Crystal structure of *trans*-dichloro(1,4-*bis*-(diphenylphosphino)butane)-(*N*,*N*'-1,2-dimethyl-ethanediamine)ruthenium(II), $C_{32}H_{40}Cl_2N_2P_2Ru$

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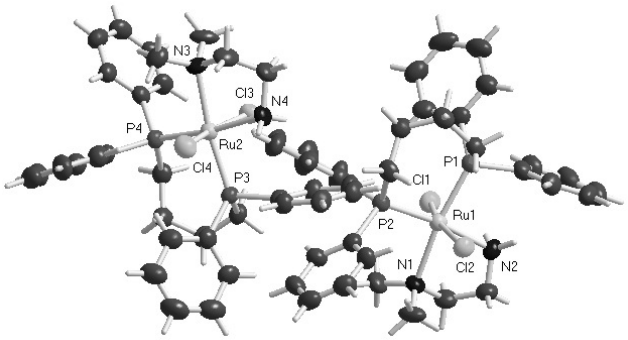

Abstract

$C_{32}H_{40}Cl_2N_2P_2Ru$, orthorhombic, $Pca2_1$ (no. 29), a = 17.0123(3) Å, b = 14.5469(3) Å, c = 25.8211(5) Å, V = 6390.1 Å^3, Z = 8, $R_{gt}(F)$ = 0.0640, $wR_{ref}(F^2)$ = 0.1807, T = 294 K.

Table 1. Data collection and handling.

Crystal:	red blocks, size 0.20×0.20×0.40 mm
Wavelength:	Mo K_α radiation (0.71075 Å)
μ:	7.82 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS RAPID, ω
$2\theta_{max}$:	54.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7483, 7483
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 6078
$N(param)_{refined}$:	721
Programs:	PLATON [15], CrystalClear [16], SIR92 [17], DIAMOND [18], PublCIF [19]

Source of material

All reactions were carried out under inert atmosphere (argon) using high vacuum Schlenk line technique. Starting chemicals were purchased from Aldrich and used without further treatments. The *N*,*N*'-dimethyl-1,2-ethanediamine (0.3 mmol) was dissolved in 10 ml of dry dichloromethane and the resultant solution was added dropwise to a stirred solution of 1,4-*bis*-(diphenylphosphino) butane dichloro ruthenium(II) (0.3 mmol) dissolved in 5 ml of dry dichloromethane. The reaction mixture was stirred for 10 min. at room temperature under inert atmosphere resulting in the change of color from green to light yellow. The yellow solution was concentrated *in vacuo* to 1 ml followed by addition of 30 ml of diethyl ether. The resulting precipitate was collected and recrystallized from dichloromethane/*n*-hexane. Crystals suitable for *X*-ray diffraction were grown by slow diffusion of *n*-hexane into dichloromethane solution.

Experimental details

Most of carbon-bound H-atoms were located from difference Fourier maps, placed in ideal calculated positions [aromatic C–H: 0.93 Å, methylene C–H: 0.97 Å, methyl C–H: 0.96 Å, U_{iso}(H) = 1.2U_{eq}(C)] and refined with their appropriate riding models. Each amine H-atoms were constrained into their positions using three geometrical restraints [N–H: 0.91 Å, U_{iso}(H) = 1.2U_{eq}(N)].

Discussion

Molecular ruthenium complexes are currently used as catalysts to promote a variety of reactions with novel activation routes [1]. Coordination and organometallic complexes of ruthenium have unfolded wide possibilities for a collection of catalytic organic processes, taking advantages of the development in the theoretical *priori* designed high-throughput synthesis of selective catalysts [2-4]. Some of these complexes, in addition to their roles in enantioselective catalytic hydrogenation, photolysis and electrocatalysis, are also showing considerable promises for the production of pharmaceutical intermediates as well as in treatment of cancer [5, 6]. During the past decade, phosphine-based ligands have played an important role in the development of metal complexes-mediated catalysis [1], and many ruthenium diphosphine complexes have been synthesized and characterized [7-9]. Ruthenium(II) complexes of diamine and phosphine mixed ligands have received an attention in recent years owing to their remarkable performance in hydrogen transfer catalytic reactions [10, 11]. According to CSD 2012 [12], there are only two reports on crystal structures of ruthenium(II) complexes bearing 1,4-bis(diphenylphosphino)butane with diamine ligands, namely the *cis*- [13] and *trans*- [14] isomers of dichloro(1,4-*bis*-(diphenyl phosphino)butane)-(1,2-ethylenediamine)ruthenium(II).

