+). Anal. Calcd for $C_{22}H_{18}WO_4$: C, 49.84; H, 3.45. Found: C, 49.88; H, 3.52.

Compound 13



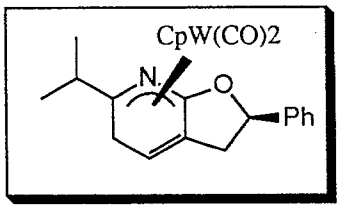
IR(neat, cm^{-1}): υ(CO) 1844 (s), 1922 (s) ^1H NMR (400 MHz, C_6D_6): δ 7.35-7.01 (5H, m, Ph), 5.13 (5H, s, Cp), 4.82 (1H, dd, *J* = 10.4, 6.3 Hz, CH-O), 4.33 (1H, br, =CH), 2.82 (1H, m, C*H* H'), 2.37 (2H, m, CH_3C*H* +CH*H* '), 1.92 (3H, s, CH_3), 1.02 (3H, d, J = 7.5 Hz, CH_3) ; ^{13}C NMR (100 MHz, C_6D_6): δ 226.5, 226.0, 141.8, 134.5, 128.8, 126.7, 125.6, 117.2, 107.7, 96.1, 82.4, 71.1, 38.5, 34.8, 28.8, 19.0 ; MS (EI, 75eV, *m/e*) 531 (M^+). Anal. Calcd for $C_{22}H_{21}O_3WN$: C, 49.74 ; H, 3.98. Found; C, 49.65 , H, 3.92.

Compound 14



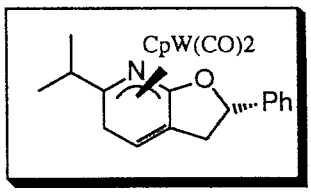
IR(neat, cm^{-1}): υ 3052 (w), 1614 (w), 1433 (m), 1410 (w), 1265(s) ; ^1H NMR (300MHz, $CDCl_3$): δ 7.70-7.19 (6H, m, Ph+HC=), 5.77 (1H, dd, *J* = 9.5, 7.5, OCH), 3.60 (1H, dd, *J* = 9.5, 15.8 Hz, CH*H'*), 3.10 (1H, dd, *J* = 15.8, 7.5 Hz, C*H* H'), 2.39 (3H, s, CH_3), 2.17 (3H, s, CH_3) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.9, 153.7, 141.7, 135.0, 134.9, 128.3, 125.3, 115.9, 81.5, 36.8, 22.1, 18.4 ; MS (EI, 75eV, *m/e*) 225 (M^+). HRMS: calcd for $C_{15}H_{15}ON$: 225.2929; found : 225.2646.

Compound 15. cis-isomer



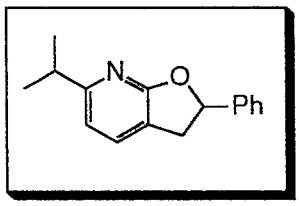
IR(neat, cm^{-1}): υ(CO) 1847 (s), 1922 (s) ; ^1H NMR (300MHz, $CDCl_3$): δ, 7.37-7.24 (5H, m, Ph), 5.69 (5H, s, Cp), 5.52 (1H, dd, *J* = 6.2, 8.2 Hz, CHO), 4.85 (1H, br, =CH), 3.24 (1H, m, CH*H'*), 2.50 (1H, m, C*H* H'), 2.14 (2H, m, CH_2) 1.83 (1H, m, CH), 1.33 (3H, d, *J* = 7.0 Hz, CH_3), 1.18 (3H, d, *J* = 7.0 Hz, CH3) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ 228.5, 225.3, 141.7, 133.4, 128.5, 128.4, 127.8, 125.8, 112.4, 95.8, 80.0, 73.5, 43.3, 37.3, 24.4, 23.8, 19.8 ; MS (EI, 75eV, *m/e*) 545 (M^+). Anal. Calcd for $C_{23}H_{23}O_3NW$: C, 50.66; H, 4.25. Found; C, 50.24; H; 4.44.

