Crystal structure of bis(2,2'-bipyridine-*N,N'*)-[1,10-phenanthroline-5,6-dione]ruthenium(II) dihexafluorophosphate acetone solvate, $[\text{Ru}(\text{C}_{10}\text{H}_8\text{N}_2)_2(\text{C}_{12}\text{H}_6\text{N}_2\text{O}_2)](\text{PF}_6)_2 \cdot 1.75(\text{CH}_3)_2\text{CO}$

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Abstract

$\text{C}_{37.25}\text{H}_{32.50}\text{F}_{12}\text{N}_6\text{O}_{3.75}\text{P}_2\text{Ru}$, monoclinic, $P12_1/n1$ (No. 14), $a = 12.1988(6)$ Å, $b = 54.679(3)$ Å, $c = 13.1694(7)$ Å, $\beta = 112.349(6)^\circ$, $V = 8124.5$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.046$, $wR_{\text{ref}}(F^2) = 0.097$, $T = 153$ K.

Source of material

The title compound was synthesised as described previously [1]. The compound was re-crystallized from an acetone solution by slow evaporation of the solvent at room temperature.

Discussion

The title compound crystallizes as an acetone solvate, and per asymmetric unit contains two independent mononuclear ruthenium complexes, four hexafluorophosphate anions, and three and a half acetone solvent molecules. The Ru atoms are coordinated to one 1,10-phenanthroline-5,6-dione (phen-dione) ligand and to two 2,2'-bipyridine (2,2'-bipy) ligands, via the nitrogen atoms. The complex exhibits distorted octahedral geometry around the metal atoms with Ru—N distances in the range 2.049 Å–2.087 Å, (average s.u. 0.005 Å). The coordination angles depart from ideal values by up to $\sim 11^\circ$, varying from 78.7° to 99.0° , due to the chelating nature of the ligands. The phen-dione and 2,2'-bipy ligands are practically planar (deviations from the least-square planes do not exceed 0.09 Å) and are almost perpendicular to one another. In

the crystal there are a number of C...O,N contacts < 3.2 Å, indicating the presence of C—H...O and C—H...F unconventional hydrogen bonds.

Table 1. Data collection and handling.

Crystal:	dark red-brown prism, size $0.10 \times 0.20 \times 0.28$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	5.68 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, φ
$2\theta_{\text{max}}$:	48.18°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	29319, 11429
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5791
$N(\text{param})_{\text{refined}}$:	1115
Programs:	SHELXS-97 [2], SHELXL-97 [3], PLATON [4]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1A)	4e		0.0248	0.0159	0.6373	0.08
H(2A)	4e		0.0918	−0.0244	0.6554	0.08
H(3A)	4e		0.1703	−0.0415	0.8345	0.08
H(4A)	4e		0.1807	−0.0172	0.9862	0.08
H(7A)	4e		0.1862	0.0076	1.1169	0.08
H(8A)	4e		0.1713	0.0368	1.2465	0.08
H(9A)	4e		0.0680	0.0732	1.1834	0.08
H(10A)	4e		−0.0039	0.0823	0.9937	0.08
H(11A)	4e		−0.1646	0.0143	0.7280	0.08
H(12A)	4e		−0.3675	0.0079	0.6869	0.08
H(13A)	4e		−0.4881	0.0409	0.6892	0.08
H(14A)	4e		−0.4084	0.0802	0.7210	0.08
H(17A)	4e		−0.3304	0.1154	0.7147	0.08
H(18A)	4e		−0.2339	0.1525	0.7174	0.08
H(19A)	4e		−0.0253	0.1518	0.7609	0.08
H(20A)	4e		0.0741	0.1144	0.7976	0.08
H(21A)	4e		−0.1738	0.0579	0.5380	0.08
H(22A)	4e		−0.1743	0.0701	0.3647	0.08
H(23A)	4e		0.0018	0.0853	0.3553	0.08
H(28A)	4e		0.4742	0.0950	0.8343	0.08
H(29A)	4e		0.4421	0.0831	0.9929	0.08
H(30A)	4e		0.2567	0.0683	0.9759	0.08
H(33A)	4e		0.1238	0.1671	−0.0281	0.08
H(34A)	4e		0.2188	0.1300	0.0063	0.08
H(35A)	4e		0.2253	0.1069	0.1607	0.08
H(36A)	4e		0.1404	0.1243	0.2787	0.08
H(39A)	4e		0.0472	0.1400	0.3714	0.08
H(40A)	4e		−0.0410	0.1625	0.4735	0.08
H(41A)	4e		−0.1178	0.2012	0.4189	0.08
H(42A)	4e		−0.1063	0.2186	0.2531	0.08

