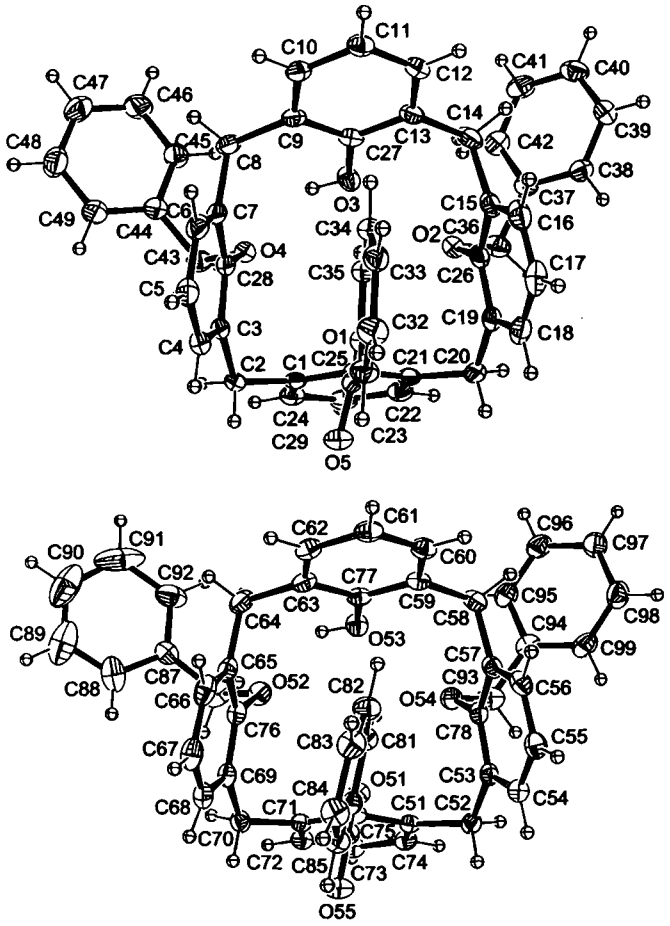
Crystal structure of 25,27-dibenzyloxy-26-benzoyloxy-28-hydroxy-calix[4]arene, $C_{49}H_{40}O_5$

P. Kuhn[I], D. Matt*[,I], L. Toupet[II] and A. Louati*[,III]

[I] Université Louis Pasteur, Laboratoire de Chimie Inorganique Moléculaire, LC3 CNRS-ULP, 1 rue Blaise Pascal, 67008 Strasbourg Cedex, France
[II] Université de Rennes I, UMR CNRS 6509, Laboratoire de Chimie Organométallique et Biologique, Campus de Beaulieu, 35042 Rennes Cedex, France
[III] Ecole Nationale Supérieure de Chimie de Mulhouse, UHA, Laboratoire d'Electrochimie Analytique, 3 rue Alfred Werner, 68093 Mulhouse Cedex, France

Received December 8, 2005, accepted and available on-line January 31, 2006; CCDC no. 1267/1697



Abstract

$C_{49}H_{40}O_5$, triclinic, $P\bar{1}$ (no. 2), a = 10.8865(2) Å, b = 18.2743(3) Å, c = 18.3545(3) Å, α = 85.166(1)°, β = 86.233(1)°, γ = 84.268(1)°, V = 3614.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.054, $wR_{ref}(F^2)$ = 0.148, T = 120 K.

Source of material

The title compound was prepared according to the reported method [1].

Discussion

The unit cell contains two pairs of structurally similar molecules. Each calixarene unit adopts a partial cone conformation in which the phenolic ring being anti-oriented with respect to the other three calixarene rings is the one bearing the benzoyl group. The dihedral angle between the two benzyl-substituted phenolic rings is 39.57(8)° in molecule 1 and 33.95(9)° in molecule 2, while the interplane angles between the other two distal rings of the calixarene are 7.54(8)° in molecule 1 and 21.57(8)° in molecule 2. In molecule 1, the O3/O2 and O3/O4 separations of 2.89 Å and 2.76 Å, respectively, are each consistent with the formation of a hydrogen bond involving the O2 hydroxyl group. A similar situation is observed in the second calixarene. In both molecules the benzoyl plane is nearly perpendicular to the phenoxy ring to which it is appended, and is sandwiched between two facing calixarene rings. The latter orientation may be favored by π-π interactions between the C35 ring and the C5 and C17 aromatic rings (d(C35···C5) = 3.49 Å, d(C35···C17) = 3.50 Å). Both benzyl rings are oriented away from the calixarene core. The carbonyl O atoms of benzoyl groups are involved in C–H···O interactions to aromatic rings of adjacent molecules with small differences apparent between the non-equivalent species.