Compound 15. trans-isomer



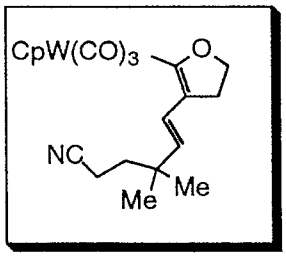
IR(neat, cm^{-1}): υ(CO) 1922 (s), 1844 (s) ; ^1H NMR (300MHz, $CDCl_3$): δ 7.41-7.30 (5H, m, Ph), 5.69 (5H, s, Cp), 5.15 (1H, dd, J = 6.7, 10.1 Hz, CHO), 4.90 (1H, br, =CH), 2.86 (2H, m, CH_2), 2.14 (2H, m, CHC*H* 2), 1.79 (1H, m, CH), 1.33 (3H, d, J = 7.0 Hz, CH_3), 1.17 (3H, d, J = 7.0 Hz, CH_3) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ 226.6, 224.9, 140.8, 134.4, 128.5, 128.0, 126.3, 111.7, 109.0, 95.8, 82.3, 74.2, 43.2, 38.1, 24.2, 23.7, 19.7 ; MS (EI, 75eV, *m/e*) 545 (M^+). Anal.Calcd for $C_{23}H_{23}O_3NW$: C, 50.66; H, 4.25. Found; C, 50.44; H, 4.34.

Compound 16



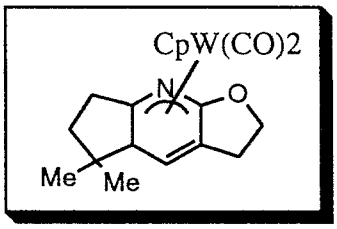
IR(neat, cm^{-1}): υ 2958 (w), 1647 (m), 1600 (m), 1440 (m), 1412 (s), 1233 (w) ; ^1H NMR (300MHz, $CDCl_3$): δ 7.42-7.23 (6H, m, Ph+HC=), 6.67 (1H, d, J = 7.4 Hz, =CH), 5.79 (1H, dd, J = 9.4, 7.8 Hz, CHO), 3.57 (1H, dd, J = 15.4, 9.4 Hz, C*H* H'), 3.18 (1H, dd, J = 15.4, 7.8 Hz CH*H* '), 2.95 (1H, m, CH), 1.27 (6H, J = 7.1 Hz, CH_3) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ 167.6, 165.7, 141.4, 133.7, 128.5, 127.9, 125.5, 116.1, 113.2, 81.7, 36.6, 35.8, 22.6, 22.4 ; MS (EI, 75eV, *m/e*) 239 (M^+). HRMS: calcd for $C_{15}H_{15}ON$: 239.3199; found: 239.3299.

compound **17:**



IR(neat, cm^{-1}): υ(CN) 2246(w), υ(CO) 2024(s), 1940(s); υ(C=C) 1755(w) ^1H NMR(300MHz, $CDCl_3$): δ 5.97 (1H, d, *J* = 15.8Hz, HC=C), 5.54 (5H, s, Cp), 4.94 (1H, d, *J* = 15.8 Hz, C=CH), 4.19 (2H, t, *J* = 9.4 Hz, CH_2O), 2.53 (2H, t, *J* = 9.4Hz, CH_2CH_2O), 2.19 (2H, t, *J* = 7.6 Hz, CH_2CN), 1.66 (2H, t, *J* = 7.6 Hz, CH_2CH_2CN), 1.05 (6H, s, $2CH_3$). ^{13}C NMR (100 MHz, $CDCl_3$): δ 227.5, 215.8, 149.0, 130.3, 129.1, 126.5, 120.7, 91.6, 70.9, 38.6, 35.9, 31.1, 27.3, 12.7. MS (EI, 75eV, *m/e*) 523 (M^+). Anal. Calcd for $C_{20}H_{21}WO_4N$: C, 45.91, H, 4.05. Found: C, 45.80, H, 4.24.

Compound 18



IR(neat, cm^{-1}): υ(CO) 1914(s), 1841(s); υ(C=C) 1679(w) ^1H NMR(300MHz, $CDCl_3$): δ 5.65 (5H, s, Cp), 4.94 (1H, d, *J* = 4.4 Hz, C=CH), 4.30 (1H, m, C*H*H'O), 4.09 (1H, m, CH*H'*O), 2.84 (1H, m, C*H*H'CH_2O), 2.49 (1H, m, CH*H'*CH_2O), 2.47 (1H, m, C*H*H'), 2.12 (1H, m, CH*H'*), 1.78 (1H, d, *J* = 4.4Hz, $CHC(Me)_2$), 1.53 (2H, m, $CH_2C(Me)_2$), 1.06 (3H, s, CH_3), 0.73 (3H, s, CH_3). ^{13}C NMR (100 MHz, $CDCl_3$): δ 226.8, 223.5, 134.2, 112.7, 111.0, 95.2, 68.4, 63.4, 50.9, 39.4, 38.7, 36.5, 28.3, 27.1, 22.2. MS (EI, 75eV, *m/e*) 495 (M^+). Anal. Calcd. for $C_{19}H_{21}WO_3N$: C, 46.08; H, 4.27. Found: C, 46.24; H, 4.32.