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Table 2. Continued.

Atom	Site	Occ.	x	y	z	U _{iso}
H(43A)	4e		-0.1472	0.1575	-0.0317	0.08
H(44A)	4e		-0.3476	0.1480	-0.1327	0.08
H(45A)	4e		-0.4908	0.1791	-0.1659	0.08
H(46A)	4e		-0.4280	0.2191	-0.1005	0.08
H(49A)	4e		-0.3622	0.2539	-0.0211	0.08
H(50A)	4e		-0.2725	0.2898	0.0689	0.08
H(51A)	4e		-0.0669	0.2917	0.1598	0.08
H(52A)	4e		0.0477	0.2565	0.1683	0.08
H(53A)	4e		0.1915	0.2115	0.3242	0.08
H(54A)	4e		0.3920	0.2207	0.4071	0.08
H(55A)	4e		0.4999	0.2299	0.2923	0.08
H(60A)	4e		0.1780	0.2266	-0.2560	0.08
H(61A)	4e		-0.0190	0.2143	-0.3074	0.08
H(62A)	4e		-0.0959	0.2061	-0.1749	0.08
O(5)	4e		-0.3378(8)	0.0960(2)	0.4796(7)	0.147(3)
C(65)	4e		-0.3587(9)	0.1161(2)	0.436(1)	0.170(6)
C(66)	4e		-0.472(1)	0.1298(2)	0.415(1)	0.186(6)
H(66A)	4e		-0.5289	0.1206	0.4336	0.08
H(66B)	4e		-0.4604	0.1455	0.4501	0.08
H(66C)	4e		-0.5007	0.1319	0.3369	0.08
C(67)	4e		-0.267(1)	0.1298(2)	0.409(1)	0.147(5)

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U _{iso}
H(67A)	4e		-0.2819	0.1459	0.3776	0.08
H(67B)	4e		-0.2018	0.1305	0.4791	0.08
H(67C)	4e		-0.2478	0.1190	0.3614	0.08
H(69A)	4e		-0.6421	0.0702	0.3560	0.08
H(69B)	4e		-0.5860	0.0439	0.3798	0.08
H(69C)	4e		-0.5069	0.0670	0.4277	0.08
H(70A)	4e		-0.4390	0.0522	0.1932	0.08
H(70B)	4e		-0.3764	0.0555	0.3206	0.08
H(70C)	4e		-0.4556	0.0325	0.2728	0.08
H(72A)	4e		0.6036	0.1956	0.1724	0.08
H(72B)	4e		0.6271	0.1700	0.2305	0.08
H(72C)	4e		0.6691	0.1751	0.1338	0.08
H(73A)	4e		0.3961	0.1412	-0.0023	0.08
H(73B)	4e		0.5308	0.1391	0.0171	0.08
H(73C)	4e		0.4888	0.1340	0.1138	0.08
H(75A)	4e	0.50	0.4482	0.0296	0.9029	0.08
H(75B)	4e	0.50	0.5719	0.0167	0.9536	0.08
H(75C)	4e	0.50	0.4664	0.0037	0.8605	0.08
H(76A)	4e	0.50	0.4695	-0.0275	1.0968	0.08
H(76B)	4e	0.50	0.4782	-0.0330	0.9833	0.08
H(76C)	4e	0.50	0.5836	-0.0201	1.0764	0.08

