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2,3-Diruthenocenylcyclopropenone

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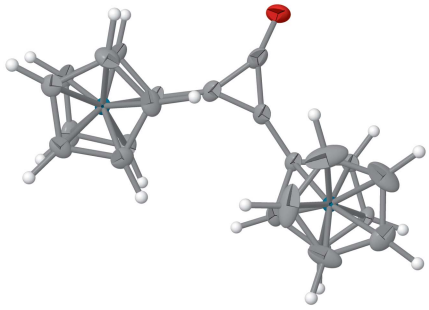
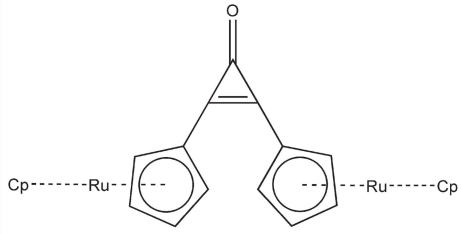
In the title compound, $[Ru_2(C_5H_5)_2(C_{13}H_8O)]$, the Ru—C bond lengths in the two ruthenocenyl moieties are in the range of 2.155 (4)–2.196 (3) Å. Both cyclopentadienyl (Cp) rings are planar and parallel with staggered (18.6 °) and eclipsed (3.1°) conformations between the mutual orientations of rings in the independent sandwiches of each ruthenocenyl molecule. In the crystal, there are intermolecular C—H···O hydrogen bonds between Cp carbon donor atoms and the cyclopropenone O atom of adjacent molecules, forming $R_2^2(14)$ and $R_6^6(38)$ motifs along the *b* and *c* axes.

3D view



Chemical scheme



Structure description

Cyclopropenone is a molecule of interest since it combines remarkable stability with extreme strain; various physical studies (Benson *et al.*, 1973) suggest that much of its stability is derived from the special conjugative stabilization of the two-π electron systems. In addition, cyclopropenone has a number of interesting chemical properties (Breslow *et al.*, 1972) that suggest it could be a useful synthetic intermediate.

Metallocenyl-substituted cyclopropenones allow introducing ruthenium or iron atoms in a large number of different organic compounds (Klimova *et al.*, 2006), with different properties in medicinal chemistry mainly as anticancer compounds (Ornelas, 2011; Jaouen *et al.*, 2015; Gasser *et al.*, 2011), antibacterial properties (Patra *et al.*, 2010), and antimalarial agents (Beagley *et al.*, 2002).

Investigations into the chemistry of 2,3-diferrocenylcyclopropenones (Klimova *et al.*, 2003) has allowed the construction of heterocycles with two ferrocene units (Klimova *et al.*, 2009). 2,3-Diruthenocenylcyclopropenone extended conjugated metal-containing systems are of interest because they can be used as model compounds for molecular wires (Klimova *et al.*, 2007; Li *et al.*, 2010; Ward, 1995). Furthermore, the electron-donating nature of ruthenocenyl functionalities on cyclopropenone systems enhances their stabi-

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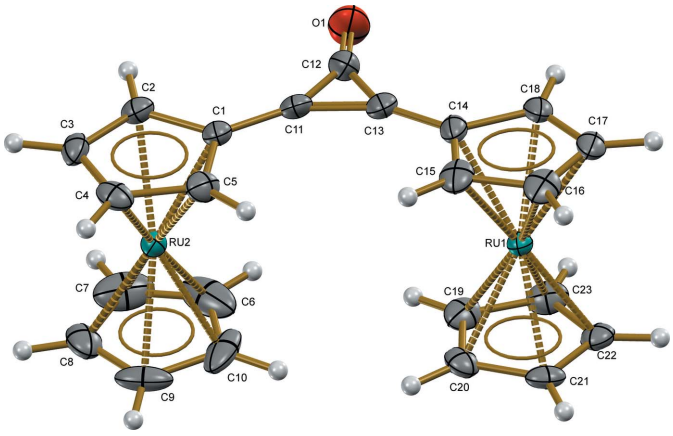

Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 60% probability level.

lity by delocalizing the positive charge (Agranat & Aharon-Shalom, 1975; Hauser & Lednicer, 1972).

In a continuation of this work, we present here the synthesis and crystal structure of 2,3-diruthenocenylcyclopropenone. The synthesis of this compound was made by using Friedel–Crafts alkylation of ruthenocene with tetrachlorocyclopropene in the presence of $AlCl_3$ (Agranat & Aharon-Shalom, 1975; Klimova *et al.*, 2003).