We present here the crystal structure of *trans*-dichloro(1,4-*bis*-(diphenylphosphino)butane)-(1,2-dimethyldiaminoethane) ruthenium(II) complex. The title compound is crystallizes with Z' = 2 in an inversion-twinned crystal, with the twinning ratio being 0.43:0.57. The two chemically identical, but crystallographically independent complexes are in *trans*-configuration, with each Ru(II) ion is six-coordinated by two nitrogen atoms of one bidentate dimethyl diaminoethane ligand, two phosphine atoms from one bidentate diphenylphosphino butane ligand and two chloride atoms, forming a slightly distorted ctahedron. The average Ru–N bond distance, for N1 and N3 bearing the methyl groups is 2.318(8) Å, that is longer than the observed 2.179(1) Å of the analogous methyl-free compound [14]. The relative flexibility of the sp^2 carbon atoms in the bidentate diphenylphosphino butane ligand is exemplified by the variation of torsion angles in the asymmetric unit two molecules {49.737(12)° and 49.954(12)°}, also with dimethyl diaminoethane ligand as torsion angles are changed from 60.38(11)° to 60.58(11)°. The two molecules Cl–Ru–Cl *trans*-angles are 164.45(9)° and 164.62(9)°, smaller than the reported value of 165.57(1)° in [14]. The two chloride atoms act as hydrogen bond acceptors, and are engaged in a multitude of intramolecular C–H···Cl non-conventional hy-