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

TITLE	*** 11 ***
Formula	W O3 N C22 H21
Formular wt	531.26
Diffractometer used	Nonius
Space Group	Monoclinic P 21/c
a (angstron)	14.000(3)
b (angstron)	8.1414(14)
c (angstron)	16.945(3)
beta (deg.)	100.492(14)
V (A**3)	1899.1(6)
Z	4
Dcalc. (g.cm**-3)	1.858
lambda (Angstron)	0.7107
F(000)	1028.
Unit cell detn: #;(2theta range)	25;(23.94 - 34.68 deg.)
Scan type	theta/2theta
Scan width (deg.)	2(0.65+0.35tan(theta))
Scan Speed (deg./min)	2.06-8.24
(2Theta)max.	50.0
h k l ranges	(-16; 16)(0; 9)(0; 20)
mu (cm**-1)	62.330
Crystal size (mm)	0.50 X 0.50 X 0.30
Transmission	0.055; 0.172
Temperature (K)	298.
# of meas. reflns.	3335
# of obsed relfns. (I>2.0sig(I))	2853
# of unique reflns.	3335
Rf;Rw	0.030;0.035
GoF	2.10
Refinement program	NRCVAX
# of atoms	48
# of refined params.	245 (2853 out of 3335 reflns.)
Minimize function	Sum(w|Fo-Fc|**2)
Weights scheme	1/[sigma**2(Fo)]
The weight modifier K in KFo**2 is	0.000100
g (2nd. ext. coeff.) x 10E4	0.394(10)
(delta/sigma)max.	0.0083
Residual in final D-map (e/A**3)	-1.380; 2.050

NOTE :

Rf = Sum(Fo-Fc)/Sum(Fo)

Rw = Sqrt[Sum(w(Fo-Fc)**2)/Sum(wFo**2)]

GoF = Sqrt[Sum(w(Fo-Fc)**2)/(No. of reflns - No. of params.)]

3 standard reflections (10,0,0 ; 0,6,0 ; 0,0,14) monitored every 3600 seconds, intensity variation < 2%.

