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Bis[μ-(*S*)-1-(1-ferrocenylethylidene)-4-methylthiosemicarbazide-κ^2C^2,*N*,*S*:*S*]bis(dicarbonylruthenium) dichloromethane solvate

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Key indicators

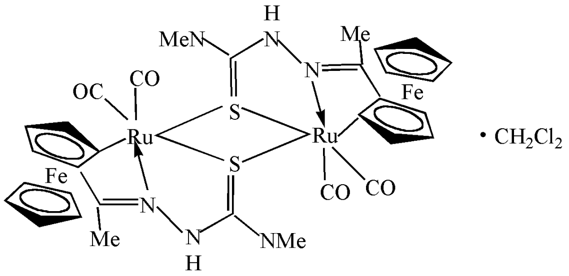
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.012 Å
R factor = 0.066
wR factor = 0.158
Data-to-parameter ratio = 18.5

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

The title compound, $[Ru_2Fe_2(C_5H_5)_2(C_9H_{11}N_2S)_2(CO)_4]\cdot CH_2Cl_2$ or $[FcC(Me){=}NNHC(S)NHMe]_2Ru_2(CO)_4\cdot CH_2Cl_2$ (where Fc = ferrocenyl), was isolated from the reaction of FcC(Me)=NNHC(S)NHMe and $Ru_3(CO)_{12}$. In the dimeric molecule, which lies on a crystallographic twofold axis, the two symmetry-related Ru atoms are in distorted octahedral coordination environments with two bridging S atoms in the equatorial plane and Ru—S bond distances of 2.514 (2) and 2.451 (2) Å.

Comment

The title compound, (I), was obtained from the reaction of FcC(Me)=NNHC(S)NHMe with $Ru_3(CO)_{12}$ under reflux in tetrahydrofuran (Krause & Ruggles, 1958). This type of reaction is useful in the synthesis of transition metal complexes (Zhao & Cao, 1993). The structure of (I) is shown in Fig. 1.



(I)

The molecule lies on a crystallogarphic twofold axis which passes through the center of the four-membered ring made by the two Ru atoms and the two bridging S atoms. Each Ru atom is in a distorted octahedral coordination environment (see Table 1), with bridging Ru—S distances of 2.514 (2) and 2.451 (2) Å. The thio–Schiff base ligand forms a five-membered chelate ring involving atoms Ru, C11, C12, C13 and N3, with the *ortho* H atom of the cyclopendadienyl ring having been displaced in the cyclometallation reaction. Dimer molecules which are related by further twofold symmetry are linked into chains in the *a*-axis direction by N—H···O hydrogen bonds (see Table 2 and Fig. 2).

Experimental

The compounds FcC(Me)=NNHC(S)NHMe (200 mg, 6.6 mmol) and $Ru_3(CO)_{12}$ (425 mg, 6.6 mmol) were dissolved in tetrahydrofuran (THF, 30 ml). After the mixture had been refluxed for 4 h, the THF was removed under reduced pressure. The residue was separated on a silica-gel column. The main band was eluted with petroleum ether (b.p. 333–363 K)—CH_2Cl_2 (*v*/*v* 3:1), from which the title compound

(452 mg, 48%) was obtained as a dark-red solid. Calculated for $C_{33}H_{34}Cl_2Fe_2N_6O_4Ru_2S_2$: C 38.54, H 3.31, N 8.18%; found: C 40.41, H 3.25, N 8.09%.IR (KBr disc): 3398.6 (N—H), 2028.3, 2013.2, 1956.5 (C═O), 1570.4 (C—N). ^{1}H NMR ($CDCl_3$): 4.19 (*t*, 6H, C_5H_3), 4.14 (*s*, 10H, C_5H_5), 2.18(*s*, 1H, NH), 1.57 (*s*, 6H, CH_3), 1.25(*s*, 6H, CH_3).