The title compound (Fig. 1) consist of two ruthenocenyl units bonded on 2,3-disubstitution of the three-membered ring of the cyclopropenone. The C═O [1.221 (4) Å], C—C [1.404 (5) Å] and C═C [1.379 (4) Å] bond lengths are similar to the analogous bond lengths observed in 2,3-diferrocenylcyclopropenone and 2,3-diphenylcyclopropenone (Tsukada *et al.*, 1974). The Ru—C distances in the two ruthenocenyl moieties are in the range 2.155 (4)–2.196 (3) Å. Both Cp rings are planar and parallel, with an average value of torsion angles for C—*Cg*—*Cg*—C of 3.1 and 18.6° for the eclipsed (Ru1) and staggered (Ru2) conformations between the mutual orientations of rings in the independent sandwiches of each molecule of ruthenocenyl.

In the crystal packing, there are intermolecular C—H···O hydrogen bonds between Cp carbon donor atoms and the cyclopropenone O atom of adjacent molecules. The C5—H5···O1 (2.37 Å) and C23—H23···O1 (2.41 Å) intermolecular interactions form $R_2^2(14)$ and $R_6^6(38)$ motifs and show a complex growing along the *b* and *c* axes (Fig. 2).

Table 1
Experimental details.

Crystal data	
Chemical formula	$[Ru_2(C_5H_5)_2(C_{13}H_8O)]$
M_r	512.51
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	130
a, *b*, *c* (Å)	10.9880 (6), 14.2341 (8), 12.2219 (7)
β (°)	106.357 (5)
V (Å^3)	1834.19 (18)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.66
Crystal size (mm)	0.25 × 0.2 × 0.13
Data collection	
Diffractometer	Agilent Xcalibur Atlas Gemini
Absorption correction	Analytical (*CrysAlis RED*; Agilent, 2013)
T_{min}, T_{max}	0.726, 0.818
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10469, 4368, 3246
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.693
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.063, 1.03
No. of reflections	4368
No. of parameters	235
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.46, −0.57

Computer programs: *CrysAlis PRO* (Agilent, 2013, *CrysAlis RED* (Agilent, 2013), *SHELXT* (Sheldrick, 2015*a*), *SHELXS2014* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *WinGX* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008).

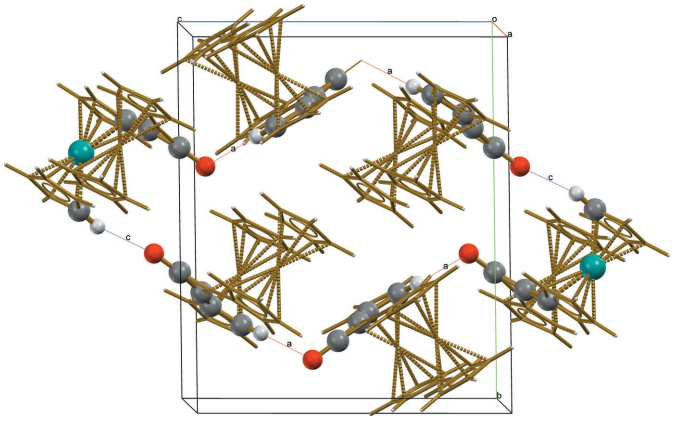

Figure 2
The crystal structure of the title compound, showing intermolecular C—H···O interactions, forming graph-set motifs $R_2^2(14)$ and $R_6^6(38)$ along the *b* and *c* axes.

Synthesis and crystallization

Aluminium chloride (0.67 g, 0.005 mol) was added portion wise over 30 min to a stirred solution of ruthenocene (4.63 g, 0.02 mol) and tetrachlorocyclopropene (3.6 g, 0.02 mol) in dry dichloromethane (200 ml). The mixture was stirred for 1 h at 293 K and then quenched by addition of water (2 × 50 ml), and dried with $MgSO_4$. The solvent was evaporated *in vacuo* and the residue was chromatographed (Al_2O_3; hexane/dichloromethane, 3:1) to give the title compound (yield 68%, yellow crystals, m.p. 529–530 K).

