Crystal structure of tetrachloro-5,11,17,23-tetra-*tert*-butyl-[[*cis*-(*P,P*)-25,26-bis(diphenylphosphinomethoxy)]-(*P,P*)-27,28-bis(diphenylphosphinomethoxy)calix[4]arene}platinum(II)digold(I) hexachloroform hemihydrate, $C_{96}H_{100}Cl_4O_4P_4Au_2Pt \cdot 6CHCl_3 \cdot 1/2H_2O$, an evidence for a weak CH–platinum interaction

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Abstract

$C_{102}H_{107}Au_2Cl_2O_4.5P_4Pt$, monoclinic, $P12_1/c1$ (No. 14), $a = 30.227(1)$ Å, $b = 15.1401(3)$ Å, $c = 26.4571(5)$ Å, $\beta = 102.365(4)^\circ$, $V = 11827(1)$ Å³, $Z = 4$, $R_{gt}(F) = 0.060$, $wR_{ref}(F) = 0.077$, $T = 173$ K.

Source of material

The digold platinum complex was prepared according to the method reported previously, using a one-pot procedure that starts from the corresponding tetraphosphine [1]. Crystals suitable for X-ray diffraction were obtained by slow diffusion of hexane into a chloroform solution of the complex.

Experimental details

Several solvent molecules are disordered. All hydrogen atoms have been calculated except those of the disordered solvent molecules.

Discussion

This work complements a previous study on the coordination chemistry of calix[4]arenes equipped with phosphine ligands [1]. The calixarene unit of the title complex adopts a distorted cone shape with two facing phenolic units almost parallel (interplane angle 7.0°) and the other two approaching perpendicularity (dihedral angle 79.4°). The $PAu(2)Cl$ unit is oriented parallel to the appended phenoxy ring, while the other $PAuCl$ fragment lies roughly orthogonal to the calixarene axis. The two PPt vectors are

pointing away from the cavity (figure). The most striking feature of the structure of this molecule concerns the particular position of the axial H of the $ArCH_2Ar$ group (H(98)) lying between the two phosphines. This hydrogen atom is apically sited with respect to the coordination plane, the $Pt \cdots H$ separation being only ca. 2.61 Å. The NMR signal of H(98) is *strongly* deshielded (7.35 ppm, vs. 3.63 ppm for the adjacent proton), suggesting a weak $Pt \leftrightarrow H-C$ interaction of the “Pregosin type” (so-called “pregostic” interaction) [2]. This situation is similar to that found in a related calixarene, bearing OH substituents instead of “ OCH_2PPh_2AuCl ” units [1]. In the latter case, the $Pt \cdots H_{ax}$ distance is ca. 2.55 Å, while the corresponding H_{ax} signal appears at 7.34 ppm (adjacent H δ 3.76).

Table 1. Data collection and handling.

Crystal:	colorless prism, size $0.16 \times 0.20 \times 0.25$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	42.53 cm^{-1}
Diffractometer, scan mode:	KappaCCD, 119 frames, $\Delta\phi = 1.1^\circ$
$2\theta_{max}$:	61.08°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	63318, 31655
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 3 \sigma(I_{obs})$, 20284
$N(param)_{refined}$:	1306
Programs:	OpenMoleN [3], SIR88 [4]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.2686	−0.1206	0.7865	0.046
H(2)	4e	0.3723	−0.0064	0.7394	0.044
H(3)	4e	0.3686	−0.2520	0.7174	0.106
H(4)	4e	0.3225	−0.2061	0.6961	0.106
H(5)	4e	0.3673	−0.1550	0.6983	0.106
H(6)	4e	0.4159	−0.2080	0.8018	0.086
H(7)	4e	0.4152	−0.1114	0.7819	0.086
H(8)	4e	0.4000	−0.1322	0.8331	0.086
H(9)	4e	0.3476	−0.2878	0.7989	0.104
H(10)	4e	0.3303	−0.2147	0.8309	0.104
H(11)	4e	0.3004	−0.2455	0.7787	0.104
H(12)	4e	0.2260	0.3518	0.6398	0.074
H(13)	4e	0.2143	0.4177	0.5577	0.087
H(14)	4e	0.1454	0.3951	0.4994	0.116
H(15)	4e	0.0882	0.3184	0.5261	0.109
H(16)	4e	0.0995	0.2502	0.6068	0.082
H(17)	4e	0.2446	0.2967	0.7894	0.051
H(18)	4e	0.2549	0.4222	0.8417	0.052

