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Crystal structure of 5-acetyl-25,27-dipropyloxy-26,28-acetyloxy-calix[4]arene ethyl acetate hemisolvate, $C_{40}H_{42}O_7 \cdot \frac{1}{2}C_4H_8O_2$, *partial cone*

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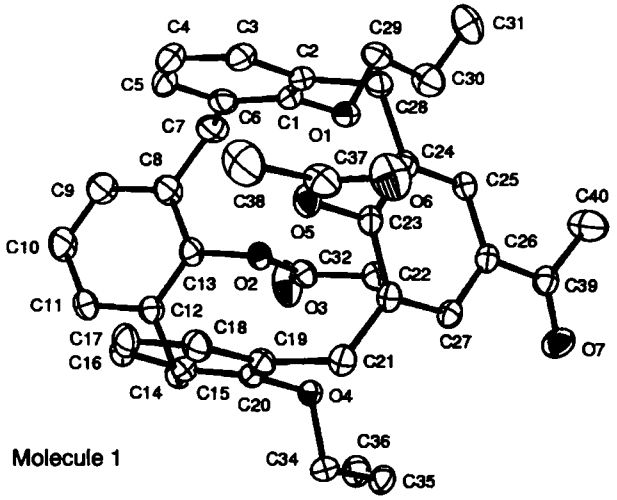

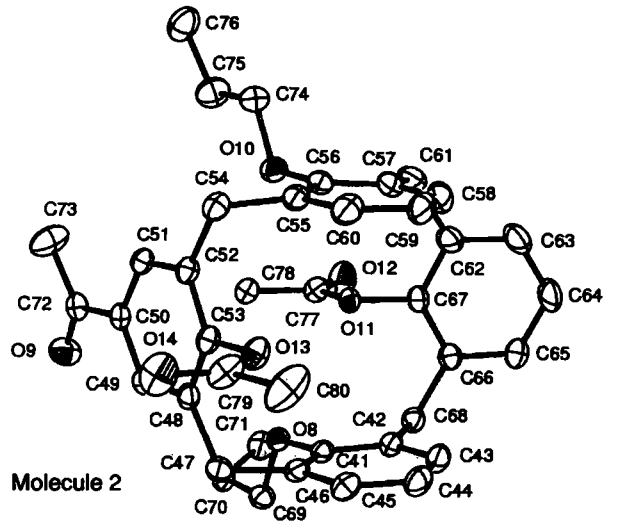

Abstract

$C_{42}H_{46}O_8$, triclinic, $P\bar{1}$ (no. 2), a = 11.721(1) Å, b = 15.937(1) Å, c = 20.137(1) Å, α = 94.468(1)°, β = 95.472(1)°, γ = 102.088(1)°, V = 3642.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.067, $wR_{ref}(F^2)$ = 0.238, T = 150 K.

Source of material

The title compound was prepared from 25,27-dipropyloxy-26,28-dihydroxy-calix[4]arene (cone) using an electrochemical method previously applied for the preparation of other upper rim acetylated calixarenes [1]. The starting compound was electrolyzed in the presence of $ZnBr_2$, generated electrochemically, in an undivided cell fitted with a sacrificial zinc anode using acetonitrile as solvent. After formation of an organozinc species, acetyl chloride was added in excess. After 24 h, the product was extracted with dichloromethane and chromatographied on silica gel using hexane-ethyl acetate (80:20, *v/v*) as eluent (R_f = 0.4; yield ca. 30 %). ^{1}H NMR data are available in the CIF.

Experimental details

A disordered ethyl acetate molecule located near ¼,0,¼ was accounted for using the PLATON squeeze function [2].

Discussion

The unit cell of the title compound contains two pairs of structurally closely related molecules as well as two molecules of AcOEt. The calixarene units adopt a so-called partial cone conformation in which the doubly acetylated aryl ring (Ar) is *anti*-oriented with respect to the other three rings. We note that this conformation is in agreement with the ^{13}C NMR spectrum ($CDCl_3$), which displays two $ArCH_2Ar$ signals, at 30.63 ppm and 37.47 ppm. The latter values are typical for *syn*- and *anti*-oriented Ar rings, respectively. The two facing propylated phenoxy rings are almost parallel (dihedral angles of 3.6° in molecule 1, 4.6° in molecule 2), while the interplane angle between the other two distal aryl rings are 49.7° in molecule 1 and 51.2° in molecule 2. In fact, only the monoacetylated ring is strongly inclined with respect to the calixarene axis. The C39 carbonyl group of molecule 1 lies in the plane defined by the appended aryl ring, thus reflecting the high degree of conjugation between these groups. The same holds for the ArC(O) unit of molecule 2. Interestingly, in both molecules the acetyl group of the monoacetylated phenoxy ring is pointing towards the calixarene axis. This particular orientation is likely to favor weak interactions between the corresponding methyl group and the two propoxy oxygen atoms (d(O10···C78) = 3.334 Å, d(O8···C78) = 3.283 Å).

