JOURNAL OF THE AMERICAN CHEMICAL SOCIETY
J. Am. Chem. Soc., 1997, 119(45), 11114-11115, DOI:10.1021/ja9720927

Experimental procedure

General Information and Materials. Infrared spectra were recorded on a Perkin-Elmer 2000 FT-IR spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on Bruker AM-400 and AMX-300 instruments; chemical shifts are quoted with respect to internal standard tetramethylsilane (^{1}H and ^{13}C NMR). Mass spectra were obtained on a JEOL-HX110 instrument operating in fast atom bombardment (FAB) mode. All reactions were performed under a nitrogen atmosphere using deoxygenated solvents dried with an appropriate reagent. The reactions were monitored by analytical thin-layer chromatography (5735 Kieselgel 60 F_{254}, Merck) and the products were separated on commercially available preparative thin-layer chromatographic plates (Kieselgel 60 F_{254}, Merck). Elemental analyses were carried out at the NSC Regional Instrumentation Center at National Cheng Kung University, Tainan, Taiwan.

Preparation of $[C_5H_3(SiMe_3)_2]W(CO)_3H$:

A toluene solution (70 mL) of $W(CO)_3(NCEt)_3$ (4.94 g, 0.011 mmol) and $C_5H_4(SiMe_3)_2$ (2 g, 0.01 mmol) was heated at 80° C for three hours. The solvent was then removed under vacuum and the residue was transferred into a sublimator and purified by sublimation (70°C, 0.5 mmHg), giving $[C_5H_3(SiMe_3)_2]W(CO)_3H$ (3 g, 60 %) as light yellow solid. The hydride complex $[C_5H_4(SiMe_3)]W(CO)_3H$ was obtained in 14% yield under similar condition.

Selected spectral data for $[C_5H_3(SiMe_3)_2]W(CO)_3H$: IR ($C_6H_{12}$): ν(CO), 2020 (m), 1932 (s, br) cm^{-1}. ^{1}H NMR ($CDCl_3$, 294 K): 5.51 (s, 2H), 5.30 (s, 1H), 0.17 (s, 18H, $SiMe_3$), –7.47 (s, 1H, J_{WH} = 37 Hz).

Selected spectral data for$[C_5H_4(SiMe_3)]W(CO)_3H$: IR (C_6H_{12}): ν(CO), 2022 (m), 1934 (s, br) cm^{-1}. ^{1}H NMR ($CDCl_3$, 294 K): 5.71 (t, 2H, J_{HH} = 1.5 Hz), 5.52 (t, 2H, J_{HH} = 1.5 Hz), 0.17 (s, 9H, $SiMe_3$), –7.28 (s, 1H, J_{WH} = 37 Hz).

Preparation of $[C_5H_4(CHMe_2)]W(CO)_3H$:

A toluene solution (70 mL) of $W(CO)_3(NCEt)_3$ (10 g, 0.023 mmol) and $C_5H_5(CHMe_2)$ (2 g, 0.01 mmol), which was freshly prepared from the dimer $[C_5H_5(CHMe_2)]_2$, was heated at 80° C for three hours. After the solvent was removed under vacuum, the hydride complex $[C_5H_4(CHMe_2)]W(CO)_3H$ (1.78 g, 22 %) was isolated as an oily liquid.

Selected spectral data: IR (C_6H_{12}): ν(CO), 2022 (m), 1933 (s, br) cm^{-1}. ^1H NMR ($CDCl_3$, 294 K): 5.42 (t, 2H, J_{HH} = 2.0 Hz), 5.31 (t, 2H, J_{HH} = 2.4 Hz), 2.63 (m, 1H, J_{HH} = 6.8 Hz), 1.15 (d, 6H, J_{HH} = 6.8 Hz), –7.20 (s, 1H, J_{WH} = 38 Hz).

IR and NMR data for $CpW(CO)_3H$

^1H NMR ($CDCl_3$, 294 K) for $CpW(CO)_3H$: 5.47 (s, 5H), –7.32 (s, 1H, J_{WH} = 37 Hz); IR (isooctane): ν(CO), 2026 (m), 1936 (s, br) cm^{-1}.

Preparation of $[C_5H_3(SiMe_3)_2]WRu_6(\mu_3\text{-}H)(CO)_{18}$

A toluene solution (20 ml) of $[C_5H_3(SiMe_3)_2]W(CO)_3H$ (200 mg, 0.418 mmol) was added dropwise to a refluxing toluene solution (40 ml) of $Ru_3(CO)_{12}$ (134 mg, 0.209 mmol) over a period of three hours through a syringe pump. After the addition of $[C_5H_3(SiMe_3)_2]W(CO)_3H$ was completed, the heating was continued for another 20 minutes. Then the solvent was removed *in vacuo* and the residue was taken up in CH_2Cl_2 and separated by preparative TLC (dichloromethane : hexane = 2 : 3), giving 88 mg of dark-brown $[C_5H_3(SiMe_3)_2]WRu_6(\mu_3\text{-}H)(CO)_{18}$ (**1a**, 0.058 mmol, 28%). Single crystals of **1a** were obtained from a mixture of CH_2Cl_2 and methanol at room temperature. The analogous compounds $[C_5H_4(SiMe_3)]WRu_6(\mu_3\text{-}H)(CO)_{18}$ (**1b**) and $[C_5H_4(CHMe_2)]WRu_6(\mu_3\text{-}H)(CO)_{18}$ (**1c**) were prepared in 26 % and 5 % of yield, respectively.

Selected spectral data for (**1a**): MS (FAB, ^{102}Ru, ^{184}W), m/z 1509 (M^+). IR (C_6H_{12}): ν(CO), 2097 (m), 2073 (vs), 2024 (vs), 2010 (w, br), 2004 (w), 1991 (vw), 1960 (w) cm^{-1}. IR (KBr): $\nu(\mu_4\text{-}CO)$, 1430 (br) cm^{-1}. ^1H NMR ($CDCl_3$, 298 K):

6.11 (t, 1H, J_{HH} = 1.3 Hz), 6.09 (d, 2H, J_{HH} = 1.3 Hz), 0.16 (s,18H, $SiMe_3$), –24.91 (s, 1H). ^{13}C NMR (CD_2Cl_2, 294K): δ 288.1 (μ_4-CO, 3*C*O, J_{WC} = 168 Hz), 207.6 (3*C*O), 200.5 (d, 3*C*O, J_{CH} = 2.4 Hz), 189.6 (d, 3*C*O, J_{CH} = 12 Hz), 184.5 (s, 6*C*O). Anal. Calcd. for $C_{29}H_{22}O_{18}Ru_6Si_2W_1$: C, 23.15; H, 1.47; Found: C, 23.23; H, 1.51.