* Correspondence authors (e-mail: dmatt@chimie.u-strasbg.fr)

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.18 × 0.35 × 0.42 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	0.83 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16513, 16513
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 12766
$N(param)_{refined}$:	974
Programs:	SIR97 [2], SHELXL-97 [3], PLATON [4]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	2*i*	0.4318	0.4841	0.5508	0.033
H(2B)	2*i*	0.4628	0.5496	0.5934	0.033
H(3)	2*i*	0.5338	0.4154	0.8282	0.050
H(4)	2*i*	0.2362	0.4561	0.5887	0.036
H(5)	2*i*	0.0880	0.4214	0.6777	0.042
H(6)	2*i*	0.1290	0.4163	0.7996	0.041
H(8A)	2*i*	0.2468	0.4685	0.8940	0.042
H(8B)	2*i*	0.3862	0.4801	0.8743	0.042
H(10)	2*i*	0.1958	0.3524	0.9521	0.036
H(11)	2*i*	0.2515	0.2328	0.9978	0.038
H(12)	2*i*	0.4480	0.1780	0.9703	0.037
H(14A)	2*i*	0.6604	0.1881	0.9229	0.046
H(14B)	2*i*	0.6993	0.2661	0.8936	0.046
H(16)	2*i*	0.5482	0.1211	0.8310	0.049

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(17)	2*i*	0.5536	0.0869	0.7129	0.053
H(18)	2*i*	0.6537	0.1546	0.6188	0.045
H(20A)	2*i*	0.7628	0.2522	0.5733	0.039
H(20B)	2*i*	0.8690	0.2628	0.6234	0.039
H(22)	2*i*	0.9224	0.3817	0.5901	0.039
H(23)	2*i*	0.8755	0.5073	0.5695	0.043
H(24)	2*i*	0.6724	0.5574	0.5736	0.038
H(31)	2*i*	0.2785	0.2436	0.5601	0.040
H(32)	2*i*	0.1423	0.1724	0.6289	0.043
H(33)	2*i*	0.1407	0.1649	0.7555	0.038
H(34)	2*i*	0.2712	0.2307	0.8134	0.036
H(35)	2*i*	0.4133	0.2976	0.7448	0.033
H(36A)	2*i*	0.9570	0.3275	0.7654	0.044
H(36B)	2*i*	0.9447	0.2484	0.7411	0.044
H(38)	2*i*	0.9433	0.1437	0.8322	0.041
H(39)	2*i*	0.9713	0.0943	0.9510	0.049
H(40)	2*i*	0.9863	0.1714	1.0431	0.050
H(41)	2*i*	0.9761	0.2972	1.0160	0.051
H(42)	2*i*	0.9500	0.3470	0.8978	0.044
H(43A)	2*i*	0.4774	0.5974	0.6964	0.040
H(43B)	2*i*	0.5981	0.5836	0.7397	0.040
H(45)	2*i*	0.5707	0.5815	0.8792	0.046
H(46)	2*i*	0.4722	0.6409	0.9760	0.056
H(47)	2*i*	0.2888	0.7134	0.9601	0.062
H(48)	2*i*	0.2074	0.7303	0.8460	0.067
H(49)	2*i*	0.3046	0.6692	0.7490	0.051
H(52A)	2*i*	1.2947	0.9199	0.5933	0.035
H(52B)	2*i*	1.1970	0.9224	0.5346	0.035
H(53)	2*i*	0.9703	0.7655	0.8162	0.046
H(54)	2*i*	1.0602	1.0230	0.5495	0.036
H(55)	2*i*	0.9154	1.0945	0.6173	0.039
H(56)	2*i*	0.8820	1.0641	0.7410	0.039
H(58A)	2*i*	1.0531	0.9259	0.8509	0.039

