Crystal structure of 5,11,17,23-tetra-*tert*-butyl-25,26-bis(diphenylphosphinoylmethoxy)-27,28-bis(benzoylmethoxy)calix[4]arene — ethyl acetate — hexane (1:1:0.5), $C_{86}H_{90}O_8P_2 \cdot C_4H_8O_2 \cdot 0.5C_6H_{14}$, *a new cavity-shaped ligand with eight oxygen donors*

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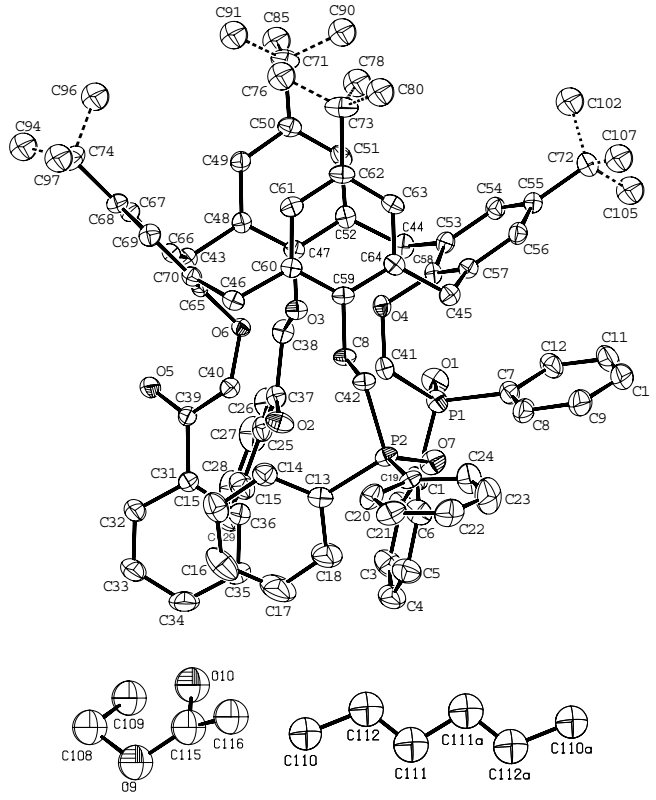
Received April 24, 2003, accepted in revised form and available on-line September 8, 2003; CCDC-No. 1267/1077



Abstract

$C_{93}H_{105}O_{10}P_2$, triclinic, $P\bar{1}$ (No. 2), a = 10.4556(2) Å, b = 15.1838(2) Å, c = 26.8979(5) Å, α = 103.1431(9)°, β = 91.4030(7)°, γ = 95.4738(7)°, V = 4134.7 Å^3, Z = 2, $R_{gt}(F)$ = 0.111, $wR_{ref}(F^2)$ = 0.285, T = 173 K.

Source of material

The title compound was prepared by treatment of 5,11,17,23-tetra-*tert*-butyl-25,26-bis(diphenylphosphinoylmethoxy)-27,28-dihydroxy-calix[4]arene [1] with K_2CO_3/bromoacetophenone (yield, 57% after work-up; mp 401 K – 402 K). Analysis: found – C 78.52%; H 7.08%; calculated for $C_{86}H_{90}O_8P_2$ – C 78.63%; H 6.91%. The ^{1}H and ^{13}C NMR data are consistent with a cone conformation. The chemical shift of the phosphorus signal (25.3 ppm) is typical for a phosphine oxide. More details of the sample synthesis and characteriation data are included in the deposited CIF-file. Crystals suitable for X-ray diffraction were obtained by cooling a solution of the compound dissolved in an ethyl acetate-hexane mixture.

Experimental details

The relatively high R values may be assigned to disordered *tert*-butyl groups. This disorder was inferred from Fourier difference peaks and is described as a rigid rotation of the sets of C atoms C85/C90/C91, C102/C105/C107 and C94/C96/C97 about the C50/C71, C55/C72 and C68/C74 axes, respectively. All C—C distances within the *tert*-butyl groups were fixed at 1.55 Å.

Discussion

The calix[4]arene core of the title compound adopts a typically flattened cone conformation, with two facing phenol rings being almost parallel, the other two approaching perpendicularity (interplane angles 1.5(5)° and 89.1(5)°, respectively). The angles between the calixarene reference plane, defined as the mean plane of the bridging carbon atoms (here C43—C46) and the four phenolic rings (C47—C52, C65—C70, C59—C64, C53—C58) are respectively 101.8(5)°, 130.5(5)°, 100.3(5)°, 140.2(5)°. The distances between the pair of opposite centroids are 5.34(1) Å (C59-C64 and opposite ring) and 7.55(1) Å (C53-C58) and opposite ring). The only X=O vector (X=P or C) that points towards the calixarene axis, is the C37—O2 bond, the other three X=O bonds being roughly oriented tangentially with respect to the "tube" defined by the four lower rim substituents. The separations between proximal O(phenol) atoms are d(O3···O4) = 2.82(1) Å), d(O4···O8) = 3.44(1) Å, d(O8···O6) = 3.44(1) Å and d(O6···O3) = 3.06(1) Å. The bond lengths and angles about the phosphorus atoms may be considered as normal.