^{1}H NMR (300 MHz, $CDCl_3$) δ: 4.60 (10*H*, s, $2C_5H_5$), δ: 4.83 (4*H*, m, C_5H_4), δ: 5.10 (4*H*, m, C_5H_4) p.p.m., ^{13}C NMR (75 MHz, $CDCl_3$) δ: 69.16 ($2C_{ipso}$Ru), 72.30 ($2C_5H_5$), δ: 72.76, 73.46 ($2C_5H_4$), 115.73 (2 C), 195.83 (C═O) p.p.m., MS: *m/z* 512 $[M]^+$. Anal. Calcd for $C_{23}H_{18}ORu_2$: C, 53.90, H, 3.54, Ru, 39.44. Found C, 53.76, H, 3.64, Ru 39.63%.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. During the refinement, electron-density peaks were located that were believed to be highly disordered solvent molecules (possibly water). Attempts made to model the solvent molecule were not successful. The SQUEEZE (Spek, 2015) option in *PLATON* (Spek, 2009) indicated there was a small solvent cavity of 9 Å^3. In the final cycles of refinement, this contribution of four electrons to the electron density was removed from the observed data.

Acknowledgements

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full crystallographic data

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Crystal data

$[Ru_2(C_5H_5)_2(C_{13}H_8O)]$
M_r = 512.51
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 10.9880 (6) Å
b = 14.2341 (8) Å
c = 12.2219 (7) Å
β = 106.357 (5)°
V = 1834.19 (18) Å^3
Z = 4
$F(000)$ = 1008
D_x = 1.856 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3321 reflections
θ = 3.5–29.5°
μ = 1.66 mm^{-1}
T = 130 K
Prism, yellow
0.25 × 0.2 × 0.13 mm