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.20 × 0.20 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	0.85 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	22959, 16338
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 9690
$N(param)_{refined}$:	857
Programs:	PLATON [2], SIR97 [3], SHELXL-97 [4]

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

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	0.5442	0.2428	0.2465	0.049
H(4)	2*i*	0.4842	0.0992	0.2006	0.057
H(5)	2*i*	0.5412	0.0584	0.0987	0.053
H(7A)	2*i*	0.7107	0.1989	–0.0061	0.056
H(7B)	2*i*	0.5960	0.1222	–0.0201	0.056
H(9)	2*i*	0.6232	–0.0254	–0.0183	0.065
H(10)	2*i*	0.7420	–0.1217	0.0014	0.061
H(11)	2*i*	0.9264	–0.0755	0.0634	0.046
H(14A)	2*i*	1.0849	0.1332	0.1033	0.039
H(14B)	2*i*	1.0807	0.0364	0.1212	0.039
H(16)	2*i*	0.9139	–0.0102	0.2052	0.042
H(17)	2*i*	0.8688	0.0271	0.3119	0.045
H(18)	2*i*	0.9312	0.1685	0.3609	0.041
H(21A)	2*i*	1.0226	0.3145	0.3592	0.037
H(21B)	2*i*	1.1339	0.3322	0.3180	0.037
H(25)	2*i*	0.8425	0.4973	0.1560	0.035
H(27)	2*i*	1.1390	0.4437	0.2472	0.034
H(28A)	2*i*	0.6529	0.4211	0.1725	0.039
H(28B)	2*i*	0.6427	0.3863	0.2446	0.039
H(29A)	2*i*	0.5999	0.4026	0.0608	0.051
H(29B)	2*i*	0.5796	0.3275	0.0002	0.051
H(30A)	2*i*	0.7449	0.3956	–0.0440	0.063
H(30B)	2*i*	0.7802	0.4660	0.0199	0.063
H(31A)	2*i*	0.5733	0.4524	–0.0708	0.090
H(31B)	2*i*	0.6933	0.5204	–0.0753	0.090
H(31C)	2*i*	0.6230	0.5278	–0.0115	0.090
H(33A)	2*i*	1.0487	0.3687	0.0341	0.053
H(33B)	2*i*	1.0437	0.3407	0.1086	0.053
H(33C)	2*i*	0.9302	0.3623	0.0688	0.053
H(34A)	2*i*	1.2649	0.2044	0.2016	0.041
H(34B)	2*i*	1.2643	0.2828	0.2566	0.041
H(35A)	2*i*	1.3935	0.3430	0.1872	0.045
H(35B)	2*i*	1.2763	0.3750	0.1650	0.045
H(36A)	2*i*	1.3448	0.2341	0.0956	0.068
H(36B)	2*i*	1.3589	0.3281	0.0704	0.068
H(36C)	2*i*	1.2307	0.2696	0.0727	0.068
H(38A)	2*i*	0.7628	0.2122	0.4069	0.099
H(38B)	2*i*	0.6531	0.2034	0.3511	0.099
H(38C)	2*i*	0.6618	0.2632	0.4198	0.099
H(40A)	2*i*	1.0851	0.6511	0.1083	0.094
H(40B)	2*i*	0.9781	0.6393	0.1530	0.094
H(40C)	2*i*	0.9708	0.5756	0.0865	0.094