Selected spectral data for (**1b**): MS (FAB, ^{102}Ru, ^{184}W), m/z 1437 (M^+). IR (C_6H_{12}): ν(CO), 2097 (m), 2074 (vs), 2024 (vs), 2010 (w, br), 1960 (w, br) cm^{-1}. IR (KBr): ν(μ_4-CO), 1427 (br) cm^{-1}. ^1H NMR ($CDCl_3$, 294 K): 6.20 (t, 2H, J_{HH} = 2.1 Hz), 6.05 (t, 2H, J_{HH} = 2.1 Hz), 0.7 (s, 9H, $SiMe_3$), –24.98 (s, 1H). ^{13}C NMR (CD_2Cl_2, 294 K): 287.9 (μ_4-*C*O, J_{WC} = 168 Hz), 207.4 (3*C*O), 200.4 (d, 3*C*O, J_{CH} = 2.3 Hz), 189.3 (d, 3*C*O, J_{CH} = 12 Hz), 184.4 (6*C*O). Anal. Calcd. for $C_{26}H_{14}O_{18}Ru_6Si_1W_1$: C, 21.80; H, 0.98; Found: C, 21.78; H, 0.95.

Selected spectral data for (**1c**): MS (FAB, ^{102}Ru, ^{184}W), m/z 1403 (M^+). IR (C_6H_{12}): ν(CO), 2097 (m), 2073 (vs), 2024 (vs), 2007 (w, br), 2004 (w), 1991 (vw), 1960 (w) cm^{-1}. IR (KBr): ν(μ_4-CO), 1430 (br) cm^{-1}. ^1H NMR ($CDCl_3$, 298 K): 6.08 (t, 2H, J_{HH} = 1.9 Hz), 5.85 (t, 2H, J_{HH} = 1.9 Hz), 2.90 (m, 1H, J_{HH} = 6.8 Hz), 1.15 (d, 6H, J_{HH} = 6.9 Hz), –25.02 (s, 1H). ^{13}C NMR (CD_2Cl_2, 294K): 288.4 (3*C*O, J_{WC} = 168 Hz), 207.6 (3*C*O), 200.5 (d, 3*C*O, J_{CH} = 2.4 Hz), 189.4 (d, 3*C*O, J_{CH} = 12 Hz), 184.4 (s, 6*C*O). Anal. Calcd. for $C_{26}H_{12}O_{18}Ru_6W_1$: C, 22.26; H, 0.86; Found: C, 23.23; H, 1.51.

Crystal data of **1a**: $C_{29}H_{22}O_{18}Si_2Ru_6W_1 \bullet CH_2Cl_2$, M = 1589.86, monoclinic, space group C 2/c, a = 45.846(10), b = 10.737(3), c = 19.343(3) Å, β = 101.55(1)°, V = 9329(4) $Å^3$, Z = 8, ρ_{calcd} = 2.264 gcm^{-3}, $F(000)$ = 5983, λ(Mo-K_α) = 0.7107 Å, T = 298 K, μ = 45.95 cm^{-1}. The intensities were measured on a Nonius CAD4 diffractometer on a crystal with dimensions 0.15 × 0.40 × 0.50 mm. Three standard reflections (26, 0, 0) (0, 8, 0) and (0, 0, 12) were monitored every 3600 sec. with variation of intensity < 1%. Of the 8180 unique reflections collected, 6134 reflections with I > 2 σ(I) were used for the refinement. The structure was solved by using the NRCC-SDP-VAX

package and refined to R_F = 0.035, Rw = 0.034 and G.O.F. = 1.65 for 83 atoms and 537 parameters, weighting scheme $\omega^{-1} = \sigma^2(F_o) + 0.0001\ F_o^2$, and highest Δ/σ ratio 0.03. A difference map following convergence showed residual electron density within the range –0.8/0.9 e/Å^3 (min./max.).

Hydrogenation of [$C_5H_3(SiMe_3)_2$]$WRu_6(\mu_3$-H)$(CO)_{18}$:

A toluene solution (50ml) of [$C_5H_3(SiMe_3)_2$]$WRu_6(\mu_3$-H)$(CO)_{18}$ (100 mg, 0.058 mmol) was refluxed under 1 atm of hydrogen atmosphere for 5 h. After evaporating the solvent under vacuum, the residue was separated by preparative TLC (hexane/dichloromethane, 1 : 5), giving 70 mg of [$C_5H_3(SiMe_3)_2$]$WRu_6(\mu$-H$)_3(CO)_{17}$ (**2**, 0.058 mmol, 71%).

Selected spectral data for (**2**): MS (FAB, ^{102}Ru, ^{184}W), m/z 1483 (M^+). IR (C_6H_{12}): ν(CO), 2099 (m), 2076 (vs), 2065 (vs), 2028 (s), 2015 (m), 1999 (w), 1993 (vw), 1974 (vw, br), 1962 (w), 1953 (vw) cm^{-1}. IR (KBr): (μ_4-CO), 1390cm^{-1}. ^{1}H NMR ($CDCl_3$, 294 K): 6.54 (s, 1H), 5.23 (s, 1H), 4.85 (s, 1H), 0.29 (s, 18H, $SiMe_3$), –14.14 (s, 1H, J_{WH} = 84.8 Hz), –14.70 (s, 1H), –20.71 (s, 1H). ^{13}C NMR (CD_2Cl_2, 294 K): 292.1 (μ_4-CO, J_{WC} = 157 Hz), 289.2 (μ_4-CO, J_{WC} = 158 Hz), 206.0 (br), 201.6, 199.6, 198.4, 195.7 (d, J_{CH} = 9.5 Hz), 194.1, 193.3, 190.6 (d, J_{CH} = 11 Hz), 188.8 (3 CO), 188.3 (d, J_{CH} = 16 Hz), 183.5 (2CO, br), 182.2. Anal. Calcd. for $C_{28}H_{24}O_{17}Ru_6Si_2W_1$: C, 22.74; H, 1.64; Found; C, 22.75; H, 1.71.

