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[Dicarbonyl(η^5-cyclopentadienyl)iron(II)]-μ_2-1,3-propanediyl-[dicarbonyl(η^5-cyclopentadienyl)ruthenium(II)]

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The coordination of Fe and Ru in the title compound, [RuFe(C_3H_6)(C_5H_5)$_2$(CO)$_4$], is identical in its effectively tetrahedral form with that observed in closely analogous materials. However, partial [24.08 (17)%] disorder by interchange of Fe and Ru renders the bonds to Fe and Ru here apparently slightly longer and shorter, respectively, than they are in the analogues.

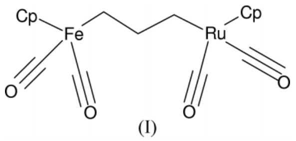
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.024
wR factor = 0.062
Data-to-parameter ratio = 25.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

This structure determination was undertaken in the context of an ongoing study of heterobimetallic compounds (Friedrich & Moss, 1993). The connectivity and atom labelling of the molecule of the title compound, (I), is shown in Fig. 1. The coordination of Fe and Ru in (I) is compared to that in the closely analogous (propane-1,3-diyl)bis[dicarbonyl(η^5-cyclopentadienyl)iron], (II) (PRCFEC; Pope *et al.*, 1976) and (pentane-1,5-diyl)-bis[dicarbonyl(η^5-cyclopentadienyl)ruthenium], (III) (JEHVUN; Finch *et al.*, 1989) in Table 2. There it is clear that, whereas the effectively tetrahedral coordination of the metal atoms is very similar throughout, in (I), the bonds to Fe tend to be slightly longer than in (II) and the bonds to Ru somewhat shorter than they are in (III). In a previous structural model where twin refinement, but no other form of disorder, had been introduced a concern, not noted in the *checkCIF* validation report obtained at that time, was the disparity between the U_{eq} values for Fe1 and Ru1 of 0.0038 (1) and 0.0168 (1) Å^2, respectively. This prompted the rerefinement presented here, in which disorder by partial replacement of Ru by Fe and *vice versa* was introduced and twin refinement abandoned. Precisely this form of disorder is found, but in a much more extreme form, in the structure of the wholly analogous [dicarbonyl(η^5-cyclopentadienyl)iron(II)]-μ_2-1,6-*n*-hexanediyl-[dicarbonyl(η^5-cyclopentadienyl)ruthenium(II)] (Archer *et al.*, 1991). The disordered model has proved to be technically superior on every count, giving much better R factors, much lower residual electron densities, lower s.u. values and a satisfactory estimate of the Flack x parameter with no indication of the need for twin refinement. Clearly it also accounts for the bond-length differences between (I) and (II) and (III).



(I)

The C—C and C—O bonds in (I) are unexceptional and are not discussed further. Selected torsion angles are given in

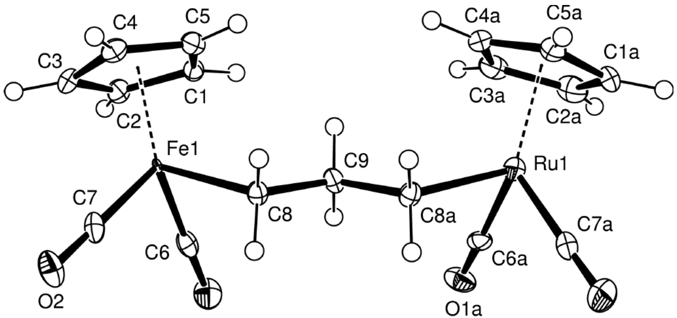


Figure 1
The molecule of (I), showing the atom-labelling scheme. Non-H atoms are shown as 50% probability displacement ellipsoids and H atoms as spheres of arbitrary radius. Metal–cyclopentadienyl bonds are represented by dashed lines joining the centroid of the ligand ring and the metal atom.

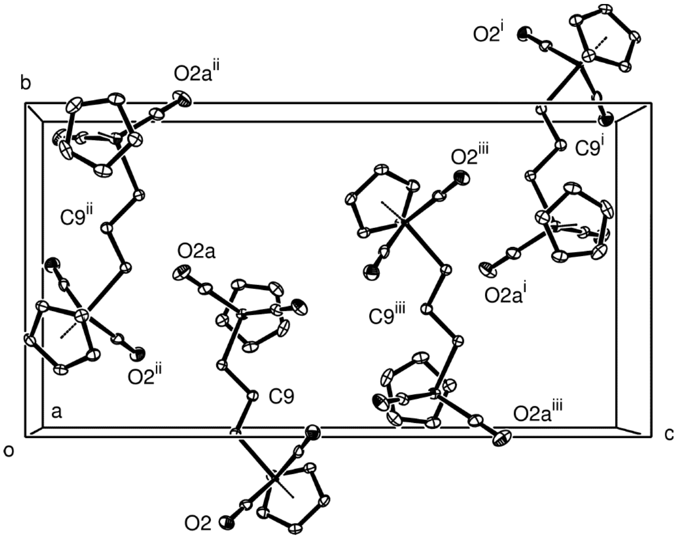


Figure 2
The unit cell of (I). Non-H atoms are shown as 50% probability displacement ellipsoids and H atoms have been omitted for clarity. Metal–cyclopentadienyl bonds are represented by dashed lines joining the centroid of the ligand ring and the metal atom. [Symmetry codes: (i) $\frac{1}{2}-x, -y, \frac{1}{2}+z$; (ii) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iii) $\frac{1}{2}+x, \frac{1}{2}-y, -z$.]