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(58B)	2*i*	0.9789	1.0035	0.8574	0.039
H(60)	2*i*	0.7513	1.0122	0.8661	0.038
H(61)	2*i*	0.5702	0.9552	0.8804	0.040
H(62)	2*i*	0.5747	0.8307	0.8639	0.040
H(64A)	2*i*	0.6872	0.7114	0.8522	0.046
H(64B)	2*i*	0.8319	0.7038	0.8527	0.046
H(66)	2*i*	0.5902	0.7479	0.7318	0.045
H(67)	2*i*	0.5976	0.7280	0.6094	0.047
H(68)	2*i*	0.7826	0.6899	0.5498	0.040
H(70A)	2*i*	1.0348	0.6130	0.6072	0.036
H(70B)	2*i*	0.9901	0.6664	0.5414	0.036
H(72)	2*i*	1.2446	0.6187	0.5871	0.034
H(73)	2*i*	1.4201	0.6801	0.5788	0.037
H(74)	2*i*	1.4049	0.8069	0.5823	0.035
H(81)	2*i*	0.8374	0.8908	0.6803	0.034
H(82)	2*i*	0.6462	0.9517	0.7100	0.042
H(83)	2*i*	0.5009	0.9755	0.6230	0.043
H(84)	2*i*	0.5454	0.9385	0.5055	0.043
H(85)	2*i*	0.7388	0.8819	0.4745	0.039
H(86A)	2*i*	1.1126	0.6033	0.7892	0.057
H(86B)	2*i*	1.0537	0.5824	0.7191	0.057
H(88)	2*i*	0.8714	0.5224	0.7288	0.063
H(89)	2*i*	0.7425	0.4484	0.7909	0.097
H(90)	2*i*	0.7277	0.4447	0.9148	0.107
H(91)	2*i*	0.8539	0.5106	0.9805	0.120
H(92)	2*i*	1.0025	0.5823	0.9109	0.068
H(93A)	2*i*	1.3374	0.9204	0.7129	0.046
H(93B)	2*i*	1.3523	0.8477	0.7648	0.046
H(95)	2*i*	1.3036	0.8545	0.8994	0.045
H(96)	2*i*	1.2961	0.9244	0.9987	0.050
H(97)	2*i*	1.2947	1.0516	0.9813	0.052
H(98)	2*i*	1.2997	1.1080	0.8636	0.054
H(99)	2*i*	1.3071	1.0383	0.7640	0.047