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

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Ru(1)	4e		0.00803(5)	0.05891(1)	0.78412(4)	0.0249(3)	0.0277(3)	0.0335(3)	0.0012(3)	0.0088(2)	0.0054(2)
Ru(2)	4e		0.00312(4)	0.202622(9)	0.08541(4)	0.0179(3)	0.0302(3)	0.0216(2)	-0.0004(3)	0.0053(2)	0.0000(2)
P(1)	4e		0.2289(2)	-0.09987(4)	0.9337(2)	0.043(1)	0.054(2)	0.046(1)	0.004(1)	0.011(1)	0.012(1)
P(2)	4e		0.2592(2)	0.01640(4)	0.5375(2)	0.040(1)	0.042(1)	0.047(1)	-0.004(1)	0.010(1)	-0.0020(9)
P(3)	4e		0.6974(2)	0.20649(3)	0.5519(1)	0.024(1)	0.039(1)	0.0383(9)	-0.0023(9)	0.0072(8)	0.0063(8)
P(4)	4e		-0.7672(2)	0.15734(4)	-0.2973(2)	0.036(1)	0.031(1)	0.051(1)	-0.000(1)	0.014(1)	0.0020(9)
F(1)	4e		0.2364(4)	-0.12590(9)	0.8806(3)	0.070(4)	0.078(4)	0.059(3)	0.009(3)	0.018(3)	-0.013(2)
F(2)	4e		0.3364(4)	-0.10763(9)	1.0431(3)	0.044(3)	0.061(3)	0.074(3)	0.006(3)	-0.012(2)	0.006(2)
F(3)	4e		0.1393(4)	-0.11137(8)	0.9832(3)	0.047(3)	0.050(3)	0.074(3)	0.003(2)	0.024(2)	0.008(2)
F(4)	4e		0.2212(4)	-0.07403(8)	0.9888(3)	0.070(4)	0.039(3)	0.084(3)	0.002(3)	0.021(3)	0.003(2)
F(5)	4e		0.1206(5)	-0.0918(1)	0.8264(3)	0.102(5)	0.100(5)	0.044(2)	0.041(4)	-0.004(3)	0.021(3)
F(6)	4e		0.3192(5)	-0.0884(1)	0.8872(5)	0.112(5)	0.112(5)	0.134(4)	0.005(4)	0.084(4)	0.041(4)
F(7)	4e		0.2917(4)	0.0151(1)	0.6670(3)	0.071(4)	0.112(5)	0.037(2)	-0.013(3)	0.010(2)	0.003(2)
F(8)	4e		0.2141(4)	-0.01132(8)	0.5252(3)	0.068(3)	0.038(3)	0.079(3)	-0.017(3)	0.018(3)	-0.001(2)
F(9)	4e		0.3888(4)	0.00734(8)	0.5549(4)	0.040(3)	0.053(3)	0.096(3)	0.003(2)	0.018(3)	-0.006(2)
F(10)	4e		0.2283(4)	0.01798(8)	0.4096(3)	0.084(4)	0.066(3)	0.045(2)	-0.009(3)	0.021(2)	-0.001(2)
F(11)	4e		0.3043(4)	0.04441(8)	0.5505(3)	0.048(3)	0.038(3)	0.085(3)	-0.002(2)	0.019(2)	-0.006(2)
F(12)	4e		0.1281(4)	0.02540(9)	0.5188(3)	0.041(3)	0.071(4)	0.082(3)	0.004(3)	0.020(2)	-0.001(3)
F(13)	4e		0.7955(4)	0.18584(8)	0.6056(3)	0.048(3)	0.044(3)	0.089(3)	0.014(2)	0.019(3)	0.024(2)