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Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(19)	4e	0.2094	0.5468	0.8184	0.061
H(20)	4e	0.1518	0.5424	0.7446	0.067
H(21)	4e	0.1401	0.4168	0.6924	0.060
H(22)	4e	0.3231	0.2027	0.7114	0.038
H(23)	4e	0.3583	0.1404	0.6952	0.038
H(24)	4e	0.4330	0.1197	0.7512	0.041
H(25)	4e	0.4661	0.2461	0.8877	0.043
H(26)	4e	0.5525	0.1280	0.7803	0.067
H(27)	4e	0.5031	0.1200	0.7488	0.067
H(28)	4e	0.5233	0.2122	0.7660	0.067
H(29)	4e	0.5353	0.0238	0.8457	0.067
H(30)	4e	0.4957	0.0427	0.8731	0.067
H(31)	4e	0.4855	0.0154	0.8152	0.067
H(32)	4e	0.5687	0.1683	0.8697	0.085
H(33)	4e	0.5401	0.2536	0.8565	0.085
H(34)	4e	0.5305	0.1896	0.8987	0.085
H(35)	4e	0.2933	0.3673	0.7403	0.038
H(36)	4e	0.3384	0.3344	0.7277	0.038
H(37)	4e	0.3827	0.5366	0.6809	0.048
H(38)	4e	0.3483	0.6139	0.6073	0.065
H(39)	4e	0.2729	0.6545	0.5950	0.082
H(40)	4e	0.2300	0.6071	0.6534	0.104
H(41)	4e	0.2657	0.5347	0.7303	0.074
H(42)	4e	0.4091	0.3774	0.7137	0.047
H(43)	4e	0.4867	0.3543	0.7270	0.059
H(44)	4e	0.5347	0.4103	0.7999	0.066
H(45)	4e	0.5064	0.4981	0.8569	0.070
H(46)	4e	0.4289	0.5235	0.8451	0.064
H(47)	4e	0.3644	0.3545	0.8836	0.042
H(48)	4e	0.4101	0.3308	0.9204	0.042
H(49)	4e	0.4148	0.1508	0.9322	0.043
H(50)	4e	0.3042	0.0476	0.9697	0.048
H(51)	4e	0.3734	-0.1290	0.9328	0.096
H(52)	4e	0.3293	-0.0753	0.9303	0.096
H(53)	4e	0.3604	-0.0542	0.8922	0.096
H(54)	4e	0.3987	-0.0899	1.0269	0.093
H(55)	4e	0.4003	0.0084	1.0439	0.093
H(56)	4e	0.3540	-0.0373	1.0234	0.093
H(57)	4e	0.4480	-0.0615	0.9680	0.104
H(58)	4e	0.4370	0.0127	0.9268	0.104

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(59)	4e	0.4517	0.0368	0.9849	0.104
H(60)	4e	0.3049	0.4477	0.9364	0.040
H(61)	4e	0.3437	0.3858	0.9635	0.040
H(62)	4e	0.3618	0.4189	1.0969	0.070
H(63)	4e	0.4022	0.5397	1.1376	0.098
H(64)	4e	0.3843	0.6818	1.1064	0.098
H(65)	4e	0.3220	0.7044	1.0389	0.076
H(66)	4e	0.2827	0.5826	0.9976	0.062
H(67)	4e	0.2084	0.3464	1.0472	0.064
H(68)	4e	0.1320	0.3822	1.0227	0.087
H(69)	4e	0.1064	0.4787	0.9522	0.086
H(70)	4e	0.1570	0.5286	0.9048	0.069
H(71)	4e	0.2332	0.4962	0.9290	0.054
H(72)	4e	0.2306	0.2276	0.9276	0.042
H(73)	4e	0.2394	0.1743	0.9788	0.042
H(74)	4e	0.2222	0.0171	0.9766	0.054
H(75)	4e	0.2056	-0.1188	0.8436	0.051
H(76)	4e	0.2494	-0.2260	0.9968	0.106
H(77)	4e	0.2724	-0.1839	0.9556	0.106
H(78)	4e	0.2629	-0.1270	1.0009	0.106
H(79)	4e	0.1891	-0.2789	0.9309	0.118
H(80)	4e	0.1620	-0.2155	0.8896	0.118
H(81)	4e	0.2119	-0.2380	0.8892	0.119
H(82)	4e	0.1703	-0.1873	0.9999	0.129
H(83)	4e	0.1824	-0.0876	1.0036	0.129
H(84)	4e	0.1436	-0.1216	0.9597	0.129
H(85)	4e	0.1796	0.2865	0.8383	0.050
H(86)	4e	0.1648	0.2135	0.8723	0.050
H(87)	4e	0.1211	0.3708	0.8508	0.078
H(88)	4e	0.0806	0.5024	0.8397	0.107
H(89)	4e	0.0267	0.5292	0.7667	0.100
H(90)	4e	0.0074	0.4216	0.7054	0.124
H(91)	4e	0.0491	0.2851	0.7134	0.097
H(92)	4e	0.1194	0.0290	0.8229	0.109
H(93)	4e	0.0877	-0.0513	0.8802	0.121
H(94)	4e	0.0367	0.0118	0.9210	0.132
H(95)	4e	0.0158	0.1631	0.9026	0.148
H(96)	4e	0.0667	0.2288	0.8641	0.143
H(97)	4e	0.1930	-0.0398	0.7618	0.048
H(98)	4e	0.1941	0.0626	0.7608	0.048