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drogen bonding, while the two molecules in the assymmetric unit are interconnected by C–H···π interactions. According to SOLV subroutine of the PLATON [15], *ca.* 2.6% of the unit cell volume is solvent accessible void.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	4*a*	0.6521	0.5588	0.4656	0.069
H(3)	4*a*	0.663	0.7005	0.4249	0.091
H(4)	4*a*	0.6273	0.7164	0.3382	0.088
H(5)	4*a*	0.5721	0.5949	0.2957	0.091
H(6)	4*a*	0.5566	0.4537	0.3357	0.078
H(8)	4*a*	0.4548	0.4473	0.4403	0.075
H(9)	4*a*	0.3287	0.3973	0.4274	0.091
H(10)	4*a*	0.3067	0.2520	0.3919	0.090
H(11)	4*a*	0.4104	0.1567	0.3757	0.076
H(12)	4*a*	0.5389	0.2053	0.3872	0.066
H(13A)	4*a*	0.6522	0.4008	0.5160	0.058
H(13B)	4*a*	0.5714	0.4492	0.5064	0.058
H(14A)	4*a*	0.5669	0.3250	0.5681	0.064
H(14B)	4*a*	0.5021	0.3206	0.5253	0.064
H(15B)	4*a*	0.5618	0.2012	0.4850	0.061
H(15A)	4*a*	0.5579	0.1761	0.5439	0.061
H(16B)	4*a*	0.6832	0.1473	0.5409	0.058
H(16A)	4*a*	0.6976	0.2531	0.5349	0.058
H(18)	4*a*	0.8352	0.2274	0.5299	0.065
H(19)	4*a*	0.9449	0.1620	0.5627	0.076
H(20)	4*a*	0.9948	0.0260	0.5295	0.081
H(21)	4*a*	0.9264	–0.0468	0.4620	0.074
H(22)	4*a*	0.8130	0.0177	0.4289	0.065
H(24)	4*a*	0.6647	0.0096	0.5035	0.070
H(25)	4*a*	0.6089	–0.1266	0.4756	0.082
H(26)	4*a*	0.5739	–0.1446	0.3913	0.100
H(27)	4*a*	0.5938	–0.0280	0.3332	0.120
H(28)	4*a*	0.6526	0.1081	0.3587	0.083
H(29A)	4*a*	0.7846	0.1618	0.3259	0.078
H(29B)	4*a*	0.8543	0.1278	0.3607	0.078
H(29C)	4*a*	0.8715	0.1725	0.3066	0.078
H(30A)	4*a*	0.9380	0.9380	0.3906	0.081
H(30B)	4*a*	0.9525	0.2276	0.3834	0.081
H(30C)	4*a*	0.9017	0.2630	0.4300	0.081
H(31A)	4*a*	0.8139	0.3185	0.2904	0.071
H(31B)	4*a*	0.9037	0.3293	0.3035	0.071
H(32A)	4*a*	0.8729	0.4450	0.3650	0.072
H(32B)	4*a*	0.8421	0.4744	0.3101	0.072
H(34)	4*a*	0.7491	0.0400	0.6008	0.062
H(35)	4*a*	0.7374	0.1846	0.6356	0.072
H(36)	4*a*	0.7785	0.2138	0.7186	0.069
H(37)	4*a*	0.8403	0.1012	0.7658	0.067