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(43)	2*i*	0.5922	1.0084	0.2954	0.044
H(44)	2*i*	0.6435	0.9729	0.1896	0.047
H(45)	2*i*	0.5865	0.8308	0.1402	0.043
H(47A)	2*i*	0.3792	0.6663	0.1790	0.037
H(47B)	2*i*	0.4929	0.6848	0.1397	0.037
H(49)	2*i*	0.3716	0.5540	0.2499	0.034
H(51)	2*i*	0.6656	0.5045	0.3473	0.032
H(54A)	2*i*	0.8703	0.6196	0.2628	0.038
H(54B)	2*i*	0.8560	0.5815	0.3335	0.038
H(58)	2*i*	0.9467	0.9422	0.4183	0.063
H(59)	2*i*	1.0153	0.9083	0.3176	0.065
H(60)	2*i*	0.9651	0.7665	0.2665	0.054
H(61A)	2*i*	0.7728	0.7933	0.5136	0.062
H(61B)	2*i*	0.8867	0.8700	0.5332	0.062
H(63)	2*i*	0.8581	1.0169	0.5351	0.069
H(64)	2*i*	0.7466	1.1161	0.5075	0.061
H(65)	2*i*	0.5710	1.0724	0.4373	0.048
H(68A)	2*i*	0.4196	0.9610	0.3744	0.038
H(68B)	2*i*	0.4129	0.8638	0.3919	0.038
H(69A)	2*i*	0.2436	0.7169	0.2379	0.041
H(69B)	2*i*	0.2427	0.7958	0.2924	0.041
H(70A)	2*i*	0.2221	0.6240	0.3285	0.045
H(70B)	2*i*	0.1084	0.6598	0.3057	0.045
H(71A)	2*i*	0.2704	0.7285	0.4215	0.063
H(71B)	2*i*	0.1401	0.6728	0.4226	0.063
H(71C)	2*i*	0.1595	0.7674	0.3982	0.063
H(73A)	2*i*	0.5283	0.4283	0.4187	0.087
H(73B)	2*i*	0.5372	0.3662	0.3539	0.087
H(73C)	2*i*	0.4217	0.3488	0.3918	0.087
H(74A)	2*i*	0.9175	0.6696	0.5072	0.049
H(74B)	2*i*	0.9014	0.5956	0.4461	0.049
H(75A)	2*i*	0.7197	0.5284	0.4843	0.062
H(75B)	2*i*	0.7508	0.5978	0.5488	0.062
H(76A)	2*i*	0.8790	0.4695	0.5165	0.088
H(76B)	2*i*	0.8073	0.4747	0.5799	0.088
H(76C)	2*i*	0.9257	0.5448	0.5763	0.088
H(78A)	2*i*	0.5650	0.6343	0.4325	0.051
H(78B)	2*i*	0.4558	0.6556	0.3883	0.051
H(78C)	2*i*	0.4415	0.6263	0.4619	0.051
H(80A)	2*i*	0.8544	0.7392	0.0847	0.099
H(80B)	2*i*	0.8730	0.7940	0.1565	0.099
H(80C)	2*i*	0.7622	0.7957	0.1040	0.099