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
Pt	4e		0.12427(1)	0.15299(3)	0.71228(1)	0.0360(2)	0.0907(3)	0.0392(2)	-0.0220(2)	0.0137(1)	-0.0177(2)
Au(1)	4e		0.32969(1)	0.53774(2)	0.83707(1)	0.0527(2)	0.0255(1)	0.0346(1)	0.0000(1)	0.0185(1)	-0.0004(1)
Au(2)	4e		0.30366(1)	0.27179(2)	1.06081(1)	0.0440(2)	0.0339(1)	0.0300(1)	0.0025(1)	0.0107(1)	0.0033(1)
Cl(1)	4e	0.50	0.13299(9)	0.0795(2)	0.63666(9)	0.063(1)	0.091(2)	0.049(1)	-0.030(1)	0.021(1)	-0.027(1)
Cl(2)	4e		0.0737(2)	0.0275(4)	0.7219(2)	0.069(3)	0.073(3)	0.077(3)	-0.033(2)	0.026(2)	-0.014(3)
Cl(3)	4e		0.30317(9)	0.6174(2)	0.89816(9)	0.087(1)	0.038(1)	0.050(1)	0.003(1)	0.0377(9)	-0.0040(9)
Cl(4)	4e		0.32278(9)	0.1500(2)	1.11179(9)	0.075(1)	0.043(1)	0.047(1)	0.007(1)	0.015(1)	0.0155(9)
C(1)	4e		0.2571(3)	0.0084(5)	0.7669(3)	0.041(4)	0.033(4)	0.028(3)	-0.008(3)	0.016(3)	-0.006(3)
C(2)	4e		0.2819(3)	-0.0702(5)	0.7743(3)	0.050(4)	0.025(3)	0.034(3)	-0.007(3)	0.016(3)	0.001(3)
C(3)	4e		0.3254(3)	-0.0779(5)	0.7645(3)	0.046(4)	0.022(3)	0.036(3)	-0.001(3)	0.013(3)	0.001(3)
C(4)	4e		0.3431(3)	-0.0027(5)	0.7471(3)	0.035(4)	0.030(3)	0.037(4)	0.000(3)	0.011(3)	-0.003(3)
C(5)	4e		0.3209(3)	0.0781(5)	0.7401(3)	0.038(4)	0.024(3)	0.029(3)	-0.002(3)	0.010(3)	-0.000(3)
C(6)	4e		0.2767(3)	0.0827(5)	0.7494(3)	0.033(4)	0.030(3)	0.026(3)	-0.001(3)	0.009(3)	-0.003(3)
C(7)	4e		0.3516(3)	-0.1660(6)	0.7690(3)	0.069(5)	0.028(4)	0.051(4)	0.004(4)	0.027(4)	0.001(4)
C(8)	4e		0.3526(5)	-0.1977(7)	0.7152(5)	0.127(9)	0.041(5)	0.078(7)	0.028(6)	0.027(7)	-0.007(5)
C(9)	4e		0.4003(4)	-0.1532(7)	0.7993(5)	0.071(7)	0.046(5)	0.077(7)	0.017(5)	0.010(6)	0.008(5)
C(10)	4e		0.3305(4)	-0.2348(7)	0.7969(5)	0.092(7)	0.041(5)	0.116(8)	0.011(5)	0.042(6)	0.031(5)
O(1)	4e		0.2539(2)	0.1624(3)	0.7453(2)	0.034(3)	0.029(2)	0.030(2)	0.004(2)	0.008(2)	0.001(2)
C(11)	4e	0.70	0.2314(3)	0.1880(6)	0.6938(3)	0.006(4)	0.023(4)	0.012(3)	0.004(3)	-0.002(3)	0.003(3)
P(1)	4e		0.17767(7)	0.2452(2)	0.69628(8)	0.033(1)	0.048(1)	0.0293(8)	0.0041(9)	0.0091(7)	0.0005(9)
C(12)	4e		0.1654(4)	0.2945(7)	0.6313(3)	0.050(5)	0.058(5)	0.035(4)	0.018(4)	0.012(3)	0.001(4)
C(13)	4e		0.1978(3)	0.3438(8)	0.6162(3)	0.059(5)	0.079(7)	0.034(4)	0.017(5)	0.016(4)	0.015(4)
C(14)	4e		0.1911(4)	0.3833(8)	0.5672(4)	0.088(6)	0.076(7)	0.045(4)	0.032(5)	0.031(4)	0.017(5)