Table 1. Data collection and handling.

Crystal:	white prism, size 0.1 × 0.1 × 0.1 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.10 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	18415, 18415
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 10124
$N(param)_{refined}$:	888
Programs:	SIR88 [2], PLATON [3], SHELXL-97 [4]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(2)	2*i*		0.4270	0.6936	0.3240	0.062
H(3)	2*i*		0.4219	0.6057	0.3838	0.080
H(4)	2*i*		0.5647	0.4988	0.3802	0.080
H(5)	2*i*		0.7148	0.4812	0.3181	0.081
H(6)	2*i*		0.7222	0.5693	0.2590	0.068
H(8)	2*i*		0.7001	0.5683	0.1765	0.063
H(9)	2*i*		0.6689	0.4743	0.0950	0.087
H(10)	2*i*		0.4880	0.4822	0.0450	0.101
H(11)	2*i*		0.3429	0.5822	0.0751	0.098
H(12)	2*i*		0.3745	0.6783	0.1554	0.071
H(14)	2*i*		1.1948	0.7289	0.3041	0.060
H(15)	2*i*		1.2284	0.7585	0.3915	0.078
H(16)	2*i*		1.1405	0.6570	0.4363	0.104
H(17)	2*i*		1.0166	0.5253	0.3933	0.125
H(18)	2*i*		0.9763	0.4982	0.3061	0.093
H(20)	2*i*		1.3004	0.5440	0.2523	0.061
H(21)	2*i*		1.4348	0.4387	0.2150	0.068
H(22)	2*i*		1.3684	0.3332	0.1416	0.080
H(23)	2*i*		1.1703	0.3340	0.1044	0.107
H(24)	2*i*		1.0347	0.4413	0.1400	0.078
H(26)	2*i*		0.3480	0.9967	0.3883	0.070
H(27)	2*i*		0.2020	0.9992	0.4521	0.091
H(28)	2*i*		0.2391	0.9272	0.5166	0.104
H(29)	2*i*		0.4097	0.8438	0.5152	0.098
H(30)	2*i*		0.5546	0.8366	0.4507	0.074
H(32)	2*i*		0.9839	0.9186	0.4957	0.058
H(33)	2*i*		0.9437	0.8167	0.5463	0.076
H(34)	2*i*		0.8332	0.6766	0.5125	0.084
H(35)	2*i*		0.7692	0.6353	0.4261	0.073
H(36)	2*i*		0.8129	0.7364	0.3752	0.056
H(38A)	2*i*		0.4766	0.9716	0.3209	0.048
H(38B)	2*i*		0.5746	1.0399	0.3599	0.048
H(40A)	2*i*		1.0555	0.8708	0.3460	0.042
H(40B)	2*i*		0.9176	0.8212	0.3300	0.042
H(41A)	2*i*		0.7334	0.7988	0.2796	0.041
H(41B)	2*i*		0.7770	0.7284	0.2330	0.041
H(42A)	2*i*		1.1959	0.7040	0.2185	0.040
H(42B)	2*i*		1.1055	0.6710	0.1688	0.040
H(43A)	2*i*		0.8147	1.0632	0.3612	0.040
H(43B)	2*i*		0.8397	1.1689	0.3683	0.040
H(44A)	2*i*		0.4433	0.9548	0.1949	0.048
H(44B)	2*i*		0.5054	0.9021	0.2315	0.048
H(45A)	2*i*		0.8897	0.6993	0.1017	0.046
H(45B)	2*i*		0.8587	0.7015	0.1588	0.046
H(46A)	2*i*		1.1665	0.8707	0.2864	0.041
H(46B)	2*i*		1.2868	0.9227	0.2680	0.041
H(49)	2*i*		0.8465	1.2220	0.2818	0.045
H(51)	2*i*		0.591	1.0851	0.1728	0.048
H(54)	2*i*		0.4579	0.8951	0.1057	0.048
H(56)	2*i*		0.7249	0.7333	0.0496	0.047
H(61)	2*i*		1.2080	1.0175	0.2003	0.046
H(63)	2*i*		0.9527	0.8748	0.0929	0.048
H(67)	2*i*		1.0491	1.2362	0.3511	0.045
H(69)	2*i*		1.3247	1.0823	0.2968	0.050
C(75)	2*i*	0.33	1.079(2)	1.1181(8)	0.1484(5)	0.0693(8)
H(75A)	2*i*	0.33	1.0957	1.1692	0.1332	0.104
H(75B)	2*i*	0.33	1.1332	1.1268	0.1788	0.104
H(75C)	2*i*	0.33	0.9904	1.1128	0.1570	0.104
C(76)	2*i*	0.33	1.171(2)	1.1250(7)	0.1385(7)	0.0693(8)
H(76A)	2*i*	0.33	1.1840	1.1632	0.1147	0.104
H(76B)	2*i*	0.33	1.2526	1.1188	0.1539	0.104
H(76C)	2*i*	0.33	1.1163	1.1519	0.1645	0.104
C(77)	2*i*	0.33	1.020(2)	1.030(1)	0.0629(5)	0.0693(8)
H(77A)	2*i*	0.33	1.0513	1.0784	0.0474	0.104
H(77B)	2*i*	0.33	0.9336	1.0388	0.0734	0.104
H(77C)	2*i*	0.33	1.0188	0.9729	0.0385	0.104
C(78)	2*i*	0.33	0.978(1)	1.071(1)	0.1065(8)	0.0693(8)
H(78A)	2*i*	0.33	0.9857	1.1128	0.0846	0.104
H(78B)	2*i*	0.33	0.9568	1.1020	0.1400	0.104
H(78C)	2*i*	0.33	0.9116	1.0228	0.0926	0.104
C(79)	2*i*	0.33	1.127(2)	0.986(1)	0.0531(3)	0.0693(8)
H(79A)	2*i*	0.33	1.1385	1.0321	0.0342	0.104
H(79B)	2*i*	0.33	1.0533	0.9441	0.0392	0.104
H(79C)	2*i*	0.33	1.2023	0.9533	0.0509	0.104