H(38)	4*a*	0.8472	–0.0485	0.7329	0.063
H(40)	4*a*	0.9488	–0.0658	0.6299	0.067
H(41)	4*a*	1.0768	–0.1115	0.6457	0.085
H(42)	4*a*	1.1018	–0.2477	0.6916	0.097
H(43)	4*a*	0.9971	–0.3434	0.7121	0.086
H(44)	4*a*	0.8689	–0.2969	0.6951	0.070
H(45A)	4*a*	0.7549	–0.1224	0.5556	0.055
H(45B)	4*a*	0.8349	–0.0719	0.5643	0.055
H(46A)	4*a*	0.9052	–0.1994	0.5498	0.064
H(46B)	4*a*	0.8411	–0.1990	0.5064	0.064
H(47A)	4*a*	0.8485	–0.3475	0.5359	0.062
H(47B)	4*a*	0.8434	–0.3161	0.5938	0.062
H(48A)	4*a*	0.7239	–0.3724	0.5376	0.058
H(48B)	4*a*	0.7111	–0.2661	0.5411	0.058
H(50)	4*a*	0.5736	–0.2869	0.5436	0.063
H(51)	4*a*	0.4595	–0.3511	0.5112	0.070
H(52)	4*a*	0.4052	–0.4763	0.5506	0.073
H(53)	4*a*	0.4668	–0.5471	0.6197	0.075
H(54)	4*a*	0.5902	–0.4919	0.6476	0.063
H(56)	4*a*	0.7370	–0.5059	0.5758	0.071
H(57)	4*a*	0.7926	–0.6421	0.6062	0.096
H(58)	4*a*	0.8367	–0.6513	0.6909	0.100
H(59)	4*a*	0.8116	–0.5296	0.7480	0.086
H(60)	4*a*	0.7516	–0.3993	0.7180	0.074
H(61A)	4*a*	0.6163	–0.3441	0.7509	0.077
H(61B)	4*a*	0.5461	–0.3774	0.7163	0.077
H(61C)	4*a*	0.5295	–0.3279	0.7691	0.077
H(62A)	4*a*	0.4695	–0.1765	0.6810	0.078
H(62B)	4*a*	0.4542	–0.2811	0.6916	0.078
H(62C)	4*a*	0.5062	–0.2510	0.6444	0.078
H(63A)	4*a*	0.5012	–0.1710	0.7687	0.078
H(63B)	4*a*	0.5906	–0.1814	0.7827	0.078
H(64A)	4*a*	0.5633	–0.0290	0.7614	0.079
H(64B)	4*a*	0.5331	–0.0601	0.7065	0.079
H(2A)	4*a*	0.721(3)	0.413(5)	0.335(3)	0.055(3)
H(2B)	4*a*	0.743(3)	0.486(3)	0.376(2)	0.06(2)
H(4A)	4*a*	0.696(4)	–0.076(7)	0.735(4)	0.055(4)
H(4B)	4*a*	0.648(6)	–0.028(5)	0.692(3)	0.054(3)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ru(1)	4*a*	0.72577(4)	0.30928(4)	0.40191(3)	0.0402(4)	0.0391(3)	0.0373(3)	–0.0005(2)	0.0003(3)	–0.0025(3)
Ru(2)	4*a*	0.67965(4)	–0.20149(4)	0.67241(3)	0.0394(3)	0.0394(3)	0.0371(3)	0.0002(2)	–0.0002(3)	–0.0021(3)
Cl(1)	4*a*	0.6582(2)	0.2711(2)	0.32115(9)	0.059(1)	0.052(1)	0.041(1)	–0.004(1)	–0.003(1)	–0.0053(9)