C(15)	4e		0.1504(5)	0.371(1)	0.5334(4)	0.111(9)	0.12(1)	0.037(5)	0.051(7)	0.018(5)	0.021(6)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(16)	4e		0.1168(4)	0.324(1)	0.5492(5)	0.069(7)	0.12(1)	0.050(6)	0.028(7)	-0.008(6)	0.006(7)
C(17)	4e		0.1230(4)	0.2837(8)	0.5971(4)	0.055(6)	0.090(8)	0.039(5)	0.022(5)	-0.003(4)	0.000(5)
C(18)	4e		0.1908(3)	0.3431(6)	0.7367(3)	0.039(4)	0.048(5)	0.036(4)	0.002(4)	0.014(3)	0.000(4)
C(19)	4e		0.2252(3)	0.3462(6)	0.7804(3)	0.039(4)	0.045(4)	0.036(4)	0.005(4)	0.013(3)	0.003(4)
C(20)	4e		0.2317(3)	0.4213(6)	0.8111(3)	0.034(4)	0.048(5)	0.039(4)	-0.003(4)	0.010(3)	-0.001(4)
C(21)	4e		0.2046(3)	0.4951(6)	0.7977(4)	0.049(5)	0.046(5)	0.047(4)	-0.001(4)	0.017(4)	-0.004(4)
C(22)	4e		0.1707(3)	0.4923(6)	0.7539(4)	0.058(5)	0.046(5)	0.055(5)	0.016(4)	0.021(4)	0.006(4)
C(23)	4e		0.1635(3)	0.4175(6)	0.7228(4)	0.041(5)	0.049(5)	0.048(4)	0.011(4)	0.007(4)	0.010(4)
C(24)	4e		0.3447(3)	0.1575(5)	0.7229(3)	0.037(4)	0.023(3)	0.030(3)	0.001(3)	0.012(3)	0.002(3)
C(25)	4e		0.3810(3)	0.1925(5)	0.7673(3)	0.037(4)	0.023(3)	0.031(3)	0.001(3)	0.012(3)	0.000(3)
C(26)	4e		0.4253(3)	0.1591(5)	0.7759(3)	0.041(4)	0.025(3)	0.032(3)	0.001(3)	0.018(3)	-0.002(3)
C(27)	4e		0.4584(3)	0.1813(5)	0.8191(3)	0.033(4)	0.026(3)	0.037(4)	0.003(3)	0.009(3)	0.003(3)
C(28)	4e		0.4447(3)	0.2330(5)	0.8567(3)	0.038(4)	0.027(3)	0.035(3)	-0.004(3)	0.009(3)	-0.000(3)
C(29)	4e		0.4015(2)	0.2655(5)	0.8504(3)	0.033(3)	0.025(3)	0.027(3)	-0.003(3)	0.012(2)	-0.002(3)
C(30)	4e		0.3711(2)	0.2520(4)	0.8036(3)	0.031(3)	0.023(3)	0.030(3)	0.000(3)	0.015(2)	0.004(3)
C(31)	4e		0.5060(3)	0.1437(5)	0.8259(3)	0.033(4)	0.032(4)	0.046(4)	0.007(3)	0.010(3)	0.001(3)
C(32)	4e		0.5228(3)	0.1517(7)	0.7755(4)	0.044(4)	0.058(5)	0.059(5)	0.008(4)	0.025(3)	0.011(4)
C(33)	4e		0.5056(3)	0.0474(6)	0.8414(4)	0.051(4)	0.039(4)	0.073(5)	0.013(4)	0.031(4)	0.011(4)
C(34)	4e		0.5394(3)	0.1934(8)	0.8664(5)	0.041(5)	0.070(6)	0.080(7)	0.017(5)	0.000(5)	-0.027(5)
O(2)	4e		0.3293(2)	0.2960(3)	0.7945(2)	0.030(2)	0.025(2)	0.033(2)	0.003(2)	0.012(2)	0.004(2)
C(35)	4e		0.3247(2)	0.3586(5)	0.7538(3)	0.030(4)	0.025(3)	0.034(3)	0.002(3)	0.010(3)	-0.002(3)
P(2)	4e		0.35064(7)	0.4682(1)	0.77159(8)	0.041(1)	0.0245(8)	0.0342(8)	-0.0002(8)	0.0145(7)	0.0000(8)
C(36)	4e		0.3266(3)	0.5282(5)	0.7117(3)	0.044(4)	0.028(4)	0.037(4)	-0.001(3)	0.012(3)	0.004(3)
C(37)	4e		0.3516(3)	0.5520(5)	0.6756(3)	0.042(4)	0.030(4)	0.042(4)	-0.006(3)	0.015(3)	-0.000(3)