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}
O(1)	2*i*	0.5350(1)	0.34129(6)	0.64283(6)	0.0248(6)	0.0266(6)	0.0242(6)	−0.0027(5)	−0.0012(4)	0.0012(4)
O(2)	2*i*	0.7826(1)	0.30246(7)	0.76900(7)	0.0373(7)	0.0302(6)	0.0279(6)	0.0018(5)	−0.0038(5)	0.0017(5)
O(3)	2*i*	0.5630(1)	0.37337(7)	0.84032(7)	0.0293(7)	0.0300(6)	0.0387(7)	−0.0035(5)	0.0043(5)	0.0104(5)
O(5)	2*i*	0.4448(1)	0.33949(7)	0.53545(6)	0.0404(7)	0.0419(7)	0.0219(6)	−0.0083(6)	0.0003(5)	−0.0028(5)
O(4)	2*i*	0.4953(1)	0.49852(7)	0.75177(6)	0.0331(7)	0.0294(6)	0.0255(6)	0.0022(5)	−0.0004(5)	0.0001(5)
C(1)	2*i*	0.5934(2)	0.46244(9)	0.60111(8)	0.0303(9)	0.0285(8)	0.0175(7)	−0.0027(7)	0.0008(6)	−0.0015(6)
C(2)	2*i*	0.4611(2)	0.49646(9)	0.59664(9)	0.0331(9)	0.0263(8)	0.0216(7)	0.0013(7)	0.0008(7)	0.0027(6)
C(3)	2*i*	0.3648(2)	0.47674(9)	0.65686(9)	0.0315(9)	0.0209(7)	0.0246(8)	0.0038(6)	0.0020(7)	0.0021(6)
C(4)	2*i*	0.2522(2)	0.45578(9)	0.6379(1)	0.0335(9)	0.0266(8)	0.0293(8)	0.0039(7)	−0.0015(7)	0.0005(7)
C(5)	2*i*	0.1633(2)	0.4344(1)	0.6912(1)	0.0307(9)	0.0292(9)	0.042(1)	0.0017(7)	0.0010(8)	0.0002(7)
C(6)	2*i*	0.1873(2)	0.4326(1)	0.7642(1)	0.035(1)	0.0274(8)	0.0359(9)	0.0013(7)	0.0110(8)	0.0011(7)
C(7)	2*i*	0.2971(2)	0.45465(9)	0.78605(9)	0.040(1)	0.0209(8)	0.0261(8)	0.0044(7)	0.0075(7)	0.0007(6)
C(8)	2*i*	0.3210(2)	0.4494(1)	0.86692(9)	0.052(1)	0.0274(9)	0.0238(8)	−0.0019(8)	0.0099(8)	−0.0035(7)
C(9)	2*i*	0.3582(2)	0.37110(9)	0.89727(8)	0.0348(9)	0.0283(8)	0.0178(7)	−0.0059(7)	0.0008(6)	−0.0030(6)
C(10)	2*i*	0.2750(2)	0.3307(1)	0.94108(9)	0.0321(9)	0.0346(9)	0.0226(8)	−0.0034(7)	0.0031(7)	−0.0018(7)
C(11)	2*i*	0.3080(2)	0.2589(1)	0.96848(9)	0.035(1)	0.0334(9)	0.0264(8)	−0.0092(7)	0.0029(7)	0.0020(7)
C(12)	2*i*	0.4258(2)	0.2262(1)	0.95189(9)	0.037(1)	0.0306(9)	0.0246(8)	−0.0043(7)	−0.0027(7)	0.0055(7)
C(13)	2*i*	0.5115(2)	0.2645(1)	0.90810(9)	0.0279(9)	0.0371(9)	0.0208(8)	−0.0023(7)	−0.0029(6)	0.0038(7)
C(15)	2*i*	0.6527(2)	0.2064(1)	0.8110(1)	0.0278(9)	0.0321(9)	0.0363(9)	0.0065(7)	−0.0009(7)	0.0080(7)
C(14)	2*i*	0.6405(2)	0.2305(1)	0.8888(1)	0.033(1)	0.050(1)	0.0289(9)	0.0031(8)	−0.0025(7)	0.0142(8)
C(16)	2*i*	0.5914(2)	0.1475(1)	0.7939(1)	0.032(1)	0.034(1)	0.053(1)	0.0047(8)	0.0035(9)	0.0098(9)
C(17)	2*i*	0.5933(2)	0.1273(1)	0.7230(1)	0.035(1)	0.0268(9)	0.068(1)	0.0027(8)	−0.001(1)	−0.0044(9)
C(18)	2*i*	0.6547(2)	0.1675(1)	0.6667(1)	0.035(1)	0.0315(9)	0.046(1)	0.0078(8)	−0.0047(8)	−0.0102(8)