Crystal data of **2**: $C_{27}H_{21}O_{17}Si_2Ru_6W_1$, M = 1463.90, monoclinic, space group C 2/c, a = 26.704(5), b = 20.578(1), c = 20.580(5) Å, β = 132.31(2)°, V = 8363(3) Å^3, Z = 8, ρ_{calcd} = 2.231 gcm^{-3}, F(000) = 5231, λ(Mo-Kα) = 0.7107 Å, T = 298 K, μ = 49.29 cm^{-1}. The intensities were measured on a Nonius CAD4 diffractometer on a crystal with dimensions 0.20 × 0.25 × 0.60 mm. Three standard reflections (14, 0, 0) (0, 14, 0) and (0, 0, 10) were monitored every 3600 sec. with variation of intensity < 2%. Of the 7327 unique reflections collected, 5887 reflections with I > 2 σ(I) were used for the refinement. The structure was solved by using the NRCC-SDP-VAX package and refined to

R_F = 0.023, Rw = 0.025 and G.O.F. = 1.29 for 78 atoms and 500 parameters, weighting scheme $\omega^{-1} = \sigma^2(F_o) + 0.0001\ F_o^2$, and highest Δ/σ ratio 0.003. A difference map following convergence showed residual electron density within the range –0.65/0.6 e/Å^3 (min./max.).

Reaction of [$C_5H_3(SiMe_3)_2$]$WRu_6(CO)_{17}(\mu$-H)$_3$ with CO.

A toluene solution (50 ml) of [$C_5H_3(SiMe_3)_2$]$WRu_6(CO)_{17}(\mu$-H)$_3$ (20mg, 0.014 mmol) was refluxed under 1 atm of CO atmosphere for five hours. After removal of the solvent under vacuum, the residue was separated by preparative TLC (hexane/dichloromethane, 3:2), giving unreacted starting material [$C_5H_3(SiMe_3)_2$]$WRu_6(CO)_{17}(\mu$-H)$_3$ (4.5mg, 0.003 mmol, 21%) as the only isolable product.

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

TITLE	*** IC4658 complex 1a ***
Formula	C30 H24 O18 Si2 Ru6 W Cl2
Formular wt	1589.84
Diffractometer used	Nonius
Space Group	Monoclinic C 2/c
a (angstron)	45.846(10)
b (angstron)	10.737(3)
c (angstron)	19.343(3)
beta (deg.)	101.549(14)
V (A**3)	9329(4)
Z	8
Dcalc. (g.cm**-3)	2.264
lambda (Angstron)	0.7107
F(000)	5912.
Unit cell detn: #;(2theta range)	25;(23.10 - 30.70 deg.)
Scan type	theta/2theta
Scan width (deg.)	2(0.65+0.35tan(theta))
Scan Speed (deg./min)	3.30-8.24
(2Theta)max.	50.0
h k l ranges	(-54; 53)(0; 12)(0; 22)
mu (cm**-1)	45.946
Crystal size (mm)	0.15 X 0.40 X 0.50
Transmission	0.432; 1.000
Temperature (K)	298.
# of meas. reflns.	8180
# of obsed relfns. (I>2.0sig(I))	6134
# of unique reflns.	8180
Rf;Rw	0.035;0.034
GoF	1.65
Refinement program	NRCVAX
# of atoms	83
# of refined params.	537 (6134 out of 8180 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	0.649(20)
(delta/sigma)max.	0.0322
Residual in final D-map (e/A**3)	-0.800; 0.900

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 (26,0,0 ; 0,8,0 ; 0,0,12) monitored every 3600 seconds, intensity variation < 1%.

Table : Bond Distances and Bond Angles of complex **1a**.

W-Ru2	2.9170(8)	Ru5-C15	1.889(8)
W-Ru4	2.9128(9)	Ru6-C1	2.415(8)
W-Ru5	2.9105(9)	Ru6-C16	1.926(9)
W-C1	1.963(8)	Ru6-C17	1.887(9)
W-C2	1.978(7)	Ru6-C18	1.899(8)
W-C3	1.959(8)	Ru6-O1	2.084(5)
W-C19	2.368(8)	Si1-C19	1.877(9)
W-C20	2.326(8)	Si1-C24	1.847(14)
W-C21	2.358(8)	Si1-C25	1.866(12)
W-C22	2.335(8)	Si1-C26	1.833(12)
W-C23	2.324(7)	Si2-C21	1.878(8)
Ru1-Ru2	2.8111(11)	Si2-C27	1.862(12)
Ru1-Ru5	2.8276(9)	Si2-C28	1.848(13)
Ru1-C2	2.407(7)	Si2-C29	1.837(12)
Ru1-C4	1.897(9)	C1-O1	1.267(8)
Ru1-C5	1.891(8)	C2-O2	1.250(9)
Ru1-C6	1.901(9)	C3-O3	1.270(9)
Ru1-O2	2.095(5)	C4-O4	1.129(11)
Ru2-Ru3	2.8201(11)	C5-O5	1.124(10)
Ru2-Ru4	2.8014(9)	C6-O6	1.123(11)
Ru2-Ru5	2.7986(10)	C7-O7	1.151(10)
Ru2-C2	2.329(8)	C8-O8	1.152(10)
Ru2-C3	2.314(7)	C9-O9	1.134(11)
Ru2-C7	1.868(8)	C10-O10	1.119(10)
Ru2-C8	1.882(8)	C11-O11	1.151(10)
Ru3-Ru4	2.8039(10)	C12-O12	1.139(9)
Ru3-C3	2.439(8)	C13-O13	1.129(9)
Ru3-C9	1.912(9)	C14-O14	1.133(11)
Ru3-C10	1.908(9)	C15-O15	1.144(10)
Ru3-C11	1.870(8)	C16-O16	1.118(11)
Ru3-O3	2.098(5)	C17-O17	1.136(10)
Ru4-Ru5	2.8148(11)	C18-O18	1.125(10)
Ru4-Ru6	2.8052(11)	C19-C20	1.431(11)
Ru4-C1	2.255(7)	C19-C23	1.406(13)
Ru4-C3	2.318(7)	C20-C21	1.414(12)
Ru4-C12	1.882(7)	C21-C22	1.427(11)
Ru4-C13	1.903(8)	C22-C23	1.408(13)
Ru5-Ru6	2.8199(9)	C30-Cl1	1.45(5)
Ru5-C1	2.314(7)	C30-Cl2	1.60(3)
Ru5-C2	2.316(7)	Cl1-Cl2	2.744(19)
Ru5-C14	1.873(8)		