C(38)	4e		0.3313(3)	0.5979(6)	0.6322(3)	0.071(6)	0.042(5)	0.039(4)	-0.005(4)	0.016(4)	0.004(4)
C(39)	4e		0.2866(4)	0.6207(7)	0.6245(4)	0.083(7)	0.056(6)	0.047(5)	0.016(5)	0.006(5)	0.012(5)
C(40)	4e		0.2615(4)	0.595(1)	0.6598(5)	0.067(7)	0.101(9)	0.070(7)	0.013(7)	0.005(6)	0.029(7)
C(41)	4e		0.2824(3)	0.5497(7)	0.7049(4)	0.050(5)	0.061(6)	0.062(5)	0.019(4)	0.017(4)	0.021(5)
C(42)	4e		0.4107(3)	0.4540(5)	0.7785(3)	0.042(4)	0.022(3)	0.036(3)	-0.000(3)	0.013(3)	0.005(3)
C(43)	4e		0.4287(3)	0.4023(5)	0.7432(3)	0.040(4)	0.035(4)	0.036(4)	-0.002(3)	0.012(3)	0.002(3)
C(44)	4e		0.4747(3)	0.3880(6)	0.7512(4)	0.043(4)	0.039(4)	0.056(5)	-0.001(4)	0.015(4)	0.006(4)
C(45)	4e		0.5031(3)	0.4221(7)	0.7939(4)	0.037(5)	0.057(5)	0.057(5)	0.001(4)	0.006(4)	0.012(5)
C(46)	4e		0.4863(3)	0.4730(7)	0.8279(4)	0.040(5)	0.070(6)	0.046(5)	-0.015(4)	0.000(4)	-0.009(5)
C(47)	4e		0.4402(3)	0.4887(7)	0.8208(4)	0.054(5)	0.050(5)	0.043(4)	-0.007(4)	0.010(4)	-0.003(4)
C(48)	4e		0.3850(3)	0.3080(5)	0.8959(3)	0.041(4)	0.028(3)	0.031(3)	0.003(3)	0.013(3)	-0.003(3)
C(49)	4e		0.3613(3)	0.2366(5)	0.9211(3)	0.038(4)	0.032(3)	0.027(3)	0.005(3)	0.009(3)	-0.000(3)
C(50)	4e		0.3842(3)	0.1564(5)	0.9355(3)	0.030(4)	0.030(3)	0.040(4)	0.003(3)	0.012(3)	0.001(3)
C(51)	4e		0.3635(3)	0.0852(5)	0.9544(3)	0.056(5)	0.034(4)	0.029(3)	0.009(4)	0.015(3)	0.006(3)
C(52)	4e		0.3190(3)	0.0962(5)	0.9576(3)	0.047(4)	0.032(4)	0.034(4)	-0.002(3)	0.013(3)	0.003(3)
C(53)	4e		0.2941(3)	0.1730(5)	0.9445(3)	0.037(4)	0.032(4)	0.027(3)	-0.003(3)	0.009(3)	-0.002(3)
C(54)	4e		0.3174(3)	0.2445(5)	0.9284(3)	0.042(4)	0.029(3)	0.022(3)	0.004(3)	0.007(3)	-0.001(3)
C(55)	4e		0.3861(3)	-0.0054(5)	0.9654(3)	0.058(5)	0.028(4)	0.053(4)	0.017(3)	0.022(4)	0.014(3)
C(56)	4e		0.3598(4)	-0.0724(7)	0.9264(5)	0.108(8)	0.036(5)	0.084(7)	0.016(5)	0.038(6)	-0.001(5)
C(57)	4e		0.3847(4)	-0.0337(7)	1.0200(4)	0.091(7)	0.063(6)	0.065(5)	0.023(5)	0.029(5)	0.037(5)
C(58)	4e		0.4353(4)	-0.0043(7)	0.9608(5)	0.079(6)	0.059(6)	0.116(8)	0.038(5)	0.054(5)	0.032(6)
O(3)	4e		0.2944(2)	0.3241(3)	0.9180(2)	0.038(3)	0.030(2)	0.028(2)	0.006(2)	0.008(2)	-0.001(2)
C(59)	4e		0.3118(3)	0.3926(5)	0.9534(3)	0.036(4)	0.026(3)	0.031(3)	0.004(3)	0.012(3)	-0.002(3)
P(3)	4e		0.28838(7)	0.3929(1)	1.01242(7)	0.038(1)	0.0314(9)	0.0275(8)	0.0035(8)	0.0105(7)	0.0002(8)
C(60)	4e		0.3171(3)	0.4900(6)	1.0445(3)	0.036(4)	0.041(4)	0.033(3)	0.001(3)	0.015(3)	-0.007(3)