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
C(80)	2*i*	0.33	1.172(2)	0.984(1)	0.0608(4)	0.0693(8)
H(80A)	2*i*	0.33	1.1865	1.0258	0.0394	0.104
H(80B)	2*i*	0.33	1.1160	0.9316	0.0427	0.104
H(80C)	2*i*	0.33	1.2522	0.9644	0.0699	0.104
C(81)	2*i*	0.33	1.230(1)	1.098(1)	0.1273(8)	0.0693(8)
H(81A)	2*i*	0.33	1.2378	1.1401	0.1058	0.104
H(81B)	2*i*	0.33	1.3040	1.0647	0.1249	0.104
H(81C)	2*i*	0.33	1.2226	1.1299	0.1621	0.104
C(82)	2*i*	0.33	1.2537(6)	1.059(1)	0.1091(8)	0.0693(8)
H(82A)	2*i*	0.33	1.2669	1.1058	0.0906	0.104
H(82B)	2*i*	0.33	1.2961	1.0073	0.0928	0.104
H(82C)	2*i*	0.33	1.2885	1.0814	0.1435	0.104
C(83)	2*i*	0.33	0.974(1)	1.040(1)	0.0866(8)	0.0693(8)
H(83A)	2*i*	0.33	0.9840	1.0837	0.0656	0.104
H(83B)	2*i*	0.33	0.9182	1.0612	0.1136	0.104
H(83C)	2*i*	0.33	0.9379	0.9826	0.0662	0.104
C(84)	2*i*	0.33	0.681(2)	1.3316(8)	0.2307(6)	0.0693(8)
H(84A)	2*i*	0.33	0.6920	1.3820	0.2146	0.104
H(84B)	2*i*	0.33	0.7275	1.3471	0.2632	0.104
H(84C)	2*i*	0.33	0.5913	1.3178	0.2355	0.104
C(85)	2*i*	0.33	0.622(2)	1.307(1)	0.2129(8)	0.0693(8)
H(85A)	2*i*	0.33	0.6313	1.3592	0.1981	0.104
H(85B)	2*i*	0.33	0.6259	1.3272	0.2495	0.104
H(85C)	2*i*	0.33	0.5412	1.2724	0.2015	0.104
C(86)	2*i*	0.33	0.601(1)	1.286(1)	0.1960(8)	0.0693(8)
H(86A)	2*i*	0.33	0.6089	1.3382	0.1810	0.104
H(86B)	2*i*	0.33	0.5746	1.3044	0.2304	0.104
H(86C)	2*i*	0.33	0.5384	1.2406	0.1764	0.104
C(87)	2*i*	0.33	0.669(2)	1.222(1)	0.1416(3)	0.0693(8)
H(87A)	2*i*	0.33	0.6775	1.2744	0.1269	0.104
H(87B)	2*i*	0.33	0.5799	1.2022	0.1431	0.104
H(87C)	2*i*	0.33	0.7111	1.1741	0.1208	0.104
C(88)	2*i*	0.33	0.836(1)	1.325(1)	0.2232(7)	0.0693(8)
H(88A)	2*i*	0.33	0.8400	1.3732	0.2054	0.104
H(88B)	2*i*	0.33	0.9185	1.3018	0.2230	0.104
H(88C)	2*i*	0.33	0.8139	1.3478	0.2578	0.104
C(89)	2*i*	0.33	0.769(2)	1.218(2)	0.1397(3)	0.0693(8)
H(89A)	2*i*	0.33	0.7685	1.2684	0.1239	0.104
H(89B)	2*i*	0.33	0.7069	1.1693	0.1219	0.104
H(89C)	2*i*	0.33	0.8527	1.1971	0.1384	0.104
C(90)	2*i*	0.33	0.723(2)	1.211(2)	0.1375(2)	0.0693(8)
H(90A)	2*i*	0.33	0.7295	1.2607	0.1210	0.104
H(90B)	2*i*	0.33	0.6422	1.1747	0.1276	0.104
H(90C)	2*i*	0.33	0.7920	1.1738	0.1273	0.104
C(91)	2*i*	0.33	0.863(1)	1.308(1)	0.2095(8)	0.0693(8)
H(91A)	2*i*	0.33	0.8642	1.3576	0.1931	0.104
H(91B)	2*i*	0.33	0.9321	1.2721	0.1979	0.104
H(91C)	2*i*	0.33	0.8733	1.3308	0.2459	0.104
C(92)	2*i*	0.33	0.8775(7)	1.276(1)	0.1914(8)	0.0693(8)
H(92A)	2*i*	0.33	0.8864	1.3301	0.1781	0.104
H(92B)	2*i*	0.33	0.9145	1.2282	0.1686	0.104
H(92C)	2*i*	0.33	0.9213	1.2887	0.2244	0.104
C(93)	2*i*	0.33	1.238(2)	1.3479(8)	0.3533(7)	0.0693(8)
H(93A)	2*i*	0.33	1.3019	1.3988	0.3570	0.104
H(93B)	2*i*	0.33	1.2039	1.3464	0.3859	0.104
H(93C)	2*i*	0.33	1.1694	1.3536	0.3301	0.104
C(94)	2*i*	0.33	1.266(2)	1.341(1)	0.3739(6)	0.0693(8)
H(94A)	2*i*	0.33	1.3282	1.3917	0.3749	0.104
H(94B)	2*i*	0.33	1.2673	1.3246	0.4065	0.104
H(94C)	2*i*	0.33	1.1818	1.3559	0.3663	0.104
C(95)	2*i*	0.33	1.301(2)	1.280(1)	0.2783(4)	0.0693(8)
H(95A)	2*i*	0.33	1.3628	1.3317	0.2791	0.104
H(95B)	2*i*	0.33	1.2176	1.2936	0.2687	0.104
H(95C)	2*i*	0.33	1.3251	1.2285	0.2538	0.104
C(96)	2*i*	0.33	1.252(2)	1.313(1)	0.2936(6)	0.0693(8)
H(96A)	2*i*	0.33	1.3135	1.3643	0.2943	0.104
H(96B)	2*i*	0.33	1.1703	1.3329	0.3030	0.104
H(96C)	2*i*	0.33	1.2445	1.2740	0.2598	0.104
C(97)	2*i*	0.33	1.4402(8)	1.241(2)	0.3216(7)	0.0693(8)
H(97A)	2*i*	0.33	1.4923	1.2972	0.3247	0.104
H(97B)	2*i*	0.33	1.4445	1.2037	0.2877	0.104
H(97C)	2*i*	0.33	1.4714	1.2092	0.3459	0.104
C(98)	2*i*	0.33	1.377(2)	1.260(1)	0.2839(5)	0.0693(8)
H(98A)	2*i*	0.33	1.4362	1.3142	0.2902	0.104

Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(98B)	2*i*	0.33	1.3198	1.2588	0.2554	0.104
H(98C)	2*i*	0.33	1.4245	1.2076	0.2765	0.104
C(99)	2*i*	0.33	1.403(1)	1.256(1)	0.3737(6)	0.0693(8)
H(99A)	2*i*	0.33	1.4617	1.3101	0.3793	0.104
H(99B)	2*i*	0.33	1.4481	1.2037	0.3627	0.104
H(99C)	2*i*	0.33	1.3617	1.2529	0.4049	0.104
C(100)	2*i*	0.33	1.4396(8)	1.240(2)	0.3416(8)	0.0693(8)
H(10A)	2*i*	0.33	1.4952	1.2958	0.3460	0.104
H(10B)	2*i*	0.33	1.4651	1.1964	0.3129	0.104
H(10C)	2*i*	0.33	1.4456	1.2171	0.3718	0.104
C(101)	2*i*	0.33	1.297(2)	1.319(1)	0.3868(3)	0.0693(8)
H(10D)	2*i*	0.33	1.3530	1.3736	0.3897	0.104
H(10E)	2*i*	0.33	1.3246	1.2861	0.4110	0.104
H(10F)	2*i*	0.33	1.2106	1.3338	0.3935	0.104
C(102)	2*i*	0.50	0.520(1)	0.8982(5)	0.0036(5)	0.0693(8)
H(10A)	2*i*	0.50	0.4847	0.8924	–0.0304	0.104
H(10B)	2*i*	0.50	0.6053	0.9286	0.0070	0.104
H(10C)	2*i*	0.50	0.4666	0.9330	0.0277	0.104
C(103)	2*i*	0.50	0.6224(9)	0.8531(8)	–0.0137(4)	0.0693(8)
H(10D)	2*i*	0.50	0.5917	0.8465	–0.0485	0.104
H(10E)	2*i*	0.50	0.7030	0.8278	–0.0133	0.104
H(10F)	2*i*	0.50	0.6341	0.9165	0.0031	0.104
C(104)	2*i*	0.50	0.3932(7)	0.8435(8)	0.0129(5)	0.0693(8)
H(10G)	2*i*	0.50	0.3645	0.8375	–0.0220	0.104
H(10H)	2*i*	0.50	0.4043	0.9067	0.0301	0.104
H(10I)	2*i*	0.50	0.3303	0.8118	0.0297	0.104
C(105)	2*i*	0.50	0.3849(6)	0.7536(8)	0.0104(5)	0.0693(8)
H(10J)	2*i*	0.50	0.3481	0.7473	–0.0234	0.104
H(10K)	2*i*	0.50	0.3331	0.7886	0.0348	0.104
H(10L)	2*i*	0.50	0.3879	0.6945	0.0173	0.104
C(106)	2*i*	0.50	0.496(1)	0.7012(4)	–0.0139(4)	0.0693(8)

Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(10M)	2*i*	0.50	0.4636	0.6972	–0.0481	0.104
H(10N)	2*i*	0.50	0.4341	0.6709	0.0038	0.104
H(10O)	2*i*	0.50	0.5748	0.6729	–0.0151	0.104
C(107)	2*i*	0.50	0.605(1)	0.7505(7)	–0.0275(4)	0.0693(8)
H(10P)	2*i*	0.50	0.5654	0.7475	–0.0606	0.104
H(10Q)	2*i*	0.50	0.6099	0.6900	–0.0229	0.104
H(10R)	2*i*	0.50	0.6898	0.7818	–0.0251	0.104
C(110)	2*i*		0.034(2)	0.698(1)	–0.0117(7)	0.236(7)
H(11A)	2*i*		0.1104	0.7402	–0.0053	0.354
H(11B)	2*i*		–0.0304	0.7208	0.0112	0.354
H(11C)	2*i*		0.0026	0.6904	–0.0464	0.354
C(111)	2*i*		–0.027(3)	0.541(2)	–0.0044(9)	0.27(1)
H(11D)	2*i*		–0.0726	0.5270	–0.0375	0.328
H(11E)	2*i*		–0.0870	0.5613	0.0215	0.328
C(112)	2*i*		0.066(2)	0.607(2)	–0.0030(9)	0.267(9)
H(11F)	2*i*		0.1261	0.5840	–0.0283	0.320
H(11G)	2*i*		0.1107	0.6188	0.0301	0.320
C(108)	2*i*		0.821(2)	0.373(1)	0.4226(7)	0.250
H(10S)	2*i*		0.7913	0.4074	0.3989	0.300
H(10T)	2*i*		0.8764	0.3293	0.4054	0.300
C(109)	2*i*		0.889(2)	0.437(1)	0.4716(7)	0.250
H(10U)	2*i*		0.9650	0.4689	0.4627	0.375
H(10V)	2*i*		0.9112	0.4009	0.4950	0.375
H(10W)	2*i*		0.8313	0.4795	0.4873	0.375
O(9)	2*i*		0.715(1)	0.3312(7)	0.4458(4)	0.250
O(10)	2*i*		0.724(1)	0.4667(7)	0.4974(4)	0.250
C(115)	2*i*		0.647(1)	0.3971(7)	0.4803(7)	0.250
C(116)	2*i*		0.5183(9)	0.418(1)	0.4940(7)	0.250
H(11H)	2*i*		0.4571	0.3682	0.4777	0.375
H(11I)	2*i*		0.5001	0.4718	0.4828	0.375
H(11J)	2*i*		0.5126	0.4290	0.5304	0.375