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

	x	y	z	Beq
W	0.118181(7)	0.42350(3)	0.095036(15)	2.477(13)
Ru1	0.144130(15)	0.21250(6)	-0.06910 (3)	3.01 (3)
Ru2	0.159136(14)	0.44264(6)	-0.00208 (3)	2.47 (3)
Ru3	0.188646(15)	0.65879(6)	0.05846 (3)	3.05 (3)
Ru4	0.182183(13)	0.45293(6)	0.14331 (3)	2.39 (3)
Ru5	0.160148(14)	0.22701(6)	0.07965 (3)	2.45 (3)
Ru6	0.191374(15)	0.23073(7)	0.22092 (3)	3.02 (3)
Si1	0.06456 (6)	0.1993 (3)	0.16787 (15)	5.76 (15)
Si2	0.05671 (6)	0.6386 (3)	-0.00916 (15)	5.19 (14)
C1	0.14535 (16)	0.3348 (8)	0.1704 (4)	3.0 (3)
C2	0.11869 (17)	0.3198 (8)	0.0108 (4)	3.0 (3)
C3	0.14269 (16)	0.5667 (7)	0.0802 (4)	2.9 (3)
C4	0.13091 (20)	0.0454 (8)	-0.0838 (4)	4.2 (4)
C5	0.13069 (23)	0.2528 (9)	-0.1654 (4)	5.0 (5)
C6	0.18319 (20)	0.1763 (8)	-0.0827 (4)	3.8 (4)
C7	0.13727 (20)	0.5282 (9)	-0.0794 (4)	4.5 (5)
C8	0.19178 (19)	0.4349 (8)	-0.0474 (4)	3.5 (4)
C9	0.18367 (20)	0.7733 (9)	-0.0187 (4)	4.4 (4)
C10	0.20496 (20)	0.7775 (9)	0.1291 (4)	4.5 (4)
C11	0.22645 (19)	0.6152 (8)	0.0440 (4)	3.6 (4)
C12	0.18097 (17)	0.5516 (8)	0.2231 (4)	3.5 (4)
C13	0.22454 (17)	0.4561 (8)	0.1637 (4)	3.2 (4)
C14	0.13919 (19)	0.0824 (8)	0.0935 (4)	4.0 (4)
C15	0.19208 (19)	0.1257 (8)	0.0652 (4)	3.4 (4)
C16	0.18377 (19)	0.0659 (9)	0.2523 (4)	4.3 (4)
C17	0.22851 (19)	0.1888 (8)	0.1993 (4)	3.8 (4)
C18	0.21057 (19)	0.2884 (8)	0.3114 (4)	4.0 (4)
C19	0.07605 (17)	0.3549 (9)	0.1380 (4)	3.7 (4)
C20	0.06664 (17)	0.4032 (8)	0.0682 (4)	3.4 (4)
C21	0.07267 (18)	0.5321 (8)	0.0659 (4)	3.4 (4)
C22	0.08704 (18)	0.5661 (9)	0.1358 (4)	4.1 (4)
C23	0.08863 (17)	0.4583 (9)	0.1777 (4)	4.0 (4)
C24	0.0549 (3)	0.0965 (12)	0.0901 (7)	8.6 (8)
C25	0.0295 (3)	0.2303 (14)	0.1998 (7)	9.6 (9)
C26	0.0925 (3)	0.1253 (13)	0.2367 (7)	9.1 (8)
C27	0.0496 (3)	0.5453 (12)	-0.0919 (5)	8.7 (8)
C28	0.0207 (3)	0.6918 (13)	0.0090 (7)	9.4 (8)
C29	0.0811 (3)	0.7715 (11)	-0.0172 (7)	9.1 (8)
O1	0.14902 (11)	0.2875 (5)	0.23151 (23)	3.6 (3)
O2	0.10391 (12)	0.2646 (5)	-0.04163 (24)	3.56 (25)
O3	0.14551 (12)	0.6841 (5)	0.0771 (3)	3.5 (3)
O4	0.12266 (18)	-0.0536 (6)	-0.0924 (4)	7.2 (4)
O5	0.12140 (17)	0.2732 (8)	-0.2225 (3)	8.1 (5)
O6	0.20553 (14)	0.1513 (7)	-0.0941 (3)	5.6 (3)
O7	0.12395 (17)	0.5793 (7)	-0.1279 (3)	7.7 (4)
O8	0.21213 (15)	0.4272 (6)	-0.0738 (3)	6.0 (4)
O9	0.18123 (17)	0.8409 (7)	-0.0645 (3)	6.9 (4)
O10	0.21425 (16)	0.8462 (7)	0.1713 (3)	6.7 (4)
O11	0.25016 (14)	0.5972 (6)	0.0349 (3)	5.3 (3)
O12	0.17993 (15)	0.6111 (7)	0.2713 (3)	6.8 (4)
O13	0.24960 (12)	0.4603 (6)	0.1791 (3)	5.1 (3)
O14	0.12774 (17)	-0.0085 (6)	0.1014 (4)	6.7 (4)
O15	0.21088 (15)	0.0599 (6)	0.0581 (3)	5.6 (3)

O16	0.17741	(17)	-0.0277	(6)	0.2699	(4)	6.8	(4)
O17	0.25151	(13)	0.1597	(7)	0.1920	(3)	5.6	(4)
O18	0.22273	(16)	0.3230	(7)	0.3643	(3)	6.5	(4)
C30	0.4635	(9)	0.2994	(24)	0.1686	(22)	43.3	(42)
C11	0.45507	(25)	0.2730	(15)	0.2356	(7)	43.4	(16)
C12	0.4677	(3)	0.4330	(10)	0.1348	(6)	36.1	(12)
H	0.1844	(15)	0.366	(6)	0.061	(3)	3.0	(15)
H20	0.057		0.354		0.029		4.1	
H22	0.094		0.647		0.151		4.9	
H23	0.097		0.455		0.227		4.8	
H24a	0.049		0.015		0.101		9.2	
H24b	0.039		0.131		0.055		9.2	
H24c	0.071		0.088		0.067		9.2	
H25a	0.022		0.159		0.219		10.8	
H25b	0.033		0.288		0.238		10.8	
H25c	0.015		0.269		0.166		10.8	
H26a	0.086		0.048		0.251		9.8	
H26b	0.111		0.114		0.222		9.8	
H26c	0.097		0.177		0.277		9.8	
H27a	0.042		0.594		-0.132		9.0	
H27b	0.067		0.510		-0.101		9.0	
H27c	0.036		0.478		-0.090		9.0	
H28a	0.011		0.747		-0.026		10.0	
H28b	0.008		0.624		0.012		10.0	
H28c	0.023		0.735		0.053		10.0	
H29a	0.073		0.824		-0.055		9.4	
H29b	0.085		0.821		0.024		9.4	
H29c	0.100		0.745		-0.025		9.4	
H30a	0.452		0.262		0.127		39.1	
H30b	0.480		0.255		0.158		39.1	