C(19)	2*i*	0.7177(2)	0.2267(1)	0.6811(1)	0.0265(9)	0.0290(8)	0.0348(9)	0.0080(7)	−0.0009(7)	−0.0006(7)
C(20)	2*i*	0.7804(2)	0.2716(1)	0.61869(9)	0.0315(9)	0.0371(9)	0.0257(8)	0.0076(7)	−0.0006(7)	−0.0042(7)
C(21)	2*i*	0.7473(2)	0.3545(1)	0.61119(9)	0.0272(8)	0.0358(9)	0.0186(7)	−0.0011(7)	−0.0010(6)	−0.0022(6)
C(22)	2*i*	0.8402(2)	0.4014(1)	0.59333(9)	0.0248(9)	0.049(1)	0.0247(8)	−0.0037(8)	−0.0003(7)	−0.0040(7)
C(23)	2*i*	0.8122(2)	0.4768(1)	0.5803(1)	0.036(1)	0.047(1)	0.0263(8)	−0.0149(8)	−0.0002(7)	−0.0018(8)
C(24)	2*i*	0.6902(2)	0.5069(1)	0.58343(9)	0.039(1)	0.0318(9)	0.0236(8)	−0.0090(8)	0.0004(7)	−0.0008(7)
C(25)	2*i*	0.6260(2)	0.38699(9)	0.61533(8)	0.0254(8)	0.0293(8)	0.0180(7)	−0.0040(6)	−0.0004(6)	−0.0018(6)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(26)	2*i*	0.7193(2)	0.24383(9)	0.7541(1)	0.0260(9)	0.0282(8)	0.0317(9)	0.0064(7)	−0.0021(7)	0.0026(7)
C(27)	2*i*	0.4758(2)	0.33674(9)	0.88162(8)	0.0300(9)	0.0321(8)	0.0184(7)	−0.0093(7)	−0.0018(6)	0.0015(6)
C(28)	2*i*	0.3837(2)	0.47839(9)	0.73135(9)	0.0301(9)	0.0218(7)	0.0259(8)	0.0039(6)	0.0013(7)	0.0002(6)
C(29)	2*i*	0.4485(2)	0.32188(9)	0.60039(9)	0.0273(8)	0.0247(8)	0.0244(8)	0.0015(6)	−0.0006(6)	−0.0051(6)
C(30)	2*i*	0.3612(2)	0.27710(9)	0.64543(9)	0.0268(8)	0.0235(7)	0.0257(8)	0.0014(6)	−0.0014(6)	−0.0023(6)
C(31)	2*i*	0.2785(2)	0.2400(1)	0.6109(1)	0.039(1)	0.0356(9)	0.0261(8)	−0.0060(8)	−0.0052(7)	−0.0031(7)
C(32)	2*i*	0.1964(2)	0.1979(1)	0.6521(1)	0.037(1)	0.038(1)	0.035(1)	−0.0108(8)	−0.0073(8)	−0.0034(8)
C(33)	2*i*	0.1952(2)	0.1938(1)	0.7278(1)	0.0311(9)	0.0289(8)	0.0342(9)	−0.0018(7)	0.0021(7)	0.0022(7)
C(34)	2*i*	0.2744(2)	0.2322(1)	0.76254(9)	0.035(1)	0.0300(8)	0.0250(8)	−0.0002(7)	0.0002(7)	−0.0009(6)
C(35)	2*i*	0.3584(2)	0.27288(9)	0.72149(9)	0.0304(9)	0.0265(8)	0.0249(8)	−0.0008(7)	−0.0019(7)	−0.0020(6)
C(36)	2*i*	0.9140(2)	0.2835(1)	0.7764(1)	0.033(1)	0.045(1)	0.0305(9)	−0.0067(8)	−0.0009(7)	0.0042(8)
C(37)	2*i*	0.9415(2)	0.2507(1)	0.85234(9)	0.0260(9)	0.0358(9)	0.0280(8)	−0.0052(7)	−0.0008(7)	−0.0009(7)
C(38)	2*i*	0.9494(2)	0.1749(1)	0.8690(1)	0.036(1)	0.0354(9)	0.0300(9)	−0.0018(8)	−0.0019(7)	−0.0057(7)
C(39)	2*i*	0.9663(2)	0.1451(1)	0.9404(1)	0.044(1)	0.038(1)	0.038(1)	0.0017(8)	−0.0049(9)	0.0026(8)
C(40)	2*i*	0.9757(2)	0.1911(1)	0.9953(1)	0.039(1)	0.058(1)	0.0284(9)	−0.0029(9)	−0.0064(8)	0.0027(8)