Table 1. In the unit cell of (I) (Fig. 2), aside from the weak intermolecular interactions given in Table 3, the packing of the molecules involves only van der Waals contacts.

Experimental

Compound (I) was prepared according to a reported procedure (Archer *et al.*, 1991) and crystals were grown by slow diffusion, over several days, of a threefold excess of hexane into a concentrated solution of (I) in dichloromethane held at 263 K.

Crystal data

$[RuFe(C_3H_6)(C_5H_5)_2(CO)_4]$
$M_r = 441.22$
Orthorhombic, $P2_12_12_1$
$a = 6.834\ (3)$ Å
$b = 11.358\ (4)$ Å
$c = 21.303\ (9)$ Å
$V = 1653.6\ (12)$ Å^3
$Z = 4$
$D_x = 1.772$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 15516 reflections
$\theta = 4.1$–31.7°
$\mu = 1.81$ mm^{-1}
$T = 100\ (2)$ K
Plate, yellow
0.40 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur 2 CCD area-detector diffractometer
ω–2θ scans
15 516 measured reflections
5254 independent reflections
5017 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.053$
$\theta_{\rm max} = 31.7°$
$h = -9 \rightarrow 10$
$k = -16 \rightarrow 16$
$l = -28 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.062$
$S = 1.11$
5254 reflections
209 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2 + 0.3168P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.002$
$\Delta\rho_{\rm max} = 0.54$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.81$ e Å^{-3}
Absolute structure: Flack (1983), 2204 Friedel pairs
Flack parameter = 0.040 (18)

Table 1
Selected torsion angles (°).

Fe1—C8—C9—C8*A*	175.08 (14)	Ru1—C8*A*—C9—C8	−175.36 (14)
C6—Fe1—C8—C9	65.20 (16)	C6*A*—Ru1—C8*A*—C9	−58.35 (16)
C7—Fe1—C8—C9	158.18 (16)	C7*A*—Ru1—C8*A*—C9	−150.64 (17)

Table 2
The coordination of *M* (*M* = Fe or Ru; Å,°) in (I), (II) and (III)^{a}.

	I		II^{b}	III^{b}	
	M = Fe1	*M* = Ru1	*M* = Fe1	*M* = Ru1	*M* = Ru1
Cg—*M*	1.7880 (12)	1.8805 (13)	1.737	1.936	1.942
C6—*M*	1.762 (2)	1.834 (2)	1.738	1.860 (8)	1.868 (8)
C7—*M*	1.770 (2)	1.839 (2)	1.753	1.871 (9)	1.888 (8)
C8—*M*	2.089 (2)	2.140 (2)	2.0815	2.179 (8)	2.164 (8)
Cg—*M*—C6	130.33	126.71	127.2	129.6	127.3
Cg—*M*—C7	125.48	130.29	127.6	128.3	129.8
Cg—*M*—C8	120.12	119.85	122.4	120.9	123.5
C6—*M*—C7	93.02 (11)	92.17 (11)	92.8	90.2 (4)	89.3 (4)
C6—*M*—C8	84.90 (9)	87.82 (9)	87.2	86.1 (3)	88.2 (3)
C7—*M*—C8	90.62 (9)	87.07 (9)	87.0	88.4 (4)	85.2 (3)

Notes: (*a*) atom designations are precisely as for Fe and, neglecting the suffix *A*, for Ru in (I). Distances and angles involving the cyclopentadienyl ligand are expressed in terms of its centroid, *Cg*, and mostly lack s.u. values as a consequence. (*b*) Values calculated (*PLATON*; Spek, 2003) from CIF data (PRCFEC; Pope *et al.*, 1976), which lacks s.u. values, for (II) and for (III) (JEHVUN; Finch *et al.*, 1989), extracted from the Cambridge Structural Database (Version 5.24; Allen, 2002), accessed at the Chemical Database Service (Fletcher *et al.*, 1996) of the EPSRC at Daresbury, England.

Table 3
Intermolecular contacts (Å,°) in (I).

	C1*A*—H1*A*···O2^{iv}	C2—H2···*Cg*^{v}
Type	hydrogen bond	C—H···π^{a}
C—H	0.95	0.95
H···*A*	2.57	2.82
$H_{\rm perp}{}^{b}$		2.81
C···*A*	3.414 (3)	3.574
C—H···*A*	148	137
γ^{c}		5.6

Notes: (*a*) acceptor *A* is the centroid of the five-membered ring C1–C5; (*b*) perpendicular from H2 to the ring plane; (*c*) angle between H···*A* and $H_{\rm perp}$. Symmetry codes: (iv) $1+x, 1+y, z$; (v) $x-\frac{1}{2}, -y-\frac{1}{2}, 1-z$.

H atoms were placed in calculated positions, with C—H = 0.99 and 0.95 Å for methylene and cyclopentadienyl H atoms, respectively. All H atoms were refined with a riding model, with $U_{\rm iso} = 1.2U_{\rm eq}$ of the C atom to which they are attached. The extent of disorder of Fe and Ru

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was established by refinement of a free variable occupancy factor, x [final refined value 0.7592 (17)], for sites of the form Fe_x/Ru_{1-x} and Ru_x/Fe_{1-x} with *SHELXL*97 EXYZ and EADP instructions rigorously enforcing identical atomic coordinates and displacement parameters for both atoms in the site.

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2001); cell refinement: *CrysAlisCCD*; data reduction: *CrysAlisRED* (Oxford Diffraction, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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