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}
P(1)	2*i*	0.5560(1)	0.70737(8)	0.23739(4)	0.0275(5)	0.0531(7)	0.0375(6)	0.0040(5)	0.0040(4)	0.0112(5)
P(2)	2*i*	1.0464(1)	0.58551(7)	0.22794(5)	0.0295(5)	0.0329(6)	0.0489(7)	0.0064(4)	0.0052(5)	0.0135(5)
O(1)	2*i*	0.4440(3)	0.7612(2)	0.2478(1)	0.031(2)	0.071(2)	0.058(2)	0.010(2)	0.011(1)	0.022(2)
O(2)	2*i*	0.6419(3)	0.8574(2)	0.3673(1)	0.048(2)	0.061(2)	0.051(2)	0.021(2)	0.012(2)	0.027(2)
O(3)	2*i*	0.6576(3)	0.9628(2)	0.3034(1)	0.035(2)	0.035(2)	0.038(2)	0.010(1)	0.009(1)	0.010(1)
O(4)	2*i*	0.7366(2)	0.8415(2)	0.2165(1)	0.028(1)	0.040(2)	0.033(1)	0.002(1)	–0.001(1)	0.004(1)
O(5)	2*i*	0.9232(3)	0.9891(2)	0.4196(1)	0.071(2)	0.038(2)	0.032(2)	0.005(2)	0.002(2)	0.005(1)
O(6)	2*i*	0.9444(2)	0.9407(2)	0.3136(1)	0.030(1)	0.033(2)	0.031(1)	0.004(1)	0.000(1)	0.010(1)
O(7)	2*i*	0.9089(3)	0.5549(2)	0.2118(1)	0.031(2)	0.053(2)	0.071(2)	0.004(1)	0.004(2)	0.017(2)
O(8)	2*i*	1.0281(2)	0.7571(2)	0.2245(1)	0.032(1)	0.031(1)	0.039(2)	0.011(1)	0.011(1)	0.014(1)
C(1)	2*i*	0.5722(4)	0.6392(3)	0.2842(2)	0.034(2)	0.058(3)	0.033(2)	0.001(2)	0.002(2)	0.011(2)
C(2)	2*i*	0.4847(5)	0.6502(4)	0.3224(2)	0.041(3)	0.071(3)	0.043(3)	0.004(2)	0.003(2)	0.016(2)
C(3)	2*i*	0.4816(5)	0.5978(5)	0.3585(2)	0.053(3)	0.102(5)	0.051(3)	0.009(3)	0.013(3)	0.026(3)
C(4)	2*i*	0.5668(6)	0.5348(4)	0.3564(2)	0.062(3)	0.084(4)	0.059(3)	–0.011(3)	0.002(3)	0.034(3)
C(5)	2*i*	0.6566(6)	0.5241(4)	0.3191(2)	0.070(4)	0.070(4)	0.068(4)	0.013(3)	–0.001(3)	0.029(3)
C(6)	2*i*	0.6605(5)	0.5762(4)	0.2835(2)	0.047(3)	0.071(4)	0.054(3)	0.011(3)	0.005(2)	0.020(3)
C(7)	2*i*	0.5394(4)	0.6317(3)	0.1745(2)	0.037(2)	0.059(3)	0.038(2)	–0.014(2)	–0.001(2)	0.014(2)
C(8)	2*i*	0.6280(5)	0.5712(3)	0.1561(2)	0.064(3)	0.046(3)	0.043(3)	–0.004(2)	0.006(2)	0.003(2)
C(9)	2*i*	0.6097(7)	0.5148(4)	0.1074(2)	0.111(5)	0.055(3)	0.044(3)	–0.007(3)	0.006(3)	0.002(3)
C(10)	2*i*	0.5014(9)	0.5199(5)	0.0776(2)	0.126(6)	0.073(4)	0.045(3)	–0.030(4)	–0.011(4)	0.014(3)
C(11)	2*i*	0.4150(7)	0.5797(5)	0.0955(2)	0.087(5)	0.099(5)	0.055(4)	–0.035(4)	–0.030(3)	0.032(4)
C(12)	2*i*	0.4331(5)	0.6368(4)	0.1437(2)	0.045(3)	0.084(4)	0.049(3)	–0.021(3)	–0.011(2)	0.027(3)
C(13)	2*i*	1.0802(4)	0.6120(3)	0.2963(2)	0.036(2)	0.046(3)	0.048(3)	0.013(2)	0.010(2)	0.018(2)
C(14)	2*i*	1.1571(5)	0.6886(3)	0.3222(2)	0.052(3)	0.047(3)	0.053(3)	0.015(2)	0.003(2)	0.013(2)
C(15)	2*i*	1.1785(6)	0.7059(4)	0.3743(2)	0.065(4)	0.074(4)	0.055(3)	0.027(3)	–0.006(3)	0.005(3)
C(16)	2*i*	1.1260(7)	0.6455(6)	0.4010(2)	0.075(4)	0.145(7)	0.048(3)	0.028(5)	0.015(3)	0.031(4)
C(17)	2*i*	1.0509(8)	0.5668(7)	0.3752(3)	0.109(6)	0.145(8)	0.074(5)	–0.013(6)	0.012(4)	0.066(5)
C(18)	2*i*	1.0274(6)	0.5503(5)	0.3232(2)	0.081(4)	0.090(5)	0.067(4)	–0.009(4)	0.013(3)	0.039(4)