C(41)	2*i*	0.9694(2)	0.2663(1)	0.9790(1)	0.040(1)	0.056(1)	0.035(1)	−0.0091(9)	−0.0039(8)	−0.0155(9)
C(42)	2*i*	0.9532(2)	0.2962(1)	0.9082(1)	0.035(1)	0.037(1)	0.040(1)	−0.0073(8)	−0.0024(8)	−0.0065(8)
C(43)	2*i*	0.5107(2)	0.5769(1)	0.7424(1)	0.036(1)	0.0349(9)	0.0290(9)	−0.0095(8)	0.0023(7)	−0.0010(7)
C(44)	2*i*	0.4466(2)	0.6181(1)	0.8041(1)	0.035(1)	0.0288(8)	0.0294(9)	−0.0076(7)	−0.0027(7)	−0.0021(7)
C(45)	2*i*	0.4967(2)	0.6108(1)	0.8725(1)	0.042(1)	0.038(1)	0.037(1)	−0.0014(8)	−0.0116(8)	−0.0080(8)
C(46)	2*i*	0.4381(2)	0.6465(1)	0.9305(1)	0.063(2)	0.045(1)	0.033(1)	−0.004(1)	−0.013(1)	−0.0112(9)
C(47)	2*i*	0.3292(2)	0.6902(1)	0.9209(1)	0.059(2)	0.053(1)	0.043(1)	0.003(1)	−0.001(1)	−0.020(1)
C(48)	2*i*	0.2798(2)	0.6996(1)	0.8528(1)	0.048(1)	0.062(2)	0.055(1)	0.017(1)	−0.010(1)	−0.019(1)
C(49)	2*i*	0.3385(2)	0.6632(1)	0.7946(1)	0.043(1)	0.050(1)	0.034(1)	0.0039(9)	−0.0087(8)	−0.0067(9)
O(51)	2*i*	0.9990(1)	0.82425(6)	0.61082(6)	0.0220(6)	0.0249(6)	0.0271(6)	0.0016(4)	−0.0029(4)	−0.0041(4)
O(52)	2*i*	0.9880(1)	0.68143(7)	0.74991(7)	0.0301(7)	0.0403(7)	0.0350(7)	−0.0061(5)	−0.0066(5)	0.0014(5)
O(53)	2*i*	0.9798(1)	0.80887(7)	0.82026(7)	0.0272(6)	0.0275(6)	0.0365(7)	0.0019(5)	−0.0009(5)	−0.0041(5)
O(54)	2*i*	1.1773(1)	0.87934(7)	0.74284(7)	0.0380(7)	0.0260(6)	0.0327(6)	−0.0005(5)	−0.0009(5)	−0.0012(5)
O(55)	2*i*	0.9587(1)	0.82076(8)	0.49274(7)	0.0405(8)	0.0465(8)	0.0251(6)	0.0104(6)	−0.0014(5)	−0.0030(5)
C(51)	2*i*	1.2183(2)	0.82294(9)	0.59311(9)	0.0261(8)	0.0259(8)	0.0217(7)	−0.0006(6)	−0.0003(6)	−0.0028(6)
C(52)	2*i*	1.2129(2)	0.90673(9)	0.5852(1)	0.0286(9)	0.0273(8)	0.0305(8)	−0.0054(7)	0.0029(7)	−0.0024(7)
C(53)	2*i*	1.1213(2)	0.95244(9)	0.63275(9)	0.0249(8)	0.0233(8)	0.0329(9)	−0.0061(6)	0.0010(7)	−0.0046(6)
C(54)	2*i*	1.0492(2)	1.01173(9)	0.5997(1)	0.0301(9)	0.0270(8)	0.0323(9)	−0.0046(7)	−0.0019(7)	−0.0020(7)
C(55)	2*i*	0.9614(2)	1.0545(1)	0.6400(1)	0.0306(9)	0.0274(8)	0.041(1)	−0.0014(7)	−0.0051(8)	−0.0044(7)
C(56)	2*i*	0.9432(2)	1.0368(1)	0.7143(1)	0.0283(9)	0.0304(9)	0.040(1)	−0.0016(7)	−0.0012(7)	−0.0126(7)
C(57)	2*i*	1.0145(2)	0.9786(1)	0.7503(1)	0.0262(9)	0.0306(8)	0.0327(9)	−0.0071(7)	−0.0013(7)	−0.0096(7)
C(58)	2*i*	0.9859(2)	0.9590(1)	0.8314(1)	0.0320(9)	0.0368(9)	0.0294(9)	−0.0027(7)	−0.0021(7)	−0.0116(7)
C(59)	2*i*	0.8664(2)	0.9226(1)	0.84356(9)	0.0290(9)	0.0336(9)	0.0200(7)	0.0003(7)	−0.0015(6)	−0.0037(6)