Data collection

Agilent Xcalibur Atlas Gemini diffractometer
Graphite monochromator
Detector resolution: 10.4685 pixels mm^{-1}
ω scans
Absorption correction: analytical (*CrysAlis RED*; Agilent, 2013)
T_{min} = 0.726, T_{max} = 0.818
10469 measured reflections
4368 independent reflections
3246 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 29.5°, θ_{min} = 3.5°
h = −14→15
k = −14→19
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.063
S = 1.03
4368 reflections
235 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0211P)^2 + 0.0994P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3407 (3)	0.7411 (2)	0.9224 (3)	0.0204 (7)
C2	0.4659 (3)	0.7248 (2)	0.9965 (3)	0.0206 (7)
H2	0.487019	0.693883	1.073071	0.025*
C3	0.5536 (3)	0.7659 (2)	0.9451 (3)	0.0222 (7)
H3	0.647806	0.767926	0.97844	0.027*
C4	0.4846 (3)	0.8069 (2)	0.8407 (3)	0.0233 (7)
H4	0.522295	0.842095	0.787323	0.028*
C5	0.3529 (3)	0.7919 (2)	0.8252 (3)	0.0235 (7)
H5	0.281909	0.815596	0.760458	0.028*
C6	0.3828 (4)	0.5115 (3)	0.8106 (4)	0.0526 (12)
H6	0.321152	0.481229	0.846122	0.063*
C7	0.5131 (4)	0.5108 (2)	0.8545 (3)	0.0438 (10)
H7	0.56174	0.480541	0.927612	0.053*
C8	0.5653 (3)	0.5545 (2)	0.7769 (3)	0.0324 (8)
H8	0.657952	0.561814	0.785208	0.039*
C9	0.4675 (4)	0.5834 (2)	0.6836 (3)	0.0368 (9)
H9	0.477253	0.614059	0.612957	0.044*
C10	0.3520 (3)	0.5568 (3)	0.7041 (4)	0.0493 (12)
H10	0.265213	0.563968	0.650192	0.059*
C11	0.2254 (3)	0.7098 (2)	0.9444 (3)	0.0240 (7)
C12	0.1613 (3)	0.6592 (2)	1.0097 (3)	0.0302 (8)
C13	0.0946 (3)	0.7084 (2)	0.9110 (3)	0.0250 (7)
C14	−0.0217 (3)	0.7386 (2)	0.8318 (3)	0.0254 (7)
C15	−0.0368 (3)	0.7944 (2)	0.7338 (3)	0.0267 (7)
H15	0.032983	0.82496	0.70902	0.032*
C16	−0.1697 (3)	0.8040 (2)	0.6792 (3)	0.0298 (8)
H16	−0.209125	0.841755	0.609213	0.036*
C17	−0.2353 (3)	0.7529 (2)	0.7442 (3)	0.0283 (8)
H17	−0.329588	0.748284	0.727203	0.034*
C18	−0.1461 (3)	0.7116 (2)	0.8391 (3)	0.0270 (8)
H18	−0.165511	0.674589	0.901634	0.032*
C19	−0.0109 (3)	0.5289 (2)	0.6727 (3)	0.0268 (7)
H19	0.069383	0.507864	0.729121	0.032*
C20	−0.0195 (3)	0.5844 (2)	0.5748 (3)	0.0239 (7)
H20	0.053534	0.609439	0.549912	0.029*
C21	−0.1498 (3)	0.5939 (2)	0.5147 (3)	0.0228 (7)
H21	−0.184815	0.627168	0.440415	0.027*
C22	−0.2210 (3)	0.5445 (2)	0.5764 (3)	0.0224 (7)
H22	−0.315311	0.537214	0.553031	0.027*
C23	−0.1362 (3)	0.5040 (2)	0.6737 (3)	0.0271 (7)
H23	−0.159553	0.462646	0.730796	0.033*
O1	0.1630 (2)	0.60901 (19)	1.0911 (2)	0.0431 (6)
Ru1	−0.11501 (2)	0.65595 (2)	0.68379 (2)	0.01556 (7)
Ru2	0.44667 (2)	0.65524 (2)	0.83492 (2)	0.01558 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0185 (14)	0.0244 (17)	0.0178 (17)	−0.0018 (13)	0.0044 (12)	−0.0078 (13)
C2	0.0234 (15)	0.0209 (16)	0.0160 (17)	−0.0026 (14)	0.0031 (12)	−0.0039 (12)
C3	0.0163 (14)	0.0209 (17)	0.0274 (19)	−0.0035 (13)	0.0030 (13)	−0.0066 (13)
C4	0.0304 (17)	0.0155 (16)	0.0279 (19)	0.0006 (14)	0.0148 (14)	−0.0034 (13)
C5	0.0241 (16)	0.0224 (17)	0.0222 (18)	0.0074 (14)	0.0039 (13)	−0.0014 (13)
C6	0.075 (3)	0.028 (2)	0.075 (4)	−0.023 (2)	0.054 (3)	−0.022 (2)
C7	0.078 (3)	0.0185 (18)	0.032 (2)	0.013 (2)	0.010 (2)	0.0039 (15)
C8	0.0294 (17)	0.0276 (19)	0.044 (2)	0.0005 (15)	0.0171 (17)	−0.0143 (16)
C9	0.073 (3)	0.0204 (18)	0.022 (2)	0.0029 (18)	0.0229 (19)	−0.0036 (14)
C10	0.0286 (19)	0.042 (2)	0.063 (3)	0.0077 (18)	−0.0107 (19)	−0.038 (2)
C11	0.0227 (15)	0.0335 (19)	0.0144 (17)	−0.0009 (15)	0.0031 (13)	−0.0084 (14)
C12	0.0220 (16)	0.046 (2)	0.024 (2)	−0.0068 (16)	0.0091 (14)	−0.0109 (16)
C13	0.0217 (15)	0.0319 (19)	0.0209 (18)	−0.0047 (15)	0.0053 (13)	−0.0123 (14)
C14	0.0155 (14)	0.0305 (19)	0.029 (2)	−0.0017 (14)	0.0052 (13)	−0.0164 (15)
C15	0.0264 (16)	0.0166 (16)	0.035 (2)	−0.0009 (14)	0.0059 (14)	−0.0111 (14)
C16	0.0283 (17)	0.0193 (17)	0.038 (2)	0.0091 (15)	0.0033 (15)	−0.0115 (15)
C17	0.0182 (15)	0.0340 (19)	0.032 (2)	0.0031 (15)	0.0066 (14)	−0.0187 (15)
C18	0.0190 (15)	0.0367 (19)	0.027 (2)	−0.0039 (15)	0.0099 (14)	−0.0179 (15)
C19	0.0294 (17)	0.0191 (17)	0.029 (2)	0.0088 (15)	0.0041 (14)	−0.0049 (14)
C20	0.0263 (16)	0.0199 (17)	0.0290 (19)	0.0023 (14)	0.0134 (14)	−0.0057 (14)
C21	0.0341 (17)	0.0187 (16)	0.0162 (17)	0.0050 (14)	0.0080 (13)	−0.0042 (12)
C22	0.0258 (16)	0.0198 (16)	0.0205 (18)	−0.0021 (14)	0.0046 (13)	−0.0078 (13)
C23	0.0393 (19)	0.0169 (16)	0.025 (2)	−0.0015 (15)	0.0090 (15)	0.0005 (13)
O1	0.0390 (14)	0.0689 (18)	0.0227 (15)	−0.0092 (14)	0.0110 (11)	0.0081 (13)
Ru1	0.01405 (12)	0.01688 (14)	0.01538 (13)	0.00008 (10)	0.00353 (9)	−0.00369 (9)
Ru2	0.01460 (12)	0.01697 (14)	0.01482 (14)	−0.00084 (10)	0.00360 (9)	−0.00068 (9)