C(61)	4e		0.3534(4)	0.4770(6)	1.0851(4)	0.055(6)	0.046(5)	0.055(5)	0.006(4)	-0.001(5)	-0.006(4)
C(62)	4e		0.3778(4)	0.5487(8)	1.1088(5)	0.073(8)	0.066(7)	0.077(7)	-0.007(6)	-0.005(6)	-0.016(6)
C(63)	4e		0.3668(4)	0.6330(8)	1.0908(5)	0.077(7)	0.070(6)	0.077(7)	-0.024(6)	0.015(6)	-0.035(5)
C(64)	4e		0.3306(4)	0.6463(7)	1.0505(4)	0.081(7)	0.041(5)	0.056(5)	-0.006(5)	0.020(5)	-0.009(4)
C(65)	4e		0.3068(3)	0.5738(6)	1.0268(3)	0.073(6)	0.034(4)	0.038(4)	0.003(4)	0.016(4)	-0.001(4)
C(66)	4e		0.2291(3)	0.4183(5)	0.9909(3)	0.039(4)	0.038(4)	0.028(3)	0.005(3)	0.013(3)	0.003(3)
C(67)	4e		0.1982(3)	0.3842(7)	1.0185(3)	0.047(5)	0.058(5)	0.045(4)	0.004(4)	0.020(3)	0.011(4)
C(68)	4e		0.1529(3)	0.4056(9)	1.0040(4)	0.037(5)	0.105(9)	0.063(6)	-0.002(5)	0.021(4)	0.005(6)
C(69)	4e		0.1374(3)	0.4622(9)	0.9615(4)	0.039(5)	0.106(9)	0.050(5)	0.009(6)	0.007(4)	0.007(6)
C(70)	4e		0.1675(3)	0.4926(7)	0.9343(4)	0.044(5)	0.070(6)	0.043(5)	0.015(5)	0.002(4)	0.010(5)
C(71)	4e		0.2128(3)	0.4727(6)	0.9484(3)	0.040(4)	0.045(5)	0.042(4)	-0.000(4)	0.012(3)	-0.000(4)
C(72)	4e		0.2436(3)	0.1754(5)	0.9443(3)	0.039(4)	0.032(4)	0.029(3)	-0.003(3)	0.015(3)	-0.000(3)
C(73)	4e		0.2216(3)	0.0950(6)	0.9152(3)	0.039(4)	0.038(4)	0.035(3)	-0.002(3)	0.017(3)	-0.001(3)
C(74)	4e		0.2185(3)	0.0164(6)	0.9400(3)	0.057(4)	0.038(4)	0.036(3)	-0.004(4)	0.024(3)	-0.001(3)
C(75)	4e		0.2101(3)	-0.0650(6)	0.9144(3)	0.044(4)	0.038(4)	0.046(4)	-0.010(3)	0.021(3)	-0.001(4)
C(76)	4e		0.2080(3)	-0.0641(5)	0.8615(3)	0.045(4)	0.036(4)	0.041(4)	-0.017(3)	0.017(3)	-0.004(3)
C(77)	4e		0.2092(3)	0.0132(6)	0.8337(3)	0.030(4)	0.042(4)	0.037(3)	-0.009(3)	0.015(3)	-0.000(3)
C(78)	4e		0.2122(3)	0.0931(5)	0.8603(3)	0.037(4)	0.032(4)	0.033(3)	-0.008(3)	0.015(3)	-0.003(3)
C(79)	4e		0.2066(3)	-0.1499(6)	0.9459(3)	0.057(5)	0.047(5)	0.050(4)	-0.012(4)	0.026(3)	0.005(4)
C(80)	4e		0.2519(5)	-0.1739(8)	0.9777(5)	0.105(9)	0.063(6)	0.074(7)	-0.016(6)	0.012(6)	0.034(5)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(81)	4e		0.1909(5)	-0.2279(8)	0.9106(5)	0.15(1)	0.051(6)	0.073(7)	-0.028(7)	0.036(7)	0.008(6)
C(82)	4e		0.1724(5)	-0.1352(8)	0.9806(5)	0.157(8)	0.049(6)	0.124(7)	0.007(6)	0.102(5)	0.028(5)
O(4)	4e		0.2084(2)	0.1730(4)	0.8331(2)	0.036(3)	0.035(3)	0.030(2)	-0.001(2)	0.013(2)	0.002(2)
C(83)	4e		0.1710(3)	0.2263(6)	0.8394(3)	0.035(4)	0.050(4)	0.033(3)	-0.002(4)	0.014(3)	-0.004(4)
P(4)	4e		0.11765(8)	0.2091(2)	0.78929(9)	0.032(1)	0.062(1)	0.036(1)	-0.005(1)	0.0119(8)	-0.002(1)