C(19)	2*i*	1.1523(4)	0.5030(3)	0.2009(2)	0.039(2)	0.025(2)	0.050(3)	0.004(2)	0.006(2)	0.013(2)
C(20)	2*i*	1.2728(5)	0.5017(3)	0.2223(2)	0.043(3)	0.036(3)	0.070(3)	0.010(2)	–0.001(2)	0.005(2)
C(21)	2*i*	1.3536(5)	0.4385(3)	0.2000(2)	0.048(3)	0.045(3)	0.084(4)	0.024(2)	0.010(3)	0.020(3)
C(22)	2*i*	1.3145(6)	0.3762(4)	0.1564(2)	0.075(4)	0.048(3)	0.078(4)	0.030(3)	0.015(3)	0.007(3)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(23)	2*i*	1.1967(8)	0.3768(5)	0.1343(3)	0.108(6)	0.075(4)	0.071(4)	0.033(4)	–0.002(4)	–0.020(3)
C(24)	2*i*	1.1145(6)	0.4407(4)	0.1559(2)	0.063(3)	0.066(4)	0.062(3)	0.021(3)	–0.007(3)	0.000(3)
C(25)	2*i*	0.4655(4)	0.9148(3)	0.4123(2)	0.034(2)	0.054(3)	0.039(2)	–0.005(2)	0.002(2)	0.005(2)
C(26)	2*i*	0.3604(5)	0.9649(4)	0.4135(2)	0.041(3)	0.073(4)	0.056(3)	0.012(2)	0.012(2)	0.003(3)
C(27)	2*i*	0.2739(6)	0.9676(5)	0.4521(3)	0.050(3)	0.096(5)	0.069(4)	0.005(3)	0.023(3)	–0.005(4)
C(28)	2*i*	0.2952(7)	0.9236(6)	0.4901(2)	0.064(4)	0.125(6)	0.052(4)	–0.016(4)	0.028(3)	–0.013(4)
C(29)	2*i*	0.3975(7)	0.8745(5)	0.4895(2)	0.078(4)	0.119(6)	0.049(3)	–0.009(4)	0.011(3)	0.028(4)
C(30)	2*i*	0.4843(5)	0.8698(4)	0.4507(2)	0.054(3)	0.086(4)	0.044(3)	0.000(3)	0.008(2)	0.018(3)
C(31)	2*i*	0.9021(4)	0.8380(3)	0.4299(2)	0.036(2)	0.035(2)	0.036(2)	0.006(2)	0.002(2)	0.008(2)
C(32)	2*i*	0.9410(5)	0.8615(3)	0.4817(2)	0.061(3)	0.046(3)	0.038(2)	0.008(2)	0.000(2)	0.009(2)
C(33)	2*i*	0.9163(6)	0.8009(4)	0.5119(2)	0.093(4)	0.063(4)	0.038(3)	0.010(3)	0.003(3)	0.017(3)
C(34)	2*i*	0.8514(7)	0.7171(4)	0.4917(2)	0.101(5)	0.058(4)	0.061(4)	0.004(3)	0.015(3)	0.033(3)
C(35)	2*i*	0.8125(6)	0.6924(3)	0.4400(2)	0.073(4)	0.044(3)	0.067(4)	–0.009(3)	–0.003(3)	0.021(3)
C(36)	2*i*	0.8385(5)	0.7529(3)	0.4097(2)	0.050(3)	0.046(3)	0.042(3)	–0.002(2)	–0.001(2)	0.012(2)
C(37)	2*i*	0.5617(4)	0.9110(3)	0.3720(2)	0.030(2)	0.045(3)	0.035(2)	0.002(2)	0.002(2)	0.006(2)
C(38)	2*i*	0.5598(4)	0.9785(3)	0.3389(2)	0.031(2)	0.044(2)	0.042(2)	0.007(2)	0.010(2)	0.003(2)
C(39)	2*i*	0.9257(4)	0.9093(3)	0.3996(2)	0.033(2)	0.031(2)	0.035(2)	0.002(2)	–0.001(2)	0.007(2)
C(40)	2*i*	0.9649(4)	0.8796(3)	0.3454(2)	0.035(2)	0.034(2)	0.036(2)	0.004(2)	0.004(2)	0.007(2)
C(41)	2*i*	0.7147(4)	0.7716(3)	0.2436(2)	0.027(2)	0.042(2)	0.032(2)	0.008(2)	0.000(2)	0.003(2)
C(42)	2*i*	1.1075(4)	0.6851(3)	0.2058(2)	0.033(2)	0.030(2)	0.041(2)	0.013(2)	0.009(2)	0.012(2)
C(43)	2*i*	0.8479(4)	1.1109(3)	0.3450(2)	0.036(2)	0.034(2)	0.031(2)	0.010(2)	0.002(2)	0.005(2)
C(44)	2*i*	0.5238(4)	0.9364(3)	0.2057(2)	0.027(2)	0.044(2)	0.046(2)	0.010(2)	0.001(2)	0.003(2)
C(45)	2*i*	0.8678(4)	0.7381(3)	0.1337(2)	0.038(2)	0.037(2)	0.038(2)	0.007(2)	0.007(2)	0.005(2)
C(46)	2*i*	1.1941(4)	0.9196(3)	0.2701(2)	0.027(2)	0.042(2)	0.037(2)	0.010(2)	0.005(2)	0.013(2)