Geometric parameters (Å, º)

C1—C5	1.429 (4)	C12—O1	1.221 (4)
C1—C2	1.438 (4)	C12—C13	1.409 (5)
C1—C11	1.438 (4)	C13—C14	1.435 (4)
C1—Ru2	2.167 (3)	C14—C15	1.407 (5)
C2—C3	1.417 (4)	C14—C18	1.446 (4)
C2—Ru2	2.166 (3)	C14—Ru1	2.160 (3)
C2—H2	1	C15—C16	1.430 (4)
C3—C4	1.414 (4)	C15—Ru1	2.168 (3)
C3—Ru2	2.188 (3)	C15—H15	1
C3—H3	1	C16—C17	1.415 (5)
C4—C5	1.422 (4)	C16—Ru1	2.187 (3)
C4—Ru2	2.197 (3)	C16—H16	1
C4—H4	1	C17—C18	1.418 (5)
C5—Ru2	2.189 (3)	C17—Ru1	2.179 (3)
C5—H5	1	C17—H17	1
C6—C7	1.380 (6)	C18—Ru1	2.170 (3)

C6—C10	1.406 (6)	C18—H18	1
C6—Ru2	2.155 (3)	C19—C20	1.415 (5)
C6—H6	1	C19—C23	1.425 (4)
C7—C8	1.386 (5)	C19—Ru1	2.165 (3)
C7—Ru2	2.172 (3)	C19—H19	1
C7—H7	1	C20—C21	1.419 (4)
C8—C9	1.391 (5)	C20—Ru1	2.169 (3)
C8—Ru2	2.185 (3)	C20—H20	1
C8—H8	1	C21—C22	1.417 (4)
C9—C10	1.412 (5)	C21—Ru1	2.180 (3)
C9—Ru2	2.180 (3)	C21—H21	1
C9—H9	1	C22—C23	1.411 (4)
C10—Ru2	2.159 (3)	C22—Ru1	2.176 (3)
C10—H10	1	C22—H22	1
C11—C13	1.379 (4)	C23—Ru1	2.175 (3)
C11—C12	1.404 (5)	C23—H23	1

C5—C1—C2	108.0 (3)	Ru1—C20—H20	126
C5—C1—C11	127.3 (3)	C22—C21—C20	107.8 (3)
C2—C1—C11	124.7 (3)	C22—C21—Ru1	70.88 (17)
C5—C1—Ru2	71.70 (17)	C20—C21—Ru1	70.53 (18)
C2—C1—Ru2	70.59 (16)	C22—C21—H21	126.1
C11—C1—Ru2	122.8 (2)	C20—C21—H21	126.1
C3—C2—C1	107.6 (3)	Ru1—C21—H21	126.1
C3—C2—Ru2	71.84 (17)	C23—C22—C21	108.6 (3)
C1—C2—Ru2	70.65 (17)	C23—C22—Ru1	71.03 (17)
C3—C2—H2	126.1	C21—C22—Ru1	71.16 (17)
C1—C2—H2	126.1	C23—C22—H22	125.7
Ru2—C2—H2	126.1	C21—C22—H22	125.7
C4—C3—C2	108.2 (2)	Ru1—C22—H22	125.7
C4—C3—Ru2	71.53 (17)	C22—C23—C19	107.5 (3)
C2—C3—Ru2	70.18 (16)	C22—C23—Ru1	71.12 (17)
C4—C3—H3	125.9	C19—C23—Ru1	70.43 (17)
C2—C3—H3	125.9	C22—C23—H23	126.2
Ru2—C3—H3	125.9	C19—C23—H23	126.2
C3—C4—C5	109.0 (3)	Ru1—C23—H23	126.2
C3—C4—Ru2	70.85 (17)	C14—Ru1—C19	111.89 (12)
C5—C4—Ru2	70.78 (17)	C14—Ru1—C15	37.96 (13)
C3—C4—H4	125.5	C19—Ru1—C15	127.06 (12)
C5—C4—H4	125.5	C14—Ru1—C20	124.95 (11)
Ru2—C4—H4	125.5	C19—Ru1—C20	38.12 (12)
C4—C5—C1	107.2 (3)	C15—Ru1—C20	112.23 (12)
C4—C5—Ru2	71.37 (16)	C14—Ru1—C18	39.02 (11)
C1—C5—Ru2	70.01 (16)	C19—Ru1—C18	125.15 (13)
C4—C5—H5	126.4	C15—Ru1—C18	64.46 (13)
C1—C5—H5	126.4	C20—Ru1—C18	158.63 (12)
Ru2—C5—H5	126.4	C14—Ru1—C23	127.39 (13)
C7—C6—C10	108.5 (3)	C19—Ru1—C23	38.35 (11)