C(84)	4e		0.0893(3)	0.3147(7)	0.7833(3)	0.039(4)	0.069(6)	0.042(4)	0.001(4)	0.015(3)	0.011(4)
C(85)	4e		0.0983(4)	0.3798(7)	0.8204(4)	0.057(6)	0.066(6)	0.058(5)	0.021(5)	0.015(4)	0.005(5)
C(86)	4e		0.0741(4)	0.4584(9)	0.8136(5)	0.065(6)	0.097(8)	0.090(8)	0.039(6)	0.027(5)	0.019(7)
C(87)	4e		0.0419(4)	0.4739(9)	0.7710(5)	0.060(6)	0.085(8)	0.086(8)	0.023(6)	0.015(6)	0.028(6)
C(88)	4e		0.0313(4)	0.412(1)	0.7346(6)	0.049(7)	0.12(1)	0.112(9)	0.015(7)	-0.000(7)	0.054(8)
C(89)	4e		0.0559(4)	0.3288(9)	0.7397(5)	0.054(7)	0.098(9)	0.065(7)	-0.002(6)	-0.004(6)	0.009(7)
C(90)	4e		0.0862(3)	0.1420(7)	0.8275(4)	0.038(4)	0.057(5)	0.055(5)	-0.013(4)	0.016(4)	0.002(4)
C(91)	4e		0.0979(4)	0.0565(8)	0.8391(6)	0.044(5)	0.053(6)	0.16(1)	-0.006(5)	0.032(6)	0.023(7)
C(92)	4e		0.0793(4)	0.0087(8)	0.8736(7)	0.053(7)	0.063(7)	0.16(1)	-0.007(6)	0.013(7)	0.034(8)
C(93)	4e		0.0498(5)	0.046(1)	0.8978(6)	0.095(9)	0.115(9)	0.094(8)	-0.032(7)	0.016(7)	0.046(7)
C(94)	4e		0.0383(4)	0.133(1)	0.8894(5)	0.152(7)	0.094(9)	0.136(7)	-0.037(7)	0.119(4)	-0.013(7)
C(95)	4e		0.0642(3)	0.1666(8)	0.8595(4)	0.128(5)	0.162(7)	0.068(4)	-0.125(4)	0.080(3)	-0.081(4)
C(96)	4e		0.2090(3)	0.0112(6)	0.7767(3)	0.043(4)	0.042(4)	0.029(3)	-0.014(3)	0.013(3)	-0.006(3)
C(97)	4e		0.7979(4)	0.0974(8)	0.3826(4)	0.070(6)	0.082(7)	0.058(5)	-0.027(5)	0.026(4)	-0.024(5)
Cl(5)	4e		0.8058(1)	0.1430(3)	0.4450(1)	0.103(2)	0.121(2)	0.073(2)	-0.037(2)	0.030(1)	-0.050(2)
Cl(6)	4e		0.7472(1)	0.0385(2)	0.3674(1)	0.076(1)	0.074(2)	0.053(1)	-0.021(1)	0.025(1)	-0.015(1)
Cl(7)	4e		0.8004(2)	0.1760(3)	0.3365(2)	0.197(4)	0.146(3)	0.093(3)	-0.100(2)	0.020(3)	0.010(2)
C(98)	4e		0.5791(4)	0.135(1)	0.5345(5)	0.066(7)	0.14(1)	0.087(7)	0.021(7)	0.024(6)	0.066(7)
Cl(8)	4e		0.5704(2)	0.1853(3)	0.5898(2)	0.122(3)	0.070(2)	0.129(3)	0.016(2)	0.021(2)	-0.012(2)
Cl(9)	4e		0.5466(2)	0.1813(4)	0.4797(2)	0.210(3)	0.343(5)	0.130(2)	0.176(3)	0.103(2)	0.164(2)
Cl(10)	4e		0.5669(2)	0.0207(3)	0.5374(2)	0.156(4)	0.078(2)	0.091(2)	-0.006(2)	0.019(2)	-0.019(2)
C(99)	4e		0.9031(6)	0.051(1)	0.9121(8)	0.08(1)	0.18(2)	0.16(2)	0.01(1)	0.02(1)	0.05(1)
Cl(11)	4e		0.9314(2)	0.0186(4)	0.8665(2)	0.081(3)	0.202(4)	0.231(4)	-0.020(3)	0.016(3)	-0.134(3)
Cl(12)	4e		0.8661(1)	0.1345(4)	0.8918(2)	0.089(2)	0.153(4)	0.142(3)	0.017(3)	0.034(2)	0.018(3)
Cl(13)	4e		0.9421(2)	0.0842(6)	0.9692(2)	0.120(4)	0.309(8)	0.080(3)	-0.007(5)	0.008(3)	0.036(4)