F(14)	4e		0.7320(3)	0.21902(8)	0.6699(3)	0.034(3)	0.085(4)	0.025(2)	0.007(2)	0.003(2)	0.005(2)
F(15)	4e		0.5991(4)	0.19080(8)	0.5735(4)	0.043(3)	0.065(3)	0.095(3)	-0.016(3)	0.032(3)	0.015(3)
F(16)	4e		0.6642(4)	0.19457(8)	0.4340(3)	0.049(3)	0.064(3)	0.054(2)	-0.008(2)	0.010(2)	-0.029(2)
F(17)	4e		0.7967(3)	0.22288(7)	0.5314(3)	0.029(2)	0.040(2)	0.046(2)	-0.005(2)	0.016(2)	0.003(2)
F(18)	4e		0.6032(3)	0.22779(7)	0.4999(2)	0.023(2)	0.054(3)	0.032(2)	0.010(2)	0.001(2)	0.006(2)
F(19)	4e		-0.8354(4)	0.13317(7)	-0.3601(3)	0.049(3)	0.031(3)	0.076(3)	-0.012(2)	0.007(2)	-0.006(2)
F(20)	4e		-0.6438(4)	0.14301(8)	-0.2535(4)	0.035(3)	0.039(3)	0.099(3)	0.005(2)	0.008(2)	-0.001(2)
F(21)	4e		-0.7470(4)	0.16524(8)	-0.4053(3)	0.081(4)	0.062(3)	0.064(3)	-0.012(3)	0.042(3)	-0.002(2)
F(22)	4e		-0.7010(3)	0.18160(7)	-0.2369(3)	0.038(3)	0.040(3)	0.070(2)	-0.005(2)	0.015(2)	-0.010(2)
F(23)	4e		-0.8921(4)	0.17157(8)	-0.3404(3)	0.040(3)	0.060(3)	0.065(3)	0.010(2)	0.005(2)	-0.015(2)
F(24)	4e		-0.7889(4)	0.1493(1)	-0.1898(3)	0.056(3)	0.109(5)	0.047(2)	-0.031(3)	0.014(2)	0.012(2)
O(1)	4e		0.2187(4)	0.0994(1)	0.4309(4)	0.049(4)	0.072(4)	0.047(3)	0.003(3)	0.018(3)	0.024(3)
O(2)	4e		0.4151(4)	0.10034(9)	0.6225(4)	0.034(3)	0.052(3)	0.058(3)	0.003(3)	0.025(3)	0.009(2)
O(3)	4e		0.5196(4)	0.23889(8)	0.1062(3)	0.021(3)	0.051(3)	0.036(2)	-0.011(3)	0.008(2)	-0.007(2)
O(4)	4e		0.3884(4)	0.23944(8)	-0.1143(3)	0.029(3)	0.046(3)	0.033(2)	0.000(2)	0.016(2)	0.002(2)
N(1)	4e		0.0634(5)	0.0232(1)	0.7936(4)	0.024(3)	0.031(3)	0.044(3)	0.001(3)	0.010(3)	0.006(3)
N(2)	4e		0.0472(5)	0.0513(1)	0.9466(4)	0.024(3)	0.045(4)	0.045(3)	-0.001(3)	0.018(3)	0.008(3)
N(3)	4e		-0.1691(5)	0.0504(1)	0.7451(4)	0.028(4)	0.032(4)	0.036(3)	-0.002(3)	0.009(3)	0.003(3)
N(4)	4e		-0.0648(5)	0.0937(1)	0.7773(4)	0.028(4)	0.027(3)	0.030(3)	0.003(3)	0.008(3)	0.002(2)
N(5)	4e		-0.0051(4)	0.06577(9)	0.6258(4)	0.021(3)	0.021(3)	0.035(3)	0.001(3)	0.003(3)	-0.003(2)
N(6)	4e		0.1782(5)	0.0708(1)	0.8133(4)	0.026(4)	0.033(3)	0.033(3)	0.002(3)	0.008(3)	0.006(2)