C(60)	2*i*	0.7537(2)	0.9620(1)	0.86030(9)	0.034(1)	0.0317(9)	0.0274(8)	0.0045(7)	0.0002(7)	−0.0047(7)
C(61)	2*i*	0.6448(2)	0.9281(1)	0.8685(1)	0.0300(9)	0.040(1)	0.0277(8)	0.0063(7)	0.0024(7)	−0.0026(7)
C(62)	2*i*	0.6480(2)	0.8533(1)	0.8588(1)	0.0288(9)	0.040(1)	0.0295(9)	−0.0032(7)	0.0060(7)	−0.0003(7)
C(63)	2*i*	0.7587(2)	0.8116(1)	0.84172(9)	0.0327(9)	0.0325(9)	0.0245(8)	−0.0012(7)	0.0040(7)	0.0024(7)
C(64)	2*i*	0.7616(2)	0.7305(1)	0.8294(1)	0.044(1)	0.0315(9)	0.037(1)	−0.0043(8)	0.0120(8)	0.0039(7)
C(65)	2*i*	0.7706(2)	0.71704(9)	0.7484(1)	0.0318(9)	0.0222(8)	0.040(1)	−0.0050(7)	0.0051(8)	0.0012(7)
C(66)	2*i*	0.6648(2)	0.7310(1)	0.7085(1)	0.0253(9)	0.0264(9)	0.059(1)	−0.0021(7)	0.0043(8)	0.0037(8)
C(67)	2*i*	0.6693(2)	0.7201(1)	0.6350(1)	0.029(1)	0.0314(9)	0.057(1)	−0.0055(7)	−0.0114(9)	0.0062(8)
C(68)	2*i*	0.7804(2)	0.6972(1)	0.5994(1)	0.034(1)	0.0265(8)	0.041(1)	−0.0079(7)	−0.0082(8)	0.0006(7)
C(69)	2*i*	0.8891(2)	0.68500(9)	0.6361(1)	0.0279(9)	0.0203(7)	0.0350(9)	−0.0039(6)	−0.0033(7)	−0.0004(6)
C(70)	2*i*	1.0091(2)	0.66423(9)	0.5925(1)	0.0318(9)	0.0243(8)	0.0337(9)	−0.0032(7)	−0.0006(7)	−0.0052(7)
C(71)	2*i*	1.1201(2)	0.70732(9)	0.59634(9)	0.0281(8)	0.0264(8)	0.0211(7)	−0.0010(6)	−0.0015(6)	−0.0030(6)
C(72)	2*i*	1.2372(2)	0.66971(9)	0.58877(9)	0.0312(9)	0.0258(8)	0.0269(8)	0.0045(7)	−0.0023(7)	−0.0020(6)
C(73)	2*i*	1.3427(2)	0.7064(1)	0.58368(9)	0.0266(9)	0.0334(9)	0.0295(8)	0.0064(7)	−0.0013(7)	−0.0017(7)
C(74)	2*i*	1.3334(2)	0.7826(1)	0.58580(9)	0.0233(8)	0.0355(9)	0.0285(8)	−0.0019(7)	−0.0002(7)	−0.0020(7)
C(75)	2*i*	1.1143(2)	0.78390(9)	0.59899(9)	0.0217(8)	0.0249(8)	0.0228(7)	0.0019(6)	−0.0010(6)	−0.0027(6)
C(76)	2*i*	0.8813(2)	0.69291(9)	0.7112(1)	0.0247(8)	0.0231(8)	0.0351(9)	−0.0033(6)	−0.0027(7)	0.0009(6)
C(77)	2*i*	0.8675(2)	0.84699(9)	0.83465(8)	0.0266(8)	0.0318(8)	0.0196(7)	0.0041(7)	−0.0009(6)	−0.0001(6)
C(78)	2*i*	1.1059(2)	0.93819(9)	0.70886(9)	0.0266(8)	0.0233(8)	0.0327(9)	−0.0061(6)	−0.0013(7)	−0.0042(6)
C(79)	2*i*	0.9272(2)	0.83957(9)	0.55311(9)	0.0282(8)	0.0227(7)	0.0258(8)	−0.0017(6)	−0.0035(6)	0.0010(6)
C(80)	2*i*	0.8075(2)	0.88030(9)	0.57450(9)	0.0253(8)	0.0224(7)	0.0283(8)	−0.0023(6)	−0.0030(6)	0.0002(6)
C(81)	2*i*	0.7794(2)	0.90103(9)	0.64495(9)	0.0287(9)	0.0279(8)	0.0286(8)	−0.0007(7)	−0.0045(7)	−0.0017(6)
C(82)	2*i*	0.6647(2)	0.9371(1)	0.6629(1)	0.035(1)	0.036(1)	0.0311(9)	0.0031(8)	0.0012(8)	−0.0044(7)