Cl(2)	4*a*	0.7953(1)	0.3875(2)	0.4708(1)	0.051(1)	0.056(1)	0.049(1)	–0.008(1)	–0.007(1)	–0.007(1)
Cl(3)	4*a*	0.6117(1)	–0.1251(2)	0.6021(1)	0.046(1)	0.053(1)	0.054(1)	0.0046(9)	–0.009(1)	0.005(1)
Cl(4)	4*a*	0.7441(2)	–0.2385(2)	0.7546(1)	0.064(1)	0.067(2)	0.042(1)	0.006(1)	–0.005(1)	0.001(1)
P(1)	4*a*	0.6109(1)	0.3731(2)	0.43109(9)	0.041(1)	0.043(1)	0.036(1)	0.0028(9)	–0.0008(8)	–0.0036(9)
P(2)	4*a*	0.7156(1)	0.1806(2)	0.4541(1)	0.043(1)	0.039(1)	0.040(1)	0.0007(9)	–0.0004(9)	0.0013(9)
P(3)	4*a*	0.7945(1)	–0.1407(2)	0.6414(1)	0.038(1)	0.039(1)	0.043(1)	–0.0041(8)	0.0009(9)	–0.0023(9)
P(4)	4*a*	0.6887(1)	–0.3324(2)	0.6221(1)	0.043(1)	0.036(1)	0.042(1)	–0.0020(8)	–0.0030(9)	–0.0054(9)
N(1)	4*a*	0.8422(4)	0.2665(5)	0.3613(4)	0.043(4)	0.049(4)	0.058(5)	0.008(3)	0.012(4)	–0.004(4)
N(2)	4*a*	0.7557(5)	0.4347(5)	0.3581(3)	0.059(5)	0.041(4)	0.047(4)	–0.008(4)	0.006(4)	0.000(3)
N(3)	4*a*	0.5631(5)	–0.2406(6)	0.7129(3)	0.043(4)	0.060(5)	0.053(5)	–0.009(3)	0.013(3)	0.011(4)
N(4)	4*a*	0.6522(5)	–0.0741(5)	0.7145(3)	0.055(5)	0.039(4)	0.051(5)	0.006(3)	0.005(4)	–0.013(4)
C(1)	4*a*	0.6051(5)	0.4910(6)	0.4047(4)	0.049(5)	0.047(5)	0.051(5)	0.008(4)	0.004(4)	–0.002(4)
C(2)	4*a*	0.6361(6)	0.5655(7)	0.4314(5)	0.063(6)	0.051(6)	0.058(6)	0.003(4)	–0.001(5)	–0.012(5)
C(3)	4*a*	0.6431(7)	0.6503(8)	0.4068(6)	0.065(7)	0.047(6)	0.12(1)	–0.004(5)	0.028(7)	–0.003(7)
C(4)	4*a*	0.6205(8)	0.6605(9)	0.3551(6)	0.079(8)	0.059(7)	0.081(9)	0.016(6)	0.022(7)	0.015(6)
C(5)	4*a*	0.5884(8)	0.5876(9)	0.3298(5)	0.079(8)	0.075(8)	0.074(8)	0.030(7)	0.009(6)	0.016(7)
C(6)	4*a*	0.5794(7)	0.5024(8)	0.3535(5)	0.076(7)	0.064(7)	0.054(6)	0.012(5)	–0.001(5)	0.011(5)
C(7)	4*a*	0.5116(5)	0.3331(7)	0.4144(3)	0.048(5)	0.053(5)	0.041(5)	–0.002(4)	–0.008(4)	0.007(4)
C(8)	4*a*	0.4463(6)	0.3890(8)	0.4267(5)	0.053(6)	0.067(7)	0.066(7)	0.007(5)	0.004(5)	0.011(5)
C(9)	4*a*	0.3708(7)	0.359(1)	0.4190(5)	0.048(6)	0.09(1)	0.086(9)	0.012(6)	0.004(6)	0.030(7)