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	2.576(16)	3.889(18)	2.973(14)	0.298(17)	0.621(12)	0.032(15)
Ru1	4.34 (4)	4.07 (4)	2.95 (3)	-0.20 (4)	0.54 (3)	-0.61 (3)
Ru2	3.48 (4)	3.32 (4)	2.59 (3)	-0.05 (3)	0.65 (3)	0.14 (3)
Ru3	3.77 (4)	3.22 (4)	4.62 (4)	-0.20 (3)	0.91 (3)	-0.11 (3)
Ru4	2.80 (3)	3.60 (4)	2.66 (3)	0.23 (3)	0.454(24)	-0.27 (3)
Ru5	3.34 (4)	3.19 (4)	2.82 (3)	0.16 (3)	0.73 (3)	0.18 (3)
Ru6	3.72 (4)	4.92 (4)	2.84 (3)	0.94 (4)	0.64 (3)	0.81 (3)
Si1	4.92 (18)	9.10 (24)	8.25 (20)	-0.48 (18)	2.23 (15)	3.34 (18)
Si2	4.53 (17)	6.09 (19)	8.55 (20)	1.03 (15)	0.03 (14)	2.37 (16)
C1	2.6 (4)	4.8 (5)	4.2 (4)	-0.3 (4)	1.5 (3)	-0.4 (4)
C2	2.8 (4)	4.8 (5)	3.6 (4)	0.1 (4)	0.4 (3)	0.7 (4)
C3	2.9 (4)	4.2 (5)	3.8 (4)	0.5 (4)	-0.1 (3)	-0.1 (4)
C4	5.8 (6)	4.6 (6)	5.7 (5)	-0.3 (5)	1.4 (4)	-0.5 (4)
C5	8.3 (8)	7.0 (7)	3.7 (5)	0.2 (6)	1.1 (5)	-0.5 (5)
C6	7.1 (7)	4.1 (5)	3.7 (4)	0.1 (5)	1.8 (4)	-1.0 (4)
C7	6.0 (6)	6.5 (7)	4.6 (5)	0.5 (5)	1.4 (4)	1.2 (4)
C8	6.1 (6)	3.9 (5)	3.2 (4)	-1.1 (5)	1.0 (4)	-0.8 (4)
C9	5.0 (6)	5.2 (6)	6.3 (6)	0.3 (5)	0.9 (4)	0.1 (5)
C10	5.3 (6)	5.5 (6)	6.2 (6)	-0.6 (5)	1.1 (5)	-0.8 (5)
C11	4.7 (5)	4.1 (5)	5.1 (5)	-0.7 (4)	1.1 (4)	-0.4 (4)
C12	3.4 (5)	5.8 (6)	4.1 (4)	0.1 (5)	0.9 (4)	-0.7 (4)
C13	3.4 (5)	5.4 (6)	3.5 (4)	0.2 (4)	1.0 (3)	-0.8 (4)
C14	4.8 (6)	4.8 (6)	5.4 (5)	-0.9 (5)	0.7 (4)	0.6 (4)
C15	5.2 (6)	4.5 (5)	3.1 (4)	0.3 (4)	0.4 (4)	0.1 (4)
C16	4.5 (5)	6.6 (7)	5.5 (5)	1.5 (5)	1.5 (4)	1.5 (5)
C17	5.6 (6)	5.4 (6)	3.4 (4)	0.5 (5)	0.6 (4)	0.2 (4)
C18	5.0 (6)	6.2 (6)	3.8 (4)	1.0 (5)	0.5 (4)	1.1 (4)
C19	2.8 (5)	7.1 (6)	4.7 (5)	0.6 (5)	1.6 (4)	1.0 (4)
C20	2.8 (4)	5.3 (6)	4.6 (4)	0.5 (4)	0.5 (4)	0.5 (4)
C21	3.3 (5)	4.5 (5)	5.2 (5)	0.6 (4)	0.8 (4)	0.1 (4)
C22	3.2 (5)	6.2 (6)	6.2 (5)	1.3 (5)	1.1 (4)	-1.4 (5)
C23	2.3 (4)	8.6 (7)	4.4 (4)	2.3 (5)	0.8 (4)	-0.1 (5)
C24	8.6 (10)	8.6 (10)	14.8 (12)	-1.3 (8)	0.7 (8)	3.2 (8)
C25	8.8 (10)	16.8 (14)	12.7 (10)	-0.6 (10)	6.5 (8)	3.9 (10)
C26	7.9 (9)	13.4 (12)	13.2 (10)	-1.9 (9)	1.6 (8)	8.1 (9)
C27	12.8 (12)	12.4 (12)	6.2 (7)	-0.5 (10)	-1.9 (7)	1.6 (7)
C28	7.4 (9)	10.7 (11)	17.1 (13)	3.2 (9)	1.0 (8)	4.9 (10)
C29	9.3 (10)	7.1 (9)	16.7 (12)	-0.3 (8)	-0.7 (9)	5.3 (8)
O1	3.8 (3)	7.2 (4)	3.1 (3)	0.4 (3)	1.61 (23)	0.6 (3)
O2	3.9 (3)	5.4 (4)	4.0 (3)	-0.7 (3)	0.31 (24)	-0.8 (3)
O3	4.0 (3)	3.6 (3)	5.6 (3)	0.6 (3)	1.2 (3)	-0.4 (3)
O4	10.4 (6)	5.0 (5)	11.9 (6)	-2.6 (5)	1.8 (5)	-1.1 (4)
O5	8.7 (6)	16.1 (8)	5.4 (4)	-1.6 (6)	-0.4 (4)	2.7 (5)
O6	6.1 (4)	9.2 (5)	6.7 (4)	0.9 (4)	2.7 (3)	-1.8 (4)
O7	10.3 (6)	11.1 (7)	6.9 (4)	2.6 (5)	-0.7 (4)	3.8 (4)
O8	8.1 (5)	8.2 (5)	8.1 (4)	-2.5 (4)	5.7 (4)	-2.4 (4)
O9	9.5 (6)	8.8 (6)	8.0 (5)	1.7 (5)	2.0 (4)	3.9 (4)
O10	10.0 (6)	7.7 (5)	7.4 (4)	-2.3 (5)	1.1 (4)	-3.6 (4)
O11	5.5 (4)	6.9 (5)	8.2 (4)	-0.1 (4)	2.6 (3)	-0.5 (4)
O12	7.6 (5)	12.0 (7)	6.2 (4)	1.5 (5)	1.0 (4)	-4.6 (4)
O13	2.7 (3)	9.4 (5)	7.0 (4)	-0.1 (4)	0.0 (3)	-0.1 (4)
O14	9.3 (6)	5.2 (4)	11.1 (6)	-2.9 (4)	2.4 (5)	0.9 (4)

