Refinement of the crystal structure of (1-ethyl-2,3,4,5-tetramethylcyclopentadienyl)[2,4-bis(trifluoromethyl)pentadienyl]ruthenium(II), $C_{18}H_{22}F_6Ru$, at 150 K

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Abstract

$C_{18}H_{22}F_6Ru$, orthorhombic, $Cmc2_1$ (No. 36), $a = 15.1084(6)$ Å, $b = 8.6434(4)$ Å, $c = 13.4790(4)$ Å, $V = 1760.2$ Å³, $Z = 4$, $R_{gt}(F) = 0.024$, $wR_{ref}(F^2) = 0.056$, $T = 150$ K.

Source of material

The synthesis of the compound was as described previously [1]. The compound was crystallized by sublimation near room temperature.

Discussion

The structure, originally determined by Trakarnpruk et al. [1], was redetermined at 150 K to obtain more precise bonding parameters. In addition to improved distances and angles, clearer tilts of several substituents towards the metal were observed (H1a, 16.6°; C4, 8.9°; H3, 6.8°), while a tilt by H1b occurs in the opposite direction, by 51.7° (from least-squares planes).

The size and anisotropy of the ellipsoid for C11 suggest that this atom actually lies in two symmetry-related, but unresolved, positions slightly displaced from the mirror plane. Likewise, the attached hydrogen atoms would be expected to display a similar disorder. Indeed, due to the greater deviations of these atoms from the mirror plane, their separate images could be resolved.

Table 1. Data collection and handling.

Crystal:	yellow prism, size 0.09 × 0.13 × 0.25 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	9.48 cm ⁻¹
Diffractometer, scan mode:	Nonius KappaCCD, 155 frames, $\Delta\phi = 1^\circ$
$2\theta_{max}$:	65.06°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2974, 2974
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 2861
$N(param)_{refined}$:	136
Programs:	SIR97 [2], SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1A)	8b		0.153(2)	0.003(4)	-0.093(2)	0.023(7)
H(1B)	8b		0.064(1)	-0.021(3)	-0.173(2)	0.009(5)
H(3)	4a		0	0.399(5)	-0.123(4)	0.04(1)
H(8A)	8b		0.0761	0.4302	0.1537	0.044
H(8B)	8b		0.1619	0.3240	0.1393	0.044
H(8C)	8b		0.1149	0.3345	0.2455	0.044
H(9A)	8b		0.1892	-0.0309	0.1946	0.045
H(9B)	8b		0.2089	0.0700	0.0975	0.045
H(9C)	8b		0.1756	-0.1050	0.0869	0.045
H(10A)	8b		0.0528	-0.2654	0.1421	0.038
H(11A)	8b	0.5	-0.0168	-0.4036	0.0078	0.075
H(11B)	8b	0.5	0.0593	-0.2851	-0.0245	0.075
H(11C)	8b	0.5	-0.0425	-0.2384	-0.0373	0.075

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Ru	4a	0	0.11968(2)	−0.00458(2)	0.01295(8)	0.01565(9)	0.0111(1)	0	0	−0.0008(1)
F(1)	8b	0.1482(1)	0.4430(2)	−0.0681(2)	0.0384(9)	0.0342(9)	0.060(1)	−0.0138(7)	0.0041(8)	−0.0128(8)
F(2)	8b	0.22768(9)	0.2392(2)	−0.0575(1)	0.0238(7)	0.050(1)	0.0557(9)	−0.0086(7)	−0.0106(7)	0.0111(8)
F(3)	8b	0.1989(1)	0.3367(3)	−0.2004(1)	0.0461(9)	0.068(1)	0.0329(9)	−0.0246(9)	0.0156(8)	0.0074(8)
C(1)	8b	0.0906(2)	0.0415(4)	−0.1174(2)	0.022(1)	0.024(1)	0.016(1)	0.0003(9)	0.0041(8)	−0.0014(9)
C(2)	8b	0.0826(2)	0.2057(3)	−0.1214(2)	0.0201(9)	0.026(1)	0.013(1)	−0.0003(8)	0.0023(8)	0.0016(8)
C(3)	4a	0	0.2836(4)	−0.1296(2)	0.025(1)	0.021(1)	0.013(1)	0	0	0.001(1)
C(4)	8b	0.1639(2)	0.3059(3)	−0.1103(2)	0.024(1)	0.033(1)	0.026(1)	−0.0058(9)	0.0053(9)	0.0047(9)
C(5)	8b	0.0473(1)	0.2000(2)	0.1470(2)	0.0191(9)	0.0201(9)	0.0118(9)	−0.0028(7)	−0.0012(7)	−0.0022(7)
C(6)	8b	0.0767(2)	0.0447(4)	0.1256(2)	0.0177(9)	0.024(1)	0.012(1)	0.0055(9)	−0.0032(8)	0.0010(8)
C(7)	4a	0	−0.0518(3)	0.1130(2)	0.029(1)	0.017(1)	0.011(1)	0	0	−0.0010(9)
C(8)	8b	0.1050(2)	0.3336(3)	0.1736(2)	0.031(1)	0.033(1)	0.025(1)	−0.0117(9)	−0.0032(9)	−0.004(1)
C(9)	8b	0.1707(1)	−0.0100(3)	0.1262(2)	0.022(1)	0.047(2)	0.021(1)	0.014(1)	−0.0006(8)	0.000(1)
C(10)	4a	0	−0.2263(4)	0.1065(3)	0.048(2)	0.020(1)	0.029(2)	0	0	0.001(1)
C(11)	4a	0	−0.2942(4)	0.0043(5)	0.096(3)	0.024(1)	0.030(2)	0	0	0.000(2)

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