C(83)	2*i*	0.5778(2)	0.9513(1)	0.6108(1)	0.0276(9)	0.036(1)	0.043(1)	0.0034(7)	0.0003(8)	−0.0015(8)
C(84)	2*i*	0.6047(2)	0.9297(1)	0.5402(1)	0.032(1)	0.036(1)	0.040(1)	0.0023(8)	−0.0100(8)	−0.0013(8)
C(85)	2*i*	0.7197(2)	0.8952(1)	0.5220(1)	0.034(1)	0.0322(9)	0.0303(9)	0.0013(7)	−0.0057(7)	−0.0029(7)
C(86)	2*i*	1.0355(2)	0.6047(1)	0.7652(1)	0.034(1)	0.053(1)	0.051(1)	0.0069(9)	−0.0006(9)	0.014(1)
C(87)	2*i*	0.9487(2)	0.5592(1)	0.8126(1)	0.0295(9)	0.0303(9)	0.0366(9)	0.0051(7)	−0.0054(7)	−0.0067(7)
C(88)	2*i*	0.8695(2)	0.5190(1)	0.7797(2)	0.043(1)	0.035(1)	0.083(2)	−0.0021(9)	−0.023(1)	−0.004(1)
C(89)	2*i*	0.7912(3)	0.4759(2)	0.8157(2)	0.054(2)	0.054(2)	0.129(3)	−0.012(1)	−0.006(2)	0.024(2)
C(90)	2*i*	0.7844(3)	0.4734(2)	0.8890(3)	0.063(2)	0.046(2)	0.147(4)	0.003(1)	0.038(2)	0.019(2)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(91)	2*i*	0.8606(4)	0.5130(2)	0.9296(2)	0.169(4)	0.058(2)	0.049(2)	0.065(2)	0.040(2)	0.014(1)
C(92)	2*i*	0.9484(3)	0.5568(1)	0.8878(1)	0.099(2)	0.034(1)	0.034(1)	0.009(1)	–0.002(1)	–0.0026(8)
C(93)	2*i*	1.3019(2)	0.8941(1)	0.7561(1)	0.030(1)	0.047(1)	0.037(1)	0.0086(8)	–0.0025(8)	–0.0019(8)
C(94)	2*i*	1.3049(2)	0.9391(1)	0.8207(1)	0.0227(8)	0.041(1)	0.0345(9)	0.0009(7)	–0.0044(7)	0.0031(8)
C(95)	2*i*	1.3024(2)	0.9055(1)	0.8920(1)	0.038(1)	0.034(1)	0.039(1)	–0.0023(8)	–0.0051(8)	0.0063(8)
C(96)	2*i*	1.2983(2)	0.9474(1)	0.9515(1)	0.047(1)	0.044(1)	0.0316(9)	–0.0057(9)	–0.0063(8)	0.0079(8)
C(97)	2*i*	1.2973(2)	1.0234(1)	0.9413(1)	0.050(1)	0.044(1)	0.039(1)	–0.0083(9)	–0.0114(9)	–0.0003(9)
C(98)	2*i*	1.3004(2)	1.0569(1)	0.8709(1)	0.055(1)	0.034(1)	0.046(1)	–0.0090(9)	–0.012(1)	0.0061(8)
C(99)	2*i*	1.3045(2)	1.0151(1)	0.8111(1)	0.038(1)	0.044(1)	0.035(1)	–0.0072(8)	–0.0070(8)	0.0104(8)

Acknowledgment. PK thanks the Ministère de l'Education Nationale, de l'Enseignement Supérieur et de la Recherche for a grant.

References

1. Ho, Z.-C.; Ku, M.-C.; Shu, C.-M.; Lin, L.-G.: *p*-Triallylcalix[4]arene: The Final Member of the *p*-Allylcalix[4]arenes. Tetrahedron **52** (1996) 13189-13200.
2. Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R.: SIR97: a new tool for crystal structure determination and refinement. J. Appl. Crystallogr. **32** (1999) 115-119.
3. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
4. Spek, A. L.: Single-crystal structure validation with the program PLATON. J. Appl. Crystallogr. **36** (2003) 7-13.