O15	7.4	(5)	7.8	(5)	6.2	(4)	4.2	(4)	1.5	(3)	-0.3	(4)
O16	9.3	(6)	6.8	(5)	9.7	(5)	0.1	(5)	1.9	(4)	3.3	(4)
O17	4.6	(4)	9.3	(5)	7.4	(4)	1.8	(4)	1.6	(3)	-0.4	(4)
O18	9.2	(6)	10.4	(6)	4.4	(4)	0.5	(5)	-0.1	(3)	-1.1	(4)
C30	58.1	(67)	13.5	(25)	75.2	(70)	-12.4	(35)	-29.5	(54)	5.7	(34)
C11	31.7	(13)	79.7	(28)	58.5	(19)	-21.3	(17)	21.0	(13)	-13.5	(19)
C12	51.8	(19)	36.6	(15)	42.1	(14)	4.4	(14)	-6.8	(12)	-1.2	(11)
H	3.8	(19)										
H20	5.2											
H22	6.2											
H23	6.1											
H24a	11.6											
H24b	11.6											
H24c	11.6											
H25a	13.7											
H25b	13.7											
H25c	13.7											
H26a	12.4											
H26b	12.4											
H26c	12.4											
H27a	11.4											
H27b	11.4											
H27c	11.4											
H28a	12.6											
H28b	12.6											
H28c	12.6											
H29a	12.0											
H29b	12.0											
H29c	12.0											
H30a	49.5											
H30b	49.5											

Anisotropic Temperature Factors are of the form
Temp=-2*Pi*Pi*(h*h*u11*astar*astar+---+2*h*k*u12*astar*bstar+---)

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

TITLE	*** IC4690 complex 2 ***
Formula	C28 H24 O17 Ru6 W
Formular wt	1422.76
Diffractometer used	Nonius
Space Group	Monoclinic C 2/c
a (angstron)	26.704(5)
b (angstron)	20.5778(12)
c (angstron)	20.580(5)
beta (deg.)	132.308(24)
V (A**3)	8363(3)
Z	8
Dcalc. (g.cm**-3)	2.260
lambda (Angstron)	0.7107
F(000)	5327
Unit cell detn: #;(2theta range)	25;(18.72 - 28.00 deg.)
Scan type	theta/2theta
Scan width (deg.)	2(0.60+0.35tan(theta))
Scan Speed (deg./min)	2.06-8.24
(2Theta)max.	50.0
h k l ranges	(-31; 23)(0; 24)(0; 24)
mu (cm**-1)	49.304
Crystal size (mm)	0.20 X 0.25 X 0.60
Transmission	0.884; 1.000
Temperature (K)	298.
# of meas. reflns.	7327
# of obsed relfns. (I>2.0sig(I))	5887
# of unique reflns.	7327
Rf;Rw	0.023;0.025
GoF	1.29
Refinement program	NRCVAX
# of atoms	78
# of refined params.	500 (5887 out of 7327 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	11.71(13)
(delta/sigma)max.	0.0028
Residual in final D-map (e/A**3)	-0.650; 0.600

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

Table : Bond Distances and Bond Angles of complex **2**.

W-Ru1	2.9957(5)	Ru5-Ru6	2.7274(7)
W-Ru2	2.8822(12)	Ru5-C1	2.222(5)
W-Ru4	2.9216(9)	Ru5-C13	1.869(6)
W-Ru5	2.9857(6)	Ru5-C14	1.879(6)
W-C1	1.965(5)	Ru5-H3	1.96(5)
W-C2	1.970(5)	Ru6-C1	2.418(5)
W-C18	2.387(5)	Ru6-C15	1.882(6)
W-C19	2.358(5)	Ru6-C16	1.893(6)
W-C20	2.360(5)	Ru6-C17	1.913(7)
W-C21	2.323(5)	Ru6-O1	2.087(3)
W-C22	2.314(5)	Si1-C18	1.887(5)
W-H1	1.56(6)	Si1-C23	1.844(7)
Ru1-Ru2	2.8317(10)	Si1-C24	1.815(7)
Ru1-Ru5	2.7408(7)	Si1-C25	1.827(8)
Ru1-C3	1.885(6)	Si2-C20	1.883(6)
Ru1-C4	1.893(6)	Si2-C26	1.857(7)
Ru1-C5	1.906(6)	Si2-C27	1.831(8)
Ru1-H1	1.70(6)	Si2-C28	1.839(9)
Ru2-Ru3	2.8881(7)	C1-O1	1.257(6)
Ru2-Ru4	2.8377(7)	C2-O2	1.260(6)
Ru2-Ru5	2.8240(11)	C3-O3	1.129(8)
Ru2-C2	2.222(5)	C4-O4	1.127(7)
Ru2-C6	1.877(6)	C5-O5	1.132(7)
Ru2-C7	1.914(6)	C6-O6	1.140(7)
Ru2-H2	1.62(5)	C7-O7	1.129(7)
Ru2-H3	1.87(5)	C8-O8	1.120(8)
Ru3-Ru4	2.8271(13)	C9-O9	1.122(8)
Ru3-C2	2.472(5)	C10-O10	1.120(7)
Ru3-C8	1.932(6)	C11-O11	1.140(7)
Ru3-C9	1.902(6)	C12-O12	1.140(7)
Ru3-C10	1.909(6)	C13-O13	1.140(7)
Ru3-O2	2.104(3)	C14-O14	1.137(7)
Ru3-H2	1.76(5)	C15-O15	1.141(8)
Ru4-Ru5	2.7401(7)	C16-O16	1.139(8)
Ru4-Ru6	2.8508(11)	C17-O17	1.129(8)
Ru4-C1	2.314(5)	C18-C19	1.423(7)
Ru4-C2	2.297(5)	C18-C22	1.426(7)
Ru4-C11	1.874(6)	C19-C20	1.442(7)
Ru4-C12	1.894(6)	C20-C21	1.418(8)
Ru4-H3	1.76(5)	C21-C22	1.413(8)