IC5242

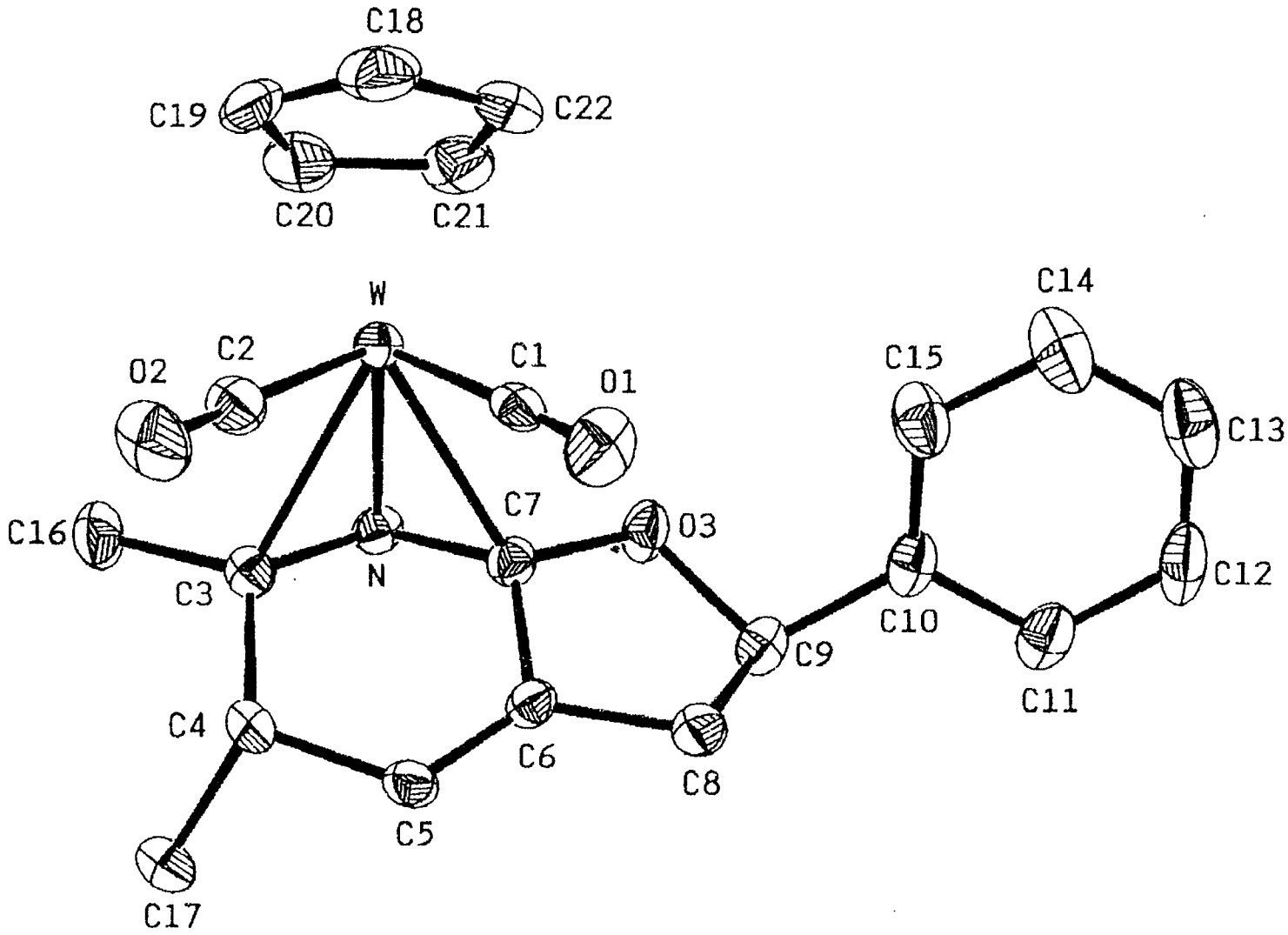

C18
C19
C22
C20
C21
W
C14
C2
C1
C15
O2
O1
C13
C7
C16
O3
N
C12
C3
C10
C9
C11
C4
C6
C8
C5
C17

Table : Bond Distances and Bond Angles of 11

W-N	2.140(5)	C5-C6	1.314(8)
W-C1	1.926(6)	C6-C7	1.462(7)
W-C2	1.940(7)	C6-C8	1.498(8)
W-C3	2.321(6)	C7-O3	1.387(7)
W-C7	2.307(6)	C8-C9	1.541(9)
W-C18	2.317(7)	C9-C10	1.508(9)
W-C19	2.351(6)	C9-O3	1.456(6)
W-C20	2.396(6)	C10-C11	1.388(9)
W-C21	2.375(6)	C10-C15	1.370(10)
W-C22	2.349(7)	C11-C12	1.389(10)
N-C3	1.399(8)	C12-C13	1.345(13)
N-C7	1.374(7)	C13-C14	1.371(12)
C1-O1	1.174(8)	C14-C15	1.378(10)
C2-O2	1.150(8)	C18-C19	1.387(12)
C3-C4	1.540(8)	C18-C22	1.401(11)
C3-C16	1.512(8)	C19-C20	1.396(13)
C4-C5	1.497(8)	C20-C21	1.387(10)
C4-C17	1.532(8)	C21-C22	1.395(12)