Table 3. continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(10)	4*a*	0.3577(6)	0.272(1)	0.3984(5)	0.045(6)	0.11(1)	0.069(7)	–0.018(6)	–0.010(5)	0.029(8)
C(11)	4*a*	0.4199(7)	0.2158(8)	0.3880(5)	0.065(7)	0.067(6)	0.058(6)	–0.020(5)	–0.006(5)	–0.005(5)
C(12)	4*a*	0.4974(6)	0.2444(7)	0.3951(4)	0.046(5)	0.071(6)	0.047(5)	–0.005(4)	0.006(4)	0.001(5)
C(13)	4*a*	0.6003(6)	0.3924(6)	0.5012(4)	0.051(5)	0.046(5)	0.047(5)	0.006(4)	0.000(4)	–0.010(4)
C(14)	4*a*	0.5581(6)	0.3149(7)	0.5315(4)	0.057(6)	0.060(6)	0.044(5)	0.010(4)	0.004(4)	0.001(4)
C(15)	4*a*	0.5822(6)	0.2170(7)	0.5189(4)	0.057(6)	0.050(5)	0.046(5)	–0.003(4)	0.007(4)	0.001(4)
C(16)	4*a*	0.6721(6)	0.2003(7)	0.5193(4)	0.058(6)	0.054(6)	0.033(5)	0.011(4)	–0.008(4)	0.003(4)
C(17)	4*a*	0.8096(5)	0.1284(6)	0.4774(4)	0.045(5)	0.046(5)	0.053(5)	–0.003(4)	0.002(4)	0.007(4)
C(18)	4*a*	0.8525(6)	0.1715(7)	0.5167(4)	0.054(6)	0.049(5)	0.060(6)	0.005(4)	–0.011(5)	0.006(5)
C(19)	4*a*	0.9187(6)	0.1329(8)	0.5357(5)	0.055(6)	0.071(7)	0.063(6)	–0.007(5)	–0.007(5)	0.000(5)
C(20)	4*a*	0.9488(6)	0.0513(8)	0.5162(5)	0.048(6)	0.073(7)	0.081(8)	–0.007(5)	–0.005(5)	0.022(6)
C(21)	4*a*	0.9075(6)	0.0080(8)	0.4757(5)	0.052(5)	0.057(6)	0.077(7)	0.012(5)	0.014(5)	0.009(5)
C(22)	4*a*	0.8393(6)	0.0462(7)	0.4561(4)	0.044(5)	0.059(6)	0.061(6)	0.006(4)	0.003(4)	–0.001(5)
C(23)	4*a*	0.6663(6)	0.0751(7)	0.4335(4)	0.046(5)	0.046(5)	0.053(5)	0.002(4)	0.000(4)	–0.010(4)
C(24)	4*a*	0.6515(6)	0.0025(7)	0.4687(5)	0.063(6)	0.052(6)	0.059(6)	–0.006(5)	0.004(5)	0.005(5)
C(25)	4*a*	0.6174(7)	–0.0791(7)	0.4521(5)	0.080(8)	0.042(5)	0.084(9)	–0.012(5)	0.018(7)	0.005(5)
C(26)	4*a*	0.5967(8)	–0.0898(8)	0.4022(6)	0.096(9)	0.048(6)	0.11(1)	–0.023(6)	–0.004(9)	–0.012(7)
C(27)	4*a*	0.609(1)	–0.0203(9)	0.3675(6)	0.16(2)	0.062(8)	0.08(1)	–0.023(9)	–0.01(1)	–0.013(7)
C(28)	4*a*	0.6441(8)	0.0618(7)	0.3829(5)	0.11(1)	0.042(5)	0.054(6)	–0.020(6)	–0.014(6)	0.000(4)
C(29)	4*a*	0.8378(7)	0.1738(8)	0.3364(5)	0.066(7)	0.052(6)	0.077(8)	0.013(5)	0.020(6)	–0.009(6)
C(30)	4*a*	0.9152(6)	0.2732(9)	0.3943(5)	0.035(5)	0.100(8)	0.069(8)	0.006(5)	0.008(5)	0.022(7)
C(31)	4*a*	0.8510(6)	0.3331(8)	0.3177(4)	0.054(6)	0.068(6)	0.054(6)	0.003(5)	0.014(5)	0.005(5)
C(32)	4*a*	0.8356(6)	0.4302(7)	0.3379(5)	0.044(5)	0.064(6)	0.071(7)	–0.003(5)	0.011(5)	0.005(5)
C(33)	4*a*	0.7986(5)	–0.0216(6)	0.6628(3)	0.044(4)	0.041(4)	0.039(5)	–0.005(3)	0.004(4)	–0.004(3)
C(34)	4*a*	0.7663(6)	0.0506(6)	0.6344(4)	0.068(6)	0.042(5)	0.045(5)	–0.003(4)	0.001(4)	–0.003(4)
C(35)	4*a*	0.7595(8)	0.1376(7)	0.6552(5)	0.078(7)	0.041(5)	0.062(6)	–0.003(5)	–0.002(5)	–0.001(5)
C(36)	4*a*	0.7851(6)	0.1554(7)	0.7045(4)	0.067(6)	0.045(5)	0.061(6)	–0.006(4)	0.010(5)	–0.009(5)
C(37)	4*a*	0.8201(6)	0.0880(7)	0.7331(4)	0.070(6)	0.052(6)	0.045(5)	–0.007(5)	0.002(4)	–0.013(4)