C7—C6—Ru2	72.0 (2)	C15—Ru1—C23	161.35 (12)
C10—C6—Ru2	71.1 (2)	C20—Ru1—C23	63.95 (12)
C7—C6—H6	125.7	C18—Ru1—C23	111.82 (13)
C10—C6—H6	125.7	C14—Ru1—C22	161.77 (13)
Ru2—C6—H6	125.7	C19—Ru1—C22	63.62 (11)
C6—C7—C8	108.3 (3)	C15—Ru1—C22	159.24 (12)
C6—C7—Ru2	70.8 (2)	C20—Ru1—C22	63.65 (11)
C8—C7—Ru2	71.98 (19)	C18—Ru1—C22	127.20 (11)
C6—C7—H7	125.8	C23—Ru1—C22	37.85 (11)
C8—C7—H7	125.8	C14—Ru1—C17	63.97 (11)
Ru2—C7—H7	125.8	C19—Ru1—C17	158.81 (14)
C7—C8—C9	108.7 (3)	C15—Ru1—C17	63.79 (12)
C7—C8—Ru2	70.92 (19)	C20—Ru1—C17	161.91 (13)
C9—C8—Ru2	71.22 (18)	C18—Ru1—C17	38.07 (12)
C7—C8—H8	125.6	C23—Ru1—C17	125.71 (12)
C9—C8—H8	125.6	C22—Ru1—C17	113.19 (11)
Ru2—C8—H8	125.6	C14—Ru1—C21	158.43 (12)
C8—C9—C10	107.5 (3)	C19—Ru1—C21	63.70 (12)
C8—C9—Ru2	71.63 (19)	C15—Ru1—C21	125.83 (13)
C10—C9—Ru2	70.2 (2)	C20—Ru1—C21	38.10 (11)
C8—C9—H9	126.2	C18—Ru1—C21	161.47 (11)
C10—C9—H9	126.2	C23—Ru1—C21	63.65 (12)
Ru2—C9—H9	126.2	C22—Ru1—C21	37.96 (11)
C6—C10—C9	107.0 (3)	C17—Ru1—C21	128.09 (12)
C6—C10—Ru2	70.8 (2)	C14—Ru1—C16	63.82 (13)
C9—C10—Ru2	71.82 (19)	C19—Ru1—C16	161.69 (13)
C6—C10—H10	126.4	C15—Ru1—C16	38.34 (11)
C9—C10—H10	126.4	C20—Ru1—C16	127.85 (13)
Ru2—C10—H10	126.4	C18—Ru1—C16	63.97 (14)
C13—C11—C12	60.8 (2)	C23—Ru1—C16	158.77 (12)
C13—C11—C1	148.1 (3)	C22—Ru1—C16	126.18 (12)
C12—C11—C1	151.1 (3)	C17—Ru1—C16	37.81 (13)
O1—C12—C11	150.3 (3)	C21—Ru1—C16	113.10 (12)
O1—C12—C13	150.9 (3)	C6—Ru2—C10	38.02 (16)
C11—C12—C13	58.7 (2)	C6—Ru2—C2	120.26 (15)
C11—C13—C12	60.5 (2)	C10—Ru2—C2	151.00 (15)
C11—C13—C14	148.4 (3)	C6—Ru2—C1	113.85 (13)
C12—C13—C14	151.2 (3)	C10—Ru2—C1	121.16 (13)
C15—C14—C13	127.8 (3)	C2—Ru2—C1	38.76 (10)
C15—C14—C18	108.4 (3)	C6—Ru2—C7	37.20 (15)
C13—C14—C18	123.8 (3)	C10—Ru2—C7	62.93 (15)
C15—C14—Ru1	71.36 (18)	C2—Ru2—C7	112.85 (13)
C13—C14—Ru1	121.6 (2)	C1—Ru2—C7	133.66 (14)
C18—C14—Ru1	70.90 (16)	C6—Ru2—C9	63.00 (14)
C14—C15—C16	108.1 (3)	C10—Ru2—C9	37.98 (15)
C14—C15—Ru1	70.69 (17)	C2—Ru2—C9	168.82 (13)
C16—C15—Ru1	71.54 (17)	C1—Ru2—C9	151.90 (13)
C14—C15—H15	125.9	C7—Ru2—C9	62.47 (14)