N-W-C1	107.66(20)	W-C2-O2	179.0(6)
N-W-C2	107.46(23)	W-C3-N	64.8(3)
N-W-C3	36.27(20)	W-C3-C4	121.6(4)
N-W-C7	35.75(17)	W-C3-C16	112.8(4)
N-W-C18	144.62(24)	N-C3-C4	120.6(5)
N-W-C19	119.3(3)	N-C3-C16	114.5(5)
N-W-C20	88.91(24)	C4-C3-C16	113.9(5)
N-W-C21	88.88(23)	C3-C4-C5	112.4(5)
N-W-C22	119.42(24)	C3-C4-C17	112.4(5)
C1-W-C2	79.3(3)	C5-C4-C17	108.0(5)
C1-W-C3	107.51(21)	C4-C5-C6	121.4(5)
C1-W-C7	73.67(21)	C5-C6-C7	119.8(5)
C1-W-C18	99.7(3)	C5-C6-C8	134.0(5)
C1-W-C19	132.3(3)	C7-C6-C8	106.1(5)
C1-W-C20	150.63(25)	W-C7-N	65.5(3)
C1-W-C21	120.3(3)	W-C7-C6	120.3(4)
C1-W-C22	93.8(3)	W-C7-O3	117.0(4)
C2-W-C3	71.90(25)	N-C7-C6	122.1(5)
C2-W-C7	111.49(23)	N-C7-O3	116.2(4)
C2-W-C18	99.2(3)	C6-C7-O3	109.8(4)
C2-W-C19	93.4(3)	C6-C8-C9	102.5(4)
C2-W-C20	119.5(3)	C8-C9-C10	114.9(5)
C2-W-C21	150.0(3)	C8-C9-O3	105.7(4)
C2-W-C22	132.3(3)	C10-C9-O3	109.9(5)
C3-W-C7	59.25(19)	C9-C10-C11	119.9(6)
C3-W-C18	148.87(25)	C9-C10-C15	121.1(5)
C3-W-C19	114.73(25)	C11-C10-C15	118.9(6)
C3-W-C20	100.18(23)	C10-C11-C12	119.7(7)
C3-W-C21	117.07(25)	C11-C12-C13	120.6(7)
C3-W-C22	151.4(3)	C12-C13-C14	120.1(7)
C7-W-C18	146.25(25)	C13-C14-C15	120.2(8)
C7-W-C19	148.3(3)	C10-C15-C14	120.4(7)
C7-W-C20	114.15(25)	W-C18-C19	74.1(4)
C7-W-C21	96.53(22)	W-C18-C22	73.8(4)
C7-W-C22	111.49(24)	C19-C18-C22	107.5(7)
C18-W-C19	34.6(3)	W-C19-C18	71.4(4)

C18-W-C20	57.3(3)	W-C19-C20	74.7(4)
C18-W-C21	57.5(3)	C18-C19-C20	108.8(7)
C18-W-C22	34.9(3)	W-C20-C19	71.1(4)
C19-W-C20	34.2(3)	W-C20-C21	72.3(4)
C19-W-C21	56.7(3)	C19-C20-C21	107.5(7)
C19-W-C22	57.2(3)	W-C21-C20	73.9(4)
C20-W-C21	33.8(3)	W-C21-C22	71.8(4)
C20-W-C22	56.8(3)	C20-C21-C22	108.5(7)
C21-W-C22	34.3(3)	W-C22-C18	71.3(4)
W-N-C3	78.9(3)	W-C22-C21	73.9(4)
W-N-C7	78.8(3)	C18-C22-C21	107.8(7)
C3-N-C7	111.1(4)	C7-O3-C9	109.6(4)
W-C1-O1	178.6(5)		

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

TITLE	*** 13-cis ***
Formula	C23 H23 N O3 W
Formular wt	545.28
Diffractometer used	Nonius
Space Group	Monoclinic P 21/n
a (angstron)	10.645(3)
b (angstron)	9.658(4)
c (angstron)	19.930(6)
beta (deg.)	93.257(24)
V (A**3)	2045.6(12)
Z	4
Dcalc. (g.cm**-3)	1.771
lambda (Angstron)	0.7107
F(000)	1060.
Unit cell detn: #;(2theta range)	25;(15.34 - 28.90 deg.)
Scan type	theta/2theta
Scan width (deg.)	2(0.65+0.35tan(theta))
Scan Speed (deg./min)	2.06-8.24
(2Theta)max.	50.0
h k l ranges	(-12; 12)(0; 11)(0; 23)
mu (cm**-1)	57.888
Crystal size (mm)	0.20 X 0.25 X 0.50
Transmission	0.539; 1.000
Temperature (K)	298.
# of meas. reflns.	3603
# of obsed relfns. (I>2.0sig(I))	2491
# of unique reflns.	3603
Rf;Rw	0.025;0.028
GoF	1.42
Refinement program	NRCVAX
# of atoms	51
# of refined params.	253 (2491 out of 3603 reflns.)
Minimize function	Sum(w|Fo-Fc|**2)
Weights scheme	1/[sigma**2(Fo)]
The weight modifier K in KFo**2 is	0.000100
(delta/sigma)max.	0.0068
Residual in final D-map (e/A**3)	-1.220; 0.790

NOTE :