N(7)	4e		0.0758(4)	0.1682(1)	0.0996(4)	0.023(3)	0.038(4)	0.025(3)	0.005(3)	0.006(3)	0.007(2)
N(8)	4e		-0.0192(4)	0.1898(1)	0.2242(4)	0.020(3)	0.047(4)	0.027(3)	0.001(3)	0.007(3)	0.007(3)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
N(9)	4e		-0.1695(4)	0.19248(9)	-0.0072(3)	0.013(3)	0.030(3)	0.025(2)	-0.003(3)	0.003(2)	-0.002(2)
N(10)	4e		-0.0860(4)	0.23534(9)	0.0782(3)	0.024(3)	0.027(3)	0.021(2)	0.001(3)	0.009(2)	-0.003(2)
N(11)	4e		0.1743(4)	0.21468(9)	0.1692(3)	0.019(3)	0.035(3)	0.021(2)	-0.001(3)	0.004(2)	-0.001(2)
N(12)	4e		0.0522(4)	0.21257(9)	-0.0426(3)	0.020(3)	0.023(3)	0.022(2)	0.002(2)	0.004(2)	-0.001(2)
C(1)	4e		0.0585(6)	0.0090(1)	0.7096(6)	0.038(5)	0.036(5)	0.057(4)	-0.003(4)	0.017(4)	0.006(4)
C(2)	4e		0.0960(7)	-0.0152(1)	0.7189(6)	0.051(6)	0.034(5)	0.076(5)	0.005(4)	0.028(5)	-0.001(4)
C(3)	4e		0.1432(7)	-0.0248(2)	0.8242(7)	0.052(6)	0.035(5)	0.090(6)	0.007(4)	0.023(5)	0.015(5)
C(4)	4e		0.1482(7)	-0.0109(2)	0.9127(6)	0.038(5)	0.049(5)	0.070(5)	0.001(4)	0.014(4)	0.029(5)
C(5)	4e		0.1063(6)	0.0129(1)	0.8953(5)	0.026(5)	0.046(5)	0.053(4)	0.005(4)	0.014(4)	0.015(4)
C(6)	4e		0.1013(6)	0.0297(1)	0.9828(5)	0.029(5)	0.045(5)	0.052(4)	-0.004(4)	0.013(4)	0.015(4)
C(7)	4e		0.1487(7)	0.0232(2)	1.0944(6)	0.048(6)	0.056(6)	0.053(5)	0.002(5)	0.010(4)	0.034(4)
C(8)	4e		0.1374(8)	0.0403(2)	1.1692(6)	0.062(6)	0.100(8)	0.042(4)	-0.004(6)	0.021(5)	0.019(5)
C(9)	4e		0.0787(7)	0.0619(2)	1.1320(5)	0.051(6)	0.093(8)	0.036(4)	0.002(5)	0.019(4)	0.004(4)
C(10)	4e		0.0354(6)	0.0670(1)	1.0193(5)	0.039(5)	0.055(5)	0.053(4)	-0.004(4)	0.026(4)	0.003(4)
C(11)	4e		-0.2150(6)	0.0280(1)	0.7258(5)	0.039(5)	0.041(5)	0.049(4)	0.001(4)	0.012(4)	0.000(4)
C(12)	4e		-0.3347(6)	0.0241(1)	0.7029(5)	0.031(5)	0.046(5)	0.052(4)	-0.014(4)	0.010(4)	-0.001(4)
C(13)	4e		-0.4059(6)	0.0435(2)	0.7022(6)	0.021(5)	0.059(6)	0.067(5)	-0.007(4)	0.012(4)	-0.007(4)
C(14)	4e		-0.3593(6)	0.0664(1)	0.7213(5)	0.026(5)	0.043(5)	0.063(4)	0.000(4)	0.015(4)	-0.002(4)