C(100)	4e		0.4326(4)	0.2597(7)	0.5859(4)	0.072(6)	0.050(5)	0.065(6)	-0.011(5)	0.021(5)	0.003(5)
Cl(14)	4e		0.4368(1)	0.1756(2)	0.5420(1)	0.091(2)	0.094(2)	0.054(1)	-0.019(2)	0.017(1)	-0.022(1)
Cl(15)	4e		0.4746(1)	0.3397(2)	0.5869(2)	0.096(2)	0.058(2)	0.140(3)	-0.021(2)	0.040(2)	-0.000(2)
Cl(16)	4e		0.4379(1)	0.2141(2)	0.6480(1)	0.111(2)	0.094(2)	0.050(1)	-0.023(2)	0.020(1)	-0.008(2)
C(101)	4e	0.50	0.8920(2)	0.2422(3)	0.2609(2)	0.91(5)	0.07(1)	0.17(1)	-0.05(2)	0.29(2)	0.01(1)
Cl(17)	4e	0.50	0.1611(2)	-0.2654(3)	0.7662(2)	0.149(5)	0.039(2)	0.059(3)	-0.019(3)	0.022(3)	-0.010(2)
Cl(18)	4e	0.50	0.0960(2)	-0.1323(3)	0.7429(2)	0.125(5)	0.053(3)	0.166(7)	-0.011(4)	0.048(5)	-0.026(4)
Cl(19)	4e	0.50	0.0570(2)	-0.2914(3)	0.7227(2)	0.220(8)	0.132(6)	0.49(2)	-0.124(5)	0.16(1)	-0.12(1)
O(5)	4e	0.50	0.0809(5)	-0.206(2)	0.8123(5)	0.042(7)	0.30(2)	0.023(6)	0.092(8)	-0.002(5)	0.02(1)
C(102)	4e		0.0461(4)	-0.0770(9)	0.5978(6)	0.053(6)	0.080(8)	0.108(9)	-0.002(6)	0.017(6)	-0.012(8)
Cl(20)	4e	0.50	-0.0060(2)	-0.1092(6)	0.5972(4)	0.090(4)	0.090(5)	0.256(8)	-0.020(3)	0.095(4)	0.009(6)
Cl(21)	4e	0.50	0.0798(3)	-0.1628(6)	0.5941(5)	0.098(5)	0.124(6)	0.187(8)	0.038(4)	0.028(5)	-0.040(6)
Cl(22)	4e	0.50	0.0755(3)	-0.0928(8)	0.5576(4)	0.120(4)	0.324(8)	0.268(6)	-0.123(5)	0.124(4)	-0.239(5)
Cl(26)	4e	0.50	0.0434(3)	-0.0054(7)	0.5402(3)	0.150(7)	0.147(6)	0.071(4)	-0.059(5)	-0.015(5)	0.025(5)
Cl(27)	4e	0.50	-0.0064(3)	-0.051(1)	0.5867(5)	0.078(5)	0.43(2)	0.215(9)	0.084(7)	-0.033(6)	-0.180(9)
Cl(28)	4e	0.50	0.0335(7)	-0.189(1)	0.6270(8)	0.30(2)	0.17(1)	0.26(2)	-0.08(1)	-0.03(2)	0.05(1)
C(103)	4e	0.50	-0.024(1)	0.197(1)	0.5815(9)	0.36(3)	0.031(7)	0.06(1)	0.10(1)	0.06(2)	0.012(8)
Cl(23)	4e	0.25	0.0151(3)	0.1942(7)	0.5299(4)	0.032(5)	0.077(6)	0.059(5)	-0.017(4)	-0.003(4)	-0.030(4)
Cl(24)	4e	0.50	-0.0032(2)	0.3123(4)	0.5960(3)	0.073(3)	0.046(3)	0.149(6)	0.013(3)	0.033(3)	0.017(3)
Cl(25)	4e	0.50	-0.0731(3)	0.1779(5)	0.5705(3)	0.100(5)	0.106(5)	0.090(4)	-0.034(4)	0.020(4)	-0.021(4)
Cl(29)	4e	0.25	0.0038(3)	0.1404(6)	0.6301(4)	0.044(5)	0.035(4)	0.077(6)	0.002(4)	-0.004(5)	0.000(4)
Cl(2a)	4e	0.50	0.0549(1)	0.0854(3)	0.7057(2)	0.035(2)	0.047(2)	0.046(2)	-0.015(2)	0.011(2)	-0.006(2)
C(11a)	4e	0.30	0.2497(8)	0.233(1)	0.7282(7)	0.05(1)	0.011(8)	0.011(7)	-0.017(8)	0.005(8)	-0.005(7)

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