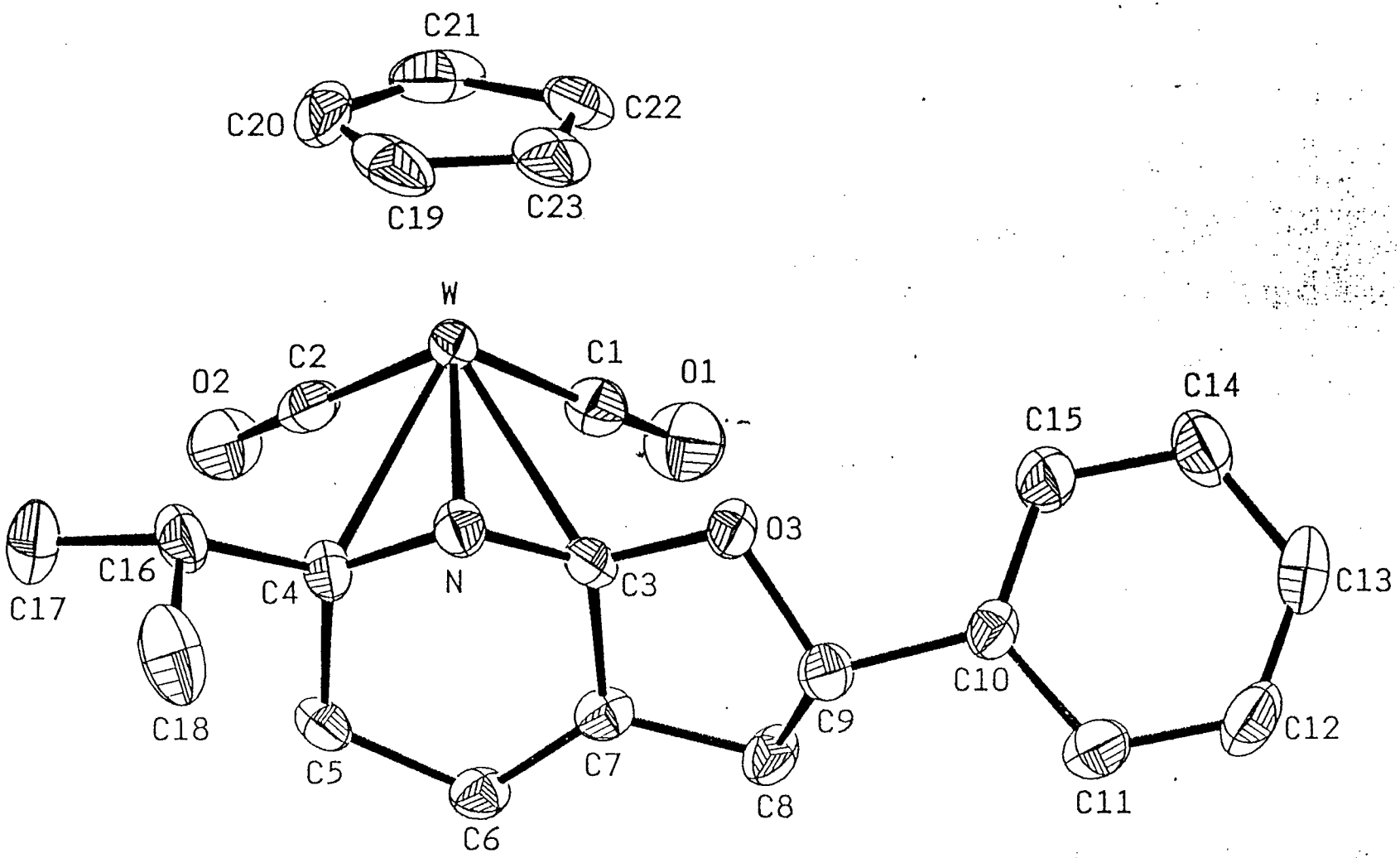
Rf = Sum(Fo-Fc)/Sum(Fo)

Rw = Sqrt[Sum(w(Fo-Fc)**2)/Sum(wFo**2)]

GoF = Sqrt[Sum(w(Fo-Fc)**2)/(No. of reflns - No. of params.)]

3 standard reflections (4,0,0 ; 0,6,0 ; 0,0,14) monitored every 3600 seconds, intensity decay 4%.

IC4635


C21
C20
C22
C19
C23
W
C2
C1
O2
O1
C14
C15
O3
C16
C4
N
C3
C13
C17
C10
C18
C9
C12
C5
C7
C8
C11
C6