C(15)	4e		-0.2392(6)	0.0699(1)	0.7407(5)	0.035(5)	0.022(4)	0.032(3)	-0.004(3)	0.010(3)	0.000(3)
C(16)	4e		-0.1838(6)	0.0938(1)	0.7525(5)	0.042(5)	0.042(5)	0.027(3)	0.003(4)	0.018(4)	-0.002(3)
C(17)	4e		-0.2468(6)	0.1157(1)	0.7305(5)	0.038(5)	0.034(4)	0.044(4)	0.009(4)	0.015(4)	0.002(3)
C(18)	4e		-0.1905(7)	0.1374(1)	0.7330(5)	0.051(6)	0.032(5)	0.049(4)	0.016(4)	0.022(4)	0.005(3)
C(19)	4e		-0.0677(7)	0.1369(1)	0.7587(5)	0.056(6)	0.029(4)	0.046(4)	-0.002(4)	0.026(4)	0.002(3)
C(20)	4e		-0.0098(6)	0.1147(1)	0.7801(5)	0.029(4)	0.034(4)	0.037(3)	0.001(4)	0.009(3)	-0.001(3)
C(21)	4e		-0.1023(6)	0.0642(1)	0.5334(5)	0.032(5)	0.028(4)	0.047(4)	0.000(3)	0.006(4)	-0.002(3)
C(22)	4e		-0.1037(6)	0.0715(1)	0.4299(5)	0.042(5)	0.042(5)	0.030(3)	0.011(4)	0.005(3)	0.003(3)
C(23)	4e		-0.0009(7)	0.0805(1)	0.4244(5)	0.044(5)	0.044(5)	0.030(3)	0.011(4)	0.013(4)	0.006(3)
C(24)	4e		0.0995(6)	0.0827(1)	0.5189(5)	0.043(5)	0.034(4)	0.037(4)	0.017(4)	0.017(4)	0.010(3)
C(25)	4e		0.0954(6)	0.0750(1)	0.6199(5)	0.027(4)	0.025(4)	0.033(3)	0.009(3)	0.010(3)	0.002(3)
C(26)	4e		0.1969(6)	0.0773(1)	0.7217(5)	0.031(4)	0.016(3)	0.037(3)	0.010(3)	0.010(3)	0.003(3)
C(27)	4e		0.3075(5)	0.0860(1)	0.7268(5)	0.018(4)	0.023(4)	0.043(4)	0.001(3)	0.013(3)	0.007(3)
C(28)	4e		0.3985(6)	0.0887(1)	0.8283(5)	0.026(4)	0.033(4)	0.046(4)	0.001(3)	0.010(4)	0.003(3)
C(29)	4e		0.3799(6)	0.0817(1)	0.9216(5)	0.019(4)	0.039(4)	0.044(4)	0.004(3)	0.003(3)	0.008(3)
C(30)	4e		0.2689(6)	0.0730(1)	0.9108(5)	0.035(5)	0.038(4)	0.034(4)	0.006(4)	0.007(4)	0.006(3)
C(31)	4e		0.2117(6)	0.0925(1)	0.5164(5)	0.034(5)	0.038(4)	0.051(4)	0.010(4)	0.021(4)	0.019(3)
C(32)	4e		0.3201(6)	0.0937(1)	0.6236(5)	0.034(5)	0.037(4)	0.045(4)	0.005(4)	0.021(4)	0.010(3)
C(33)	4e		0.1269(6)	0.1581(1)	0.0354(5)	0.027(4)	0.034(4)	0.042(4)	0.000(4)	0.010(3)	0.002(3)
C(34)	4e		0.1835(7)	0.1360(1)	0.0553(6)	0.039(5)	0.040(5)	0.053(4)	0.008(4)	0.005(4)	0.000(4)
C(35)	4e		0.1880(7)	0.1227(2)	0.1455(6)	0.050(6)	0.044(5)	0.070(5)	0.017(4)	0.015(5)	0.007(4)