C(47)	2*i*	0.6792(4)	1.0300(3)	0.2766(2)	0.033(2)	0.031(2)	0.034(2)	0.013(2)	0.006(2)	0.008(2)
C(48)	2*i*	0.7686(4)	1.1044(3)	0.2962(2)	0.031(2)	0.033(2)	0.034(2)	0.010(2)	0.004(2)	0.004(2)
C(49)	2*i*	0.7868(4)	1.1720(3)	0.2689(2)	0.039(2)	0.030(2)	0.042(2)	0.010(2)	–0.003(2)	0.005(2)
C(50)	2*i*	0.7191(4)	1.1677(3)	0.2228(2)	0.042(2)	0.038(2)	0.043(2)	0.017(2)	0.007(2)	0.013(2)
C(51)	2*i*	0.6351(4)	1.0904(3)	0.2040(2)	0.043(2)	0.045(3)	0.034(2)	0.019(2)	–0.001(2)	0.008(2)
C(52)	2*i*	0.6132(4)	1.0202(3)	0.2292(2)	0.028(2)	0.040(2)	0.037(2)	0.013(2)	0.004(2)	0.004(2)
C(53)	2*i*	0.5788(4)	0.8752(3)	0.1601(2)	0.027(2)	0.035(2)	0.039(2)	0.001(2)	0.001(2)	0.005(2)
C(54)	2*i*	0.5289(4)	0.8651(3)	0.1107(2)	0.033(2)	0.040(2)	0.044(2)	0.002(2)	–0.006(2)	0.007(2)
C(55)	2*i*	0.5804(4)	0.8119(3)	0.0682(2)	0.044(2)	0.035(2)	0.037(2)	–0.000(2)	–0.006(2)	0.007(2)
C(56)	2*i*	0.6879(4)	0.7687(3)	0.0773(2)	0.043(2)	0.037(2)	0.035(2)	–0.001(2)	0.001(2)	0.001(2)
C(57)	2*i*	0.7416(4)	0.7768(3)	0.1261(2)	0.032(2)	0.033(2)	0.036(2)	0.005(2)	0.002(2)	0.007(2)
C(58)	2*i*	0.6844(4)	0.8283(3)	0.1669(2)	0.029(2)	0.038(2)	0.028(2)	0.001(2)	–0.001(2)	0.002(2)
C(59)	2*i*	1.0460(4)	0.8254(3)	0.1973(2)	0.030(2)	0.030(2)	0.037(2)	0.013(2)	0.011(2)	0.012(2)
C(60)	2*i*	1.1306(4)	0.9011(3)	0.2167(2)	0.024(2)	0.037(2)	0.036(2)	0.009(2)	0.007(2)	0.010(2)
C(61)	2*i*	1.1501(4)	0.9664(3)	0.1878(2)	0.034(2)	0.037(2)	0.045(2)	0.006(2)	0.010(2)	0.012(2)
C(62)	2*i*	1.0862(4)	0.9575(3)	0.1410(2)	0.044(2)	0.044(3)	0.046(3)	0.011(2)	0.014(2)	0.022(2)
C(63)	2*i*	0.9979(4)	0.8815(3)	0.1240(2)	0.042(2)	0.049(3)	0.033(2)	0.009(2)	0.006(2)	0.017(2)
C(64)	2*i*	0.9745(4)	0.8153(3)	0.1516(2)	0.032(2)	0.041(2)	0.033(2)	0.012(2)	0.008(2)	0.007(2)
C(65)	2*i*	1.0320(4)	1.0163(3)	0.3187(1)	0.027(2)	0.034(2)	0.026(2)	–0.001(2)	–0.004(2)	0.008(2)
C(66)	2*i*	0.9885(4)	1.1024(3)	0.3352(1)	0.033(2)	0.037(2)	0.027(2)	0.006(2)	0.001(2)	0.008(2)
C(67)	2*i*	1.0769(4)	1.1790(3)	0.3394(2)	0.047(2)	0.030(2)	0.035(2)	0.003(2)	–0.001(2)	0.007(2)
C(68)	2*i*	1.2049(4)	1.1737(3)	0.3269(2)	0.044(2)	0.037(2)	0.042(2)	–0.004(2)	0.003(2)	0.013(2)
C(69)	2*i*	1.2408(4)	1.0875(3)	0.3069(2)	0.033(2)	0.047(3)	0.044(2)	0.000(2)	0.005(2)	0.010(2)
C(70)	2*i*	1.1572(4)	1.0083(3)	0.3013(2)	0.029(2)	0.039(2)	0.035(2)	0.004(2)	0.000(2)	0.011(2)
C(71)	2*i*	0.7330(4)	1.2476(3)	0.1962(2)	0.056(3)	0.051(3)	0.056(3)	0.024(2)	0.011(2)	0.025(2)
C(72)	2*i*	0.5230(4)	0.8025(3)	0.0145(2)	0.057(3)	0.055(3)	0.039(3)	0.002(2)	–0.006(2)	0.010(2)
C(73)	2*i*	1.1076(5)	1.0301(4)	0.1099(2)	0.070(4)	0.073(4)	0.081(4)	0.012(3)	0.014(3)	0.049(3)
C(74)	2*i*	1.2989(5)	1.2587(3)	0.3319(2)	0.062(3)	0.049(3)	0.068(3)	–0.011(3)	0.013(3)	0.013(3)

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