Crystal data

$[Ru_2Fe_2(C_5H_5)_2(C_9H_{11}N_2S)_2(CO)_4]\cdot CH_2Cl_2$
$M_r = 1027.54$
Monoclinic, $P2/n$
$a = 7.8664$ (12) Å
$b = 10.7140$ (16) Å
$c = 22.582$ (3) Å
$\beta = 99.569$ (3)°
$V = 1876.7$ (5) Å^3
$Z = 2$
$D_x = 1.818$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1101 reflections
$\theta = 5.3$–43.7°
$\mu = 1.85$ mm^{-1}
$T = 293$ (2) K
Plate, dark red
0.40 × 0.19 × 0.05 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{\min} = 0.629$, $T_{\max} = 0.910$
10883 measured reflections
4307 independent reflections
2226 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.167$
$\theta_{\max} = 28.1°$
$h = -9 \rightarrow 10$
$k = -15 \rightarrow 14$
$l = -29 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.158$
$S = 0.86$
4307 reflections
233 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.60$ e Å^{-3}
$\Delta\rho_{\min} = -0.70$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ru—C2	1.847 (10)	Ru—N3	2.105 (6)
Ru—C1	1.897 (9)	Ru—S	2.451 (2)
Ru—C11	2.069 (7)	Ru—S^i	2.514 (2)
C2—Ru—C1	89.7 (4)	C11—Ru—S	156.4 (2)
C2—Ru—C11	94.3 (3)	N3—Ru—S	79.45 (18)
C1—Ru—C11	95.3 (3)	C2—Ru—S^i	175.4 (2)
C2—Ru—N3	89.5 (3)	C1—Ru—S^i	86.9 (3)
C1—Ru—N3	174.0 (3)	C11—Ru—S^i	89.0 (2)
C11—Ru—N3	78.8 (3)	N3—Ru—S^i	94.18 (18)
C2—Ru—S	94.5 (3)	S—Ru—S^i	83.53 (8)
C1—Ru—S	106.5 (3)		

Symmetry code: (i) $\frac{1}{2} - x, y, \frac{3}{2} - z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2A⋯O2^i	0.86	2.38	3.205 (9)	160

Symmetry code: (i) $\frac{3}{2} - x, y, \frac{3}{2} - z$.

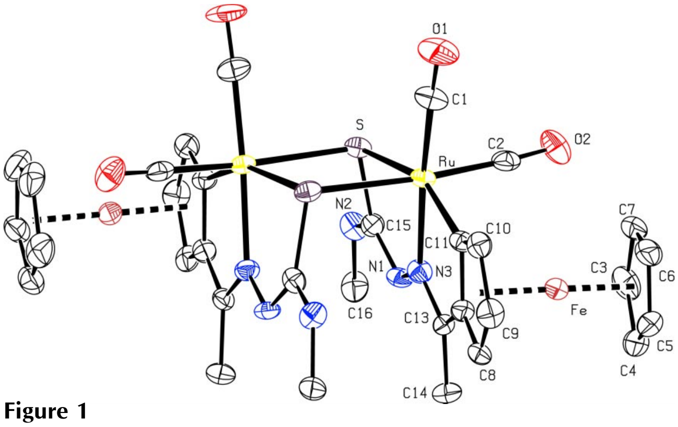


Figure 1
The title molecule with labelling of the non-H atoms and 30% probability ellipsoids. Unlabelled atoms are related by the symmetry code $\frac{3}{2} - x, y, \frac{3}{2} - z$. The solvent molecule has been omitted.

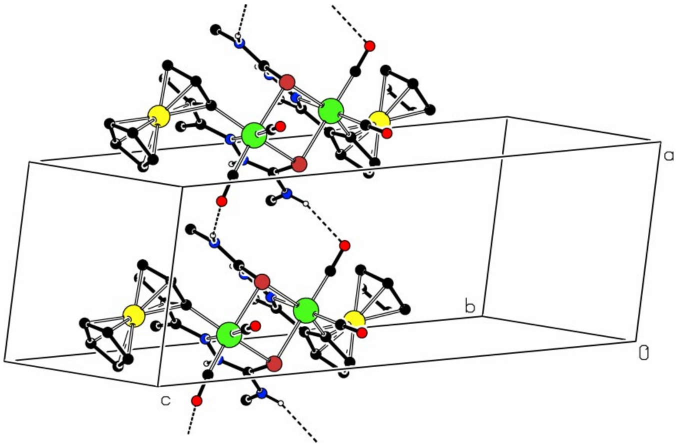


Figure 2
View (Spek, 2004) of the N—H⋯O hydrogen bonding (shown as dashed lines) in the *a*-axis direction. Colour codes: green Ru, yellow Fe, dark-red S, red O, blue N and black C. H atoms bonded to C atoms have been omitted.

All H atoms were placed in calculated positions, with C—H distances ranging from 0.96 to 0.98 Å and N—H distances of 0.86 Å. They were included in the refinement in riding-model approximation, with $U_{\rm iso} = 1.2U_{\rm eq}$ ($1.5_{\rm eq}$ for methyl) of the carrier atom. The highest three peaks in the final difference Fourier map (over 1.00 e Å^3) were within 1.36 Å of Ru. The $R_{\rm int}$ value is higher than normal because the crystal and hence the data were less than ideal quality. This, in turn, can lower the precision of the structure.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT* and *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Zhao, G. & Cao, Y. (1993). *Synth. React. Inorg. Met. Org. Chem.* **23**, 1379–1389.

Acta Cryst. (2004). E**60**, m852–m853 Yang *et al.* • $[Ru_2Fe_2(C_5H_5)_2(C_9H_{11}N_2S)_2(CO)_4].CH_2Cl_2$ **m853**