C(36)	4e		0.1366(7)	0.1327(2)	0.2133(6)	0.057(6)	0.047(5)	0.050(4)	0.007(5)	0.007(4)	0.018(4)
C(37)	4e		0.0815(6)	0.1549(1)	0.1893(5)	0.027(5)	0.043(5)	0.040(4)	-0.007(4)	0.005(3)	0.005(3)
C(38)	4e		0.0238(6)	0.1674(2)	0.2566(5)	0.022(4)	0.065(6)	0.028(3)	-0.005(4)	0.003(3)	0.002(3)
C(39)	4e		0.0168(7)	0.1562(2)	0.3499(5)	0.053(6)	0.061(6)	0.038(4)	0.000(5)	0.014(4)	0.016(4)
C(40)	4e		-0.0358(7)	0.1694(2)	0.4083(6)	0.062(6)	0.097(8)	0.026(4)	-0.021(6)	0.014(4)	0.011(5)
C(41)	4e		-0.0818(7)	0.1923(2)	0.3769(5)	0.053(6)	0.115(9)	0.023(4)	-0.017(6)	0.025(4)	-0.005(4)
C(42)	4e		-0.0737(6)	0.2027(2)	0.2790(5)	0.032(4)	0.068(6)	0.033(3)	-0.004(4)	0.012(3)	-0.001(4)
C(43)	4e		-0.2060(6)	0.1701(1)	-0.0456(5)	0.037(5)	0.030(4)	0.034(3)	0.000(3)	0.017(4)	0.005(3)
C(44)	4e		-0.3247(6)	0.1643(1)	-0.1055(5)	0.025(4)	0.042(5)	0.038(4)	-0.004(4)	0.006(3)	0.003(3)
C(45)	4e		-0.4083(6)	0.1826(1)	-0.1261(5)	0.026(4)	0.041(5)	0.043(4)	-0.003(4)	0.010(3)	0.001(3)
C(46)	4e		-0.3713(6)	0.2061(1)	-0.0860(5)	0.022(4)	0.039(5)	0.042(4)	0.006(4)	0.012(3)	0.004(3)
C(47)	4e		-0.2512(6)	0.2104(1)	-0.0252(4)	0.031(4)	0.029(4)	0.023(3)	-0.006(3)	0.013(3)	0.002(3)
C(48)	4e		-0.2049(6)	0.2343(1)	0.0233(4)	0.025(4)	0.028(4)	0.022(3)	0.002(3)	0.009(3)	0.003(3)
C(49)	4e		-0.2779(6)	0.2547(1)	0.0182(5)	0.032(4)	0.037(4)	0.034(3)	0.008(3)	0.016(3)	0.009(3)
C(50)	4e		-0.2254(6)	0.2757(1)	0.0699(5)	0.037(5)	0.038(4)	0.044(4)	0.004(4)	0.025(4)	0.003(3)
C(51)	4e		-0.1044(6)	0.2769(1)	0.1247(5)	0.052(5)	0.031(4)	0.031(3)	-0.003(4)	0.024(4)	-0.005(3)
C(52)	4e		-0.0365(6)	0.2559(1)	0.1285(5)	0.040(5)	0.037(4)	0.026(3)	-0.005(4)	0.014(3)	-0.003(3)
C(53)	4e		0.2344(6)	0.2151(1)	0.2780(5)	0.028(5)	0.055(5)	0.030(3)	-0.003(4)	0.013(3)	0.000(3)
C(54)	4e		0.3535(6)	0.2207(1)	0.3285(5)	0.027(4)	0.067(6)	0.022(3)	-0.008(4)	0.005(3)	-0.005(3)
C(55)	4e		0.4166(6)	0.2264(1)	0.2609(4)	0.022(4)	0.042(4)	0.027(3)	-0.007(3)	0.005(3)	-0.005(3)
C(56)	4e		0.3546(5)	0.2266(1)	0.1475(4)	0.019(4)	0.029(4)	0.022(3)	0.003(3)	0.006(3)	0.001(3)