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

	x	y	z	Beq
W	0.258783(10)	0.108974(10)	0.227929(13)	2.197(12)
Ru1	0.274727(23)	-0.035617(21)	0.24915 (3)	3.08 (3)
Ru2	0.357719(21)	0.047545(20)	0.39697 (3)	2.61 (3)
Ru3	0.416295(23)	0.164662(23)	0.50125 (3)	3.19 (3)
Ru4	0.274420(21)	0.146355(21)	0.37772 (3)	2.57 (3)
Ru5	0.220108(21)	0.026012(21)	0.30724 (3)	2.59 (3)
Ru6	0.139585(23)	0.114698(25)	0.29797 (3)	3.42 (3)
Si1	0.08344 (8)	0.12159 (8)	-0.01204 (10)	3.59 (10)
Si2	0.37077 (9)	0.14974 (11)	0.17514 (12)	4.83 (14)
C1	0.18761 (24)	0.11811 (24)	0.2319 (3)	2.4 (3)
C2	0.33881 (24)	0.14643 (24)	0.3410 (3)	2.6 (3)
C3	0.1986 (3)	-0.0765 (3)	0.1456 (4)	4.6 (5)
C4	0.3306 (3)	-0.0719 (3)	0.2327 (4)	4.2 (5)
C5	0.2865 (3)	-0.1051 (3)	0.3196 (4)	4.2 (5)
C6	0.4145 (3)	0.0218 (3)	0.3781 (4)	3.9 (4)
C7	0.3862 (3)	-0.0232 (3)	0.4756 (4)	3.8 (4)
C8	0.5121 (3)	0.1713 (3)	0.5668 (4)	5.0 (5)
C9	0.4091 (4)	0.2509 (3)	0.5274 (4)	5.0 (6)
C10	0.4302 (3)	0.1290 (3)	0.5980 (4)	4.4 (4)
C11	0.2516 (3)	0.2340 (3)	0.3461 (4)	3.8 (4)
C12	0.2827 (3)	0.1591 (3)	0.4758 (4)	4.3 (5)
C13	0.1419 (3)	-0.0212 (3)	0.2192 (4)	3.7 (4)
C14	0.2291 (3)	-0.0308 (3)	0.3860 (4)	3.8 (4)
C15	0.0526 (3)	0.0774 (4)	0.2238 (4)	5.4 (6)
C16	0.1637 (3)	0.0802 (3)	0.4011 (4)	4.6 (5)
C17	0.1069 (3)	0.1965 (3)	0.3005 (4)	4.8 (5)
C18	0.1752 (3)	0.1446 (3)	0.0791 (3)	2.9 (3)
C19	0.2309 (3)	0.1184 (3)	0.0926 (3)	2.9 (3)
C20	0.2917 (3)	0.1557 (3)	0.1568 (3)	3.2 (4)
C21	0.2722 (3)	0.2059 (3)	0.1832 (3)	3.2 (4)
C22	0.2028 (3)	0.1987 (3)	0.1376 (3)	3.1 (4)
C23	0.0681 (4)	0.1195 (4)	-0.1138 (4)	6.7 (6)
C24	0.0680 (3)	0.0416 (4)	0.0085 (5)	6.7 (5)
C25	0.0310 (4)	0.1844 (4)	-0.0204 (5)	7.5 (6)
C26	0.3501 (4)	0.1954 (4)	0.0818 (5)	5.9 (7)
C27	0.4422 (4)	0.1866 (6)	0.2807 (6)	9.9 (9)
C28	0.3886 (4)	0.0644 (4)	0.1705 (6)	7.2 (8)
O1	0.12923 (17)	0.14009 (18)	0.19124 (22)	3.2 (3)
O2	0.38606 (17)	0.18749 (17)	0.37948 (21)	3.00 (23)
O3	0.1528 (3)	-0.1030 (3)	0.0862 (3)	7.7 (4)
O4	0.3623 (3)	-0.09482 (25)	0.2211 (4)	7.2 (5)
O5	0.2925 (3)	-0.14882 (21)	0.3577 (3)	6.3 (5)
O6	0.45358 (21)	0.0076 (3)	0.3740 (3)	5.9 (4)
O7	0.40517 (24)	-0.06513 (23)	0.5226 (3)	6.2 (4)
O8	0.56770 (23)	0.1746 (3)	0.6053 (4)	8.1 (4)
O9	0.4075 (3)	0.30044 (24)	0.5487 (3)	8.1 (6)
O10	0.4392 (3)	0.1057 (3)	0.6542 (3)	7.0 (4)
O11	0.2362 (3)	0.28641 (20)	0.3225 (3)	6.2 (5)
O12	0.28754 (25)	0.1709 (3)	0.5339 (3)	6.8 (4)
O13	0.09526 (22)	-0.05302 (23)	0.1714 (3)	5.9 (3)
O14	0.2333 (3)	-0.06521 (25)	0.4327 (3)	6.4 (4)
O15	-0.0003 (3)	0.0552 (3)	0.1768 (4)	8.8 (5)
O16	0.1762 (3)	0.0582 (3)	0.4613 (3)	7.2 (5)

O17	0.0868	(3)	0.2449	(3)	0.3002	(4)	8.7	(6)
H1	0.265	(3)	0.040	(3)	0.207	(4)	6.7	(17)
H2	0.425	(3)	0.085	(3)	0.480	(3)	4.8	(14)
H3	0.300	(3)	0.067	(3)	0.416	(3)	5.0	(14)
H19	0.229		0.080		0.063		3.8	
H21	0.302		0.240		0.226		4.1	
H22	0.177		0.227		0.145		3.6	
H23a	0.023		0.107		-0.164		7.1	
H23b	0.078		0.160		-0.126		7.1	
H23c	0.098		0.087		-0.109		7.1	
H24a	0.022		0.028		-0.036		6.0	
H24b	0.096		0.009		0.012		6.0	
H24c	0.079		0.040		0.064		6.0	
H25a	-0.016		0.172		-0.057		6.6	
H25b	0.046		0.195		0.037		6.6	
H25c	0.032		0.225		-0.044		6.6	
H26a	0.387		0.195		0.084		7.6	
H26b	0.312		0.176		0.026		7.6	
H26c	0.339		0.239		0.081		7.6	
H27a	0.483		0.184		0.292		10.5	
H27b	0.434		0.231		0.283		10.5	
H27c	0.451		0.165		0.329		10.5	
H28a	0.428		0.060		0.178		8.4	
H28b	0.394		0.038		0.212		8.4	
H28c	0.351		0.045		0.112		8.4	