C16—C15—H15	125.9	C6—Ru2—C8	62.19 (14)
Ru1—C15—H15	125.9	C10—Ru2—C8	62.70 (13)
C17—C16—C15	107.7 (3)	C2—Ru2—C8	132.97 (13)
C17—C16—Ru1	70.77 (17)	C1—Ru2—C8	169.30 (14)
C15—C16—Ru1	70.11 (16)	C7—Ru2—C8	37.10 (14)
C17—C16—H16	126.1	C9—Ru2—C8	37.15 (13)
C15—C16—H16	126.1	C6—Ru2—C3	149.97 (17)
Ru1—C16—H16	126.1	C10—Ru2—C3	170.46 (16)
C16—C17—C18	109.1 (3)	C2—Ru2—C3	37.98 (11)
C16—C17—Ru1	71.42 (17)	C1—Ru2—C3	63.90 (11)
C18—C17—Ru1	70.65 (17)	C7—Ru2—C3	120.52 (13)
C16—C17—H17	125.4	C9—Ru2—C3	133.89 (13)
C18—C17—H17	125.4	C8—Ru2—C3	113.96 (12)
Ru1—C17—H17	125.4	C6—Ru2—C5	134.91 (15)
C17—C18—C14	106.7 (3)	C10—Ru2—C5	114.51 (13)
C17—C18—Ru1	71.28 (18)	C2—Ru2—C5	64.35 (12)
C14—C18—Ru1	70.08 (17)	C1—Ru2—C5	38.29 (11)
C17—C18—H18	126.6	C7—Ru2—C5	170.27 (14)
C14—C18—H18	126.6	C9—Ru2—C5	122.04 (13)
Ru1—C18—H18	126.6	C8—Ru2—C5	151.62 (13)
C20—C19—C23	108.1 (3)	C3—Ru2—C5	63.67 (11)
C20—C19—Ru1	71.10 (17)	C6—Ru2—C4	171.38 (16)
C23—C19—Ru1	71.22 (17)	C10—Ru2—C4	135.16 (16)
C20—C19—H19	125.9	C2—Ru2—C4	63.40 (12)
C23—C19—H19	125.9	C1—Ru2—C4	63.43 (11)
Ru1—C19—H19	125.9	C7—Ru2—C4	150.67 (14)
C19—C20—C21	107.9 (3)	C9—Ru2—C4	115.09 (12)
C19—C20—Ru1	70.78 (18)	C8—Ru2—C4	122.01 (12)
C21—C20—Ru1	71.37 (17)	C3—Ru2—C4	37.62 (11)
C19—C20—H20	126	C5—Ru2—C4	37.84 (11)
C21—C20—H20	126		