C(38)	4*a*	0.8255(6)	–0.0019(7)	0.7128(4)	0.054(5)	0.048(5)	0.055(6)	–0.002(4)	–0.005(4)	0.002(4)
C(39)	4*a*	0.8936(6)	–0.1781(6)	0.6604(4)	0.052(5)	0.044(5)	0.048(6)	0.000(4)	–0.005(4)	–0.009(4)
C(40)	4*a*	0.9581(6)	–0.1217(7)	0.6463(4)	0.046(5)	0.057(6)	0.065(6)	–0.008(4)	–0.004(5)	–0.010(5)
C(41)	4*a*	1.0353(7)	–0.1484(9)	0.6565(5)	0.048(6)	0.093(9)	0.072(8)	–0.003(6)	–0.001(5)	–0.015(6)
C(42)	4*a*	1.0506(7)	–0.231(1)	0.6830(6)	0.049(6)	0.11(1)	0.08(1)	0.014(6)	–0.009(6)	–0.002(8)
C(43)	4*a*	0.9880(7)	–0.2871(9)	0.6961(5)	0.053(6)	0.096(9)	0.066(7)	0.013(6)	–0.001(5)	0.007(6)
C(44)	4*a*	0.9103(6)	–0.2590(8)	0.6851(4)	0.051(6)	0.073(7)	0.051(6)	0.003(5)	–0.003(4)	0.001(5)
C(45)	4*a*	0.8067(6)	–0.1287(6)	0.5709(4)	0.057(5)	0.039(5)	0.040(5)	–0.005(4)	–0.002(4)	0.002(4)
C(46)	4*a*	0.8493(7)	–0.2059(6)	0.5433(4)	0.064(6)	0.051(5)	0.044(5)	–0.010(4)	0.009(4)	–0.010(4)
C(47)	4*a*	0.8241(6)	–0.3036(6)	0.5592(4)	0.052(6)	0.047(5)	0.056(6)	0.002(4)	0.006(4)	–0.014(4)
C(48)	4*a*	0.7352(6)	–0.3182(7)	0.5583(4)	0.055(6)	0.049(5)	0.041(5)	0.000(4)	–0.006(4)	0.002(4)
C(49)	4*a*	0.5945(5)	–0.3817(6)	0.5995(4)	0.045(5)	0.043(5)	0.053(5)	0.005(4)	0.001(4)	–0.008(4)
C(50)	4*a*	0.5537(6)	–0.3403(7)	0.5584(4)	0.048(5)	0.051(5)	0.057(6)	–0.003(4)	–0.013(4)	–0.010(4)
C(51)	4*a*	0.4843(6)	–0.3776(7)	0.5396(5)	0.047(5)	0.060(6)	0.067(6)	0.009(4)	–0.010(5)	–0.019(5)
C(52)	4*a*	0.4527(6)	–0.4531(7)	0.5627(5)	0.047(5)	0.065(7)	0.070(7)	0.001(5)	–0.005(5)	–0.018(5)
C(53)	4*a*	0.4896(6)	–0.4963(7)	0.6039(5)	0.055(6)	0.050(5)	0.082(8)	–0.013(4)	0.012(6)	–0.009(5)
C(54)	4*a*	0.5631(6)	–0.4615(6)	0.6215(4)	0.053(5)	0.042(5)	0.062(6)	–0.001(4)	0.009(5)	–0.007(4)
C(55)	4*a*	0.7370(5)	–0.4384(6)	0.6446(4)	0.047(5)	0.038(4)	0.048(6)	–0.003(4)	–0.005(4)	0.003(4)
C(56)	4*a*	0.7510(7)	–0.5113(7)	0.6105(5)	0.060(6)	0.053(6)	0.065(7)	0.001(5)	–0.001(5)	–0.002(5)
C(57)	4*a*	0.7861(7)	–0.5925(8)	0.6285(7)	0.067(7)	0.046(6)	0.13(1)	0.010(5)	0.018(8)	–0.003(7)
C(58)	4*a*	0.8108(7)	–0.5988(9)	0.6794(7)	0.062(7)	0.062(7)	0.13(1)	0.007(5)	0.006(8)	0.036(8)
C(59)	4*a*	0.7966(7)	–0.5254(8)	0.7135(6)	0.082(8)	0.057(7)	0.077(8)	0.013(6)	–0.005(6)	0.024(6)
C(60)	4*a*	0.7605(7)	–0.4477(8)	0.6952(5)	0.065(6)	0.057(6)	0.062(7)	0.000(5)	0.008(5)	0.003(5)
C(61)	4*a*	0.5638(7)	–0.3304(8)	0.7396(5)	0.068(7)	0.061(6)	0.064(7)	–0.012(5)	0.006(5)	0.010(5)
C(62)	4*a*	0.4919(5)	–0.2370(9)	0.6795(5)	0.029(4)	0.108(9)	0.059(7)	–0.004(5)	0.009(4)	–0.012(6)
C(63)	4*a*	0.5541(7)	–0.1684(8)	0.7548(5)	0.055(6)	0.081(7)	0.058(6)	0.002(5)	0.022(5)	–0.019(6)
C(64)	4*a*	0.5695(6)	–0.0744(7)	0.7342(5)	0.054(6)	0.054(6)	0.088(8)	0.008(5)	0.024(6)	–0.014(6)

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