C(57)	4e		0.2360(5)	0.2205(1)	0.1056(4)	0.023(4)	0.026(4)	0.020(3)	0.007(3)	0.007(3)	0.002(3)
C(58)	4e		0.1662(5)	0.2193(1)	-0.0126(4)	0.018(4)	0.020(3)	0.027(3)	0.006(3)	0.004(3)	0.004(3)
C(59)	4e		0.2172(5)	0.2250(1)	-0.0890(4)	0.019(4)	0.028(4)	0.027(3)	0.006(3)	0.007(3)	0.000(3)
C(60)	4e		0.1463(6)	0.2230(1)	-0.2011(4)	0.028(4)	0.027(4)	0.027(3)	0.000(3)	0.012(3)	-0.004(3)
C(61)	4e		0.0306(6)	0.2159(1)	-0.2310(5)	0.037(5)	0.039(4)	0.023(3)	0.007(4)	0.010(3)	0.004(3)
C(62)	4e		-0.0145(6)	0.2110(1)	-0.1519(4)	0.021(4)	0.032(4)	0.027(3)	0.002(3)	0.000(3)	-0.008(3)
C(63)	4e		0.4170(6)	0.2331(1)	0.0734(5)	0.031(5)	0.026(4)	0.039(4)	0.000(3)	0.015(4)	-0.002(3)
C(64)	4e		0.3428(6)	0.2327(1)	-0.0510(5)	0.025(4)	0.037(4)	0.036(4)	0.007(3)	0.013(3)	-0.002(3)
O(6)	4e		-0.6128(6)	0.0746(2)	0.1853(5)	0.087(6)	0.146(8)	0.081(4)	0.047(5)	0.035(4)	0.042(4)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(68)	4e		−0.5492(8)	0.0630(2)	0.2646(7)	0.049(6)	0.085(8)	0.060(5)	−0.012(6)	0.015(5)	0.003(5)
C(69)	4e		−0.5724(9)	0.0607(2)	0.3662(7)	0.103(9)	0.16(1)	0.079(6)	−0.032(9)	0.044(6)	0.005(7)
C(70)	4e		−0.4464(9)	0.0496(2)	0.2625(8)	0.092(9)	0.11(1)	0.105(7)	0.042(8)	0.036(7)	0.027(7)
O(7)	4e		0.4090(4)	0.1845(1)	0.0375(4)	0.039(3)	0.055(4)	0.050(3)	0.008(3)	0.012(3)	−0.010(3)
C(71)	4e		0.4903(7)	0.1702(2)	0.0802(6)	0.034(5)	0.057(5)	0.043(4)	0.015(5)	0.017(4)	0.005(4)
C(72)	4e		0.6083(7)	0.1783(2)	0.1617(5)	0.057(6)	0.053(6)	0.050(4)	0.003(5)	0.012(4)	−0.007(4)
C(73)	4e		0.4756(7)	0.1438(1)	0.0496(6)	0.041(6)	0.041(5)	0.085(6)	0.004(4)	0.014(5)	−0.009(4)
O(8)	4e	0.50	0.396(1)	0.0146(4)	1.054(2)	0.05(1)	0.24(3)	0.20(2)	0.02(1)	0.03(1)	−0.10(2)
C(74)	4e	0.50	0.464(2)	0.0049(3)	1.022(2)	0.06(2)	0.10(2)	0.09(2)	0.03(2)	0.01(1)	0.01(1)
C(75)	4e	0.50	0.488(3)	0.0142(5)	0.924(2)	0.15(3)	0.06(2)	0.07(2)	−0.01(2)	0.02(2)	−0.01(2)
C(76)	4e	0.50	0.499(3)	−0.0217(4)	1.043(3)	0.17(4)	0.11(3)	0.09(2)	0.04(3)	0.02(2)	−0.02(2)

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