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	2.954(10)	2.888(10)	2.589(10)	-0.178(9)	1.899(9)	-0.149(9)
Ru1	4.59 (3)	2.994(23)	4.25 (3)	-0.019(20)	3.032(24)	-0.340(20)
Ru2	2.906(22)	3.459(23)	3.035(22)	-0.028(18)	1.787(19)	0.217(18)
Ru3	3.670(25)	4.36 (3)	2.779(23)	-0.868(21)	1.638(21)	-0.317(20)
Ru4	3.424(23)	3.535(23)	2.757(21)	-0.184(19)	2.068(20)	-0.293(18)
Ru5	3.115(22)	3.614(24)	3.124(22)	-0.428(19)	2.107(20)	-0.058(18)
Ru6	3.796(25)	5.90 (3)	4.24 (3)	0.002(23)	3.079(23)	-0.292(23)
Si1	3.51 (8)	5.47 (10)	3.24 (8)	-0.10 (8)	1.69 (7)	0.37 (7)
Si2	4.87 (10)	9.74 (16)	5.12 (11)	-1.60 (10)	3.92 (10)	-0.79 (10)
C1	3.3 (3)	3.4 (3)	2.8 (3)	-0.36 (23)	2.24 (24)	-0.34 (22)
C2	3.2 (3)	3.7 (3)	2.9 (3)	0.06 (23)	2.06 (24)	-0.14 (23)
C3	5.1 (4)	4.7 (4)	6.6 (4)	0.4 (3)	3.4 (4)	-1.1 (3)
C4	6.4 (4)	4.5 (4)	6.8 (4)	-0.3 (3)	5.1 (4)	-0.6 (3)
C5	6.1 (4)	4.5 (4)	6.1 (4)	-0.4 (3)	4.3 (4)	-0.3 (3)
C6	4.3 (3)	5.2 (4)	4.3 (3)	0.4 (3)	2.5 (3)	0.4 (3)
C7	3.9 (3)	4.7 (4)	4.6 (3)	-0.6 (3)	2.3 (3)	0.7 (3)
C8	5.2 (4)	6.8 (5)	4.7 (4)	-0.8 (3)	2.4 (3)	1.1 (3)
C9	8.8 (5)	5.1 (4)	4.2 (4)	-0.6 (4)	4.0 (4)	-0.6 (3)
C10	4.6 (4)	6.4 (4)	3.6 (3)	-0.9 (3)	1.9 (3)	-0.1 (3)
C11	5.3 (4)	5.2 (4)	4.0 (3)	-0.4 (3)	3.2 (3)	-0.8 (3)
C12	5.0 (4)	6.7 (4)	4.4 (3)	-0.7 (3)	3.0 (3)	-1.2 (3)
C13	4.9 (3)	4.9 (4)	4.3 (3)	-0.4 (3)	3.1 (3)	0.0 (3)
C14	4.4 (3)	5.3 (4)	4.4 (3)	-0.6 (3)	2.8 (3)	0.5 (3)
C15	5.8 (4)	10.2 (6)	6.5 (4)	-1.8 (4)	5.0 (4)	-1.5 (4)
C16	5.3 (4)	7.4 (5)	6.1 (4)	-0.2 (4)	4.3 (4)	-0.2 (4)
C17	5.8 (4)	7.5 (5)	6.5 (4)	0.3 (4)	4.7 (4)	-0.6 (4)
C18	3.8 (3)	4.1 (3)	2.6 (3)	0.0 (3)	1.97 (25)	0.91 (24)
C19	4.2 (3)	4.5 (3)	2.6 (3)	-0.4 (3)	2.4 (3)	-0.03 (24)
C20	4.1 (3)	4.9 (3)	3.6 (3)	-0.9 (3)	2.7 (3)	0.0 (3)
C21	4.7 (3)	3.7 (3)	3.4 (3)	-1.1 (3)	2.6 (3)	0.00 (24)
C22	4.6 (3)	3.4 (3)	3.2 (3)	0.3 (3)	2.4 (3)	0.45 (24)
C23	6.9 (5)	12.5 (7)	3.9 (4)	-2.3 (5)	2.8 (4)	-1.2 (4)
C24	4.9 (4)	8.7 (6)	6.4 (5)	-2.5 (4)	1.5 (4)	1.6 (4)
C25	5.4 (5)	10.3 (7)	6.8 (5)	2.7 (4)	1.7 (4)	-0.1 (5)
C26	9.5 (6)	8.1 (5)	9.1 (6)	-1.8 (5)	7.9 (5)	-0.2 (4)
C27	7.6 (6)	23.7 (12)	9.2 (6)	-7.3 (7)	6.9 (6)	-7.4 (7)
C28	9.1 (6)	11.2 (7)	10.2 (6)	3.2 (5)	7.8 (6)	2.9 (5)
O1	3.24 (20)	5.60 (24)	3.43 (20)	0.71 (18)	2.25 (18)	0.25 (18)
O2	3.66 (20)	4.08 (21)	3.03 (19)	-0.89 (17)	2.00 (17)	-0.17 (16)
O3	6.9 (3)	8.8 (4)	7.9 (4)	-1.2 (3)	2.7 (3)	-3.5 (3)
O4	10.8 (4)	8.0 (4)	13.9 (5)	0.1 (3)	10.5 (4)	-1.6 (3)
O5	11.7 (4)	4.3 (3)	9.4 (4)	0.3 (3)	7.8 (4)	1.3 (3)
O6	4.8 (3)	11.7 (4)	6.8 (3)	1.1 (3)	4.2 (3)	-0.1 (3)
O7	7.3 (3)	7.4 (3)	7.1 (3)	1.0 (3)	4.1 (3)	4.0 (3)
O8	3.9 (3)	12.5 (5)	10.0 (4)	-0.8 (3)	2.8 (3)	2.5 (4)
O9	16.5 (6)	5.8 (3)	7.7 (4)	0.6 (4)	7.8 (4)	-0.7 (3)
O10	8.9 (4)	11.8 (5)	5.1 (3)	-0.1 (3)	4.4 (3)	2.5 (3)
O11	10.4 (4)	4.1 (3)	9.2 (4)	1.5 (3)	6.5 (3)	0.7 (3)
O12	8.6 (4)	13.5 (5)	4.3 (3)	-0.9 (3)	4.7 (3)	-1.8 (3)
O13	5.2 (3)	8.6 (3)	5.8 (3)	-3.4 (3)	2.65 (25)	-1.9 (3)
O14	9.1 (4)	9.0 (4)	7.9 (3)	0.5 (3)	6.5 (3)	3.5 (3)
O15	7.3 (4)	17.3 (6)	10.6 (5)	-4.9 (4)	6.8 (4)	-5.5 (4)

O16	10.8	(4)	12.6	(5)	7.3	(3)	0.3	(4)	7.4	(4)	1.9	(3)
O17	12.0	(5)	9.7	(4)	12.8	(5)	2.7	(4)	9.0	(5)	-1.2	(4)
H1	8.5	(21)										
H2	6.1	(17)										
H3	6.3	(17)										
H19	4.8											
H21	5.2											
H22	4.6											
H23a	9.0											
H23b	9.0											
H23c	9.0											
H24a	7.5											
H24b	7.5											
H24c	7.5											
H25a	8.3											
H25b	8.3											
H25c	8.3											
H26a	9.7											
H26b	9.7											
H26c	9.7											
H27a	13.3											
H27b	13.3											
H27c	13.3											
H28a	10.7											
H28b	10.7											
H28c	10.7											

Anisotropic Temperature Factors are of the form
Temp=-2*Pi*Pi*(h*h*u11*astar*astar+---+2*h*k*u12*astar*bstar+---)