C5—C1—C2—C3	0.4 (3)	C1—C11—C13—C14	−2.2 (9)
C11—C1—C2—C3	179.7 (3)	O1—C12—C13—C11	−179.3 (7)
Ru2—C1—C2—C3	62.7 (2)	O1—C12—C13—C14	1.8 (11)
C5—C1—C2—Ru2	−62.3 (2)	C11—C12—C13—C14	−178.9 (6)
C11—C1—C2—Ru2	117.0 (3)	C11—C13—C14—C15	2.2 (7)
C1—C2—C3—C4	−0.2 (3)	C12—C13—C14—C15	−179.6 (5)
Ru2—C2—C3—C4	61.8 (2)	C11—C13—C14—C18	179.5 (4)
C1—C2—C3—Ru2	−61.9 (2)	C12—C13—C14—C18	−2.3 (8)
C2—C3—C4—C5	−0.1 (3)	C11—C13—C14—Ru1	92.4 (6)
Ru2—C3—C4—C5	60.8 (2)	C12—C13—C14—Ru1	−89.5 (6)
C2—C3—C4—Ru2	−60.9 (2)	C13—C14—C15—C16	178.1 (3)
C3—C4—C5—C1	0.4 (3)	C18—C14—C15—C16	0.5 (3)
Ru2—C4—C5—C1	61.2 (2)	Ru1—C14—C15—C16	62.1 (2)
C3—C4—C5—Ru2	−60.8 (2)	C13—C14—C15—Ru1	116.0 (3)
C2—C1—C5—C4	−0.5 (3)	C18—C14—C15—Ru1	−61.6 (2)
C11—C1—C5—C4	−179.8 (3)	C14—C15—C16—C17	−0.4 (3)

Ru2—C1—C5—C4	−62.1 (2)	Ru1—C15—C16—C17	61.1 (2)
C2—C1—C5—Ru2	61.6 (2)	C14—C15—C16—Ru1	−61.6 (2)
C11—C1—C5—Ru2	−117.7 (3)	C15—C16—C17—C18	0.2 (3)
C10—C6—C7—C8	−0.4 (4)	Ru1—C16—C17—C18	60.9 (2)
Ru2—C6—C7—C8	−62.6 (2)	C15—C16—C17—Ru1	−60.7 (2)
C10—C6—C7—Ru2	62.2 (3)	C16—C17—C18—C14	0.2 (3)
C6—C7—C8—C9	0.3 (4)	Ru1—C17—C18—C14	61.5 (2)
Ru2—C7—C8—C9	−61.5 (2)	C16—C17—C18—Ru1	−61.4 (2)
C6—C7—C8—Ru2	61.8 (2)	C15—C14—C18—C17	−0.4 (3)
C7—C8—C9—C10	−0.1 (4)	C13—C14—C18—C17	−178.1 (3)
Ru2—C8—C9—C10	−61.4 (2)	Ru1—C14—C18—C17	−62.3 (2)
C7—C8—C9—Ru2	61.3 (2)	C15—C14—C18—Ru1	61.9 (2)
C7—C6—C10—C9	0.4 (4)	C13—C14—C18—Ru1	−115.8 (3)
Ru2—C6—C10—C9	63.1 (2)	C23—C19—C20—C21	−0.1 (3)
C7—C6—C10—Ru2	−62.8 (3)	Ru1—C19—C20—C21	−62.1 (2)
C8—C9—C10—C6	−0.2 (4)	C23—C19—C20—Ru1	61.9 (2)
Ru2—C9—C10—C6	−62.5 (2)	C19—C20—C21—C22	0.3 (3)
C8—C9—C10—Ru2	62.3 (2)	Ru1—C20—C21—C22	−61.4 (2)
C5—C1—C11—C13	−4.8 (7)	C19—C20—C21—Ru1	61.7 (2)
C2—C1—C11—C13	176.0 (4)	C20—C21—C22—C23	−0.3 (3)
Ru2—C1—C11—C13	−96.0 (5)	Ru1—C21—C22—C23	−61.5 (2)
C5—C1—C11—C12	173.1 (5)	C20—C21—C22—Ru1	61.2 (2)
C2—C1—C11—C12	−6.1 (8)	C21—C22—C23—C19	0.2 (3)
Ru2—C1—C11—C12	81.9 (6)	Ru1—C22—C23—C19	−61.4 (2)
C13—C11—C12—O1	179.4 (7)	C21—C22—C23—Ru1	61.6 (2)
C1—C11—C12—O1	0.6 (11)	C20—C19—C23—C22	−0.1 (3)
C1—C11—C12—C13	−178.7 (6)	Ru1—C19—C23—C22	61.8 (2)
C1—C11—C13—C12	178.8 (6)	C20—C19—C23—Ru1	−61.9 (2)
C12—C11—C13—C14	179.0 (6)		