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*	0.7151(1)	0.32584(7)	0.06863(6)	0.0294(7)	0.0366(6)	0.0413(7)	0.0089(5)	0.0047(6)	0.0062(5)
O(2)	2*i*	0.8953(1)	0.19756(6)	0.07916(5)	0.0356(7)	0.0243(5)	0.0284(6)	0.0061(5)	0.0023(5)	–0.0015(4)
O(3)	2*i*	0.9777(2)	0.21376(9)	–0.01651(7)	0.109(2)	0.0432(8)	0.0497(9)	0.0142(8)	0.0404(9)	0.0004(6)
O(4)	2*i*	1.1202(1)	0.25318(6)	0.18926(5)	0.0265(6)	0.0261(5)	0.0336(6)	0.0037(5)	0.0041(5)	0.0087(4)
O(5)	2*i*	0.8010(1)	0.29223(7)	0.29367(6)	0.0392(8)	0.0294(6)	0.0408(7)	0.0056(5)	0.0147(6)	0.0087(5)
O(6)	2*i*	0.7965(2)	0.39165(9)	0.37826(7)	0.077(1)	0.0544(8)	0.0434(8)	0.0213(8)	0.0250(8)	0.0034(7)
O(7)	2*i*	1.1836(1)	0.55669(8)	0.17017(7)	0.0321(8)	0.0493(8)	0.0551(8)	–0.0009(6)	0.0052(6)	0.0175(6)
C(1)	2*i*	0.6546(2)	0.2640(1)	0.10490(9)	0.0234(9)	0.0284(8)	0.044(1)	0.0031(7)	–0.0011(8)	0.0059(7)
C(2)	2*i*	0.6292(2)	0.2886(1)	0.16871(9)	0.0228(9)	0.0287(8)	0.048(1)	0.0061(7)	0.0054(8)	0.0068(7)
C(3)	2*i*	0.5646(2)	0.2263(1)	0.2036(1)	0.027(1)	0.041(1)	0.055(1)	0.0057(8)	0.0100(9)	0.0135(9)
C(4)	2*i*	0.5297(2)	0.1408(1)	0.1769(1)	0.031(1)	0.041(1)	0.068(1)	–0.0009(8)	0.001(1)	0.016(1)
C(5)	2*i*	0.5620(2)	0.1175(1)	0.1160(1)	0.033(1)	0.0297(9)	0.064(1)	–0.0005(8)	–0.012(1)	0.0059(9)
C(6)	2*i*	0.6247(2)	0.1775(1)	0.0781(1)	0.031(1)	0.0370(9)	0.051(1)	0.0101(8)	–0.0118(9)	–0.0018(8)
C(7)	2*i*	0.6652(2)	0.1484(1)	0.0126(1)	0.042(1)	0.047(1)	0.047(1)	0.0131(9)	–0.0167(9)	–0.0102(9)
C(8)	2*i*	0.7408(2)	0.0833(1)	0.02354(9)	0.050(1)	0.0352(9)	0.038(1)	0.0116(9)	–0.0091(9)	–0.0081(8)
C(9)	2*i*	0.7002(2)	–0.0049(1)	0.0042(1)	0.052(1)	0.042(1)	0.059(1)	0.006(1)	–0.021(1)	–0.0135(9)
C(10)	2*i*	0.7696(2)	–0.0625(1)	0.0173(1)	0.062(2)	0.0307(9)	0.054(1)	0.0065(9)	–0.007(1)	–0.0128(8)
C(11)	2*i*	0.8800(2)	–0.0348(1)	0.05344(9)	0.047(1)	0.0276(8)	0.041(1)	0.0111(8)	0.0062(9)	–0.0008(7)
C(12)	2*i*	0.9227(2)	0.0522(1)	0.07519(8)	0.036(1)	0.0287(8)	0.0305(9)	0.0075(7)	0.0058(7)	0.0005(7)
C(13)	2*i*	0.8529(2)	0.1092(1)	0.05783(8)	0.041(1)	0.0260(8)	0.0284(9)	0.0067(7)	0.0001(8)	–0.0043(6)
C(14)	2*i*	1.0363(2)	0.0828(1)	0.12057(8)	0.035(1)	0.0267(8)	0.0379(9)	0.0094(7)	0.0073(8)	0.0033(7)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(15)	2*i*	1.0116(2)	0.1078(1)	0.19124(8)	0.0279(9)	0.0280(8)	0.0347(9)	0.0068(7)	0.0031(7)	0.0065(7)
C(16)	2*i*	0.9421(2)	0.0472(1)	0.22557(9)	0.036(1)	0.0257(8)	0.043(1)	0.0048(7)	0.0030(8)	0.0052(7)
C(17)	2*i*	0.9138(2)	0.0694(1)	0.28852(9)	0.043(1)	0.0305(8)	0.041(1)	0.0051(8)	0.0099(8)	0.0140(7)
C(18)	2*i*	0.9512(2)	0.1536(1)	0.31757(9)	0.037(1)	0.0341(9)	0.0332(9)	0.0083(8)	0.0084(8)	0.0119(7)
C(19)	2*i*	1.0176(2)	0.2170(1)	0.28439(8)	0.032(1)	0.0290(8)	0.0292(9)	0.0074(7)	0.0004(7)	0.0071(7)
C(20)	2*i*	1.0501(2)	0.19218(9)	0.22230(8)	0.0261(9)	0.0261(8)	0.0288(8)	0.0045(7)	−0.0006(7)	0.0081(6)
C(21)	2*i*	1.0477(2)	0.31070(9)	0.31373(8)	0.035(1)	0.0295(8)	0.0269(8)	0.0033(7)	0.0019(7)	0.0037(6)
C(22)	2*i*	0.9895(2)	0.36764(9)	0.27122(8)	0.033(1)	0.0230(7)	0.0255(8)	0.0020(7)	0.0031(7)	0.0007(6)
C(23)	2*i*	0.8677(2)	0.35586(9)	0.26022(8)	0.032(1)	0.0219(7)	0.0297(8)	0.0042(7)	0.0106(7)	0.0019(6)
C(24)	2*i*	0.8097(2)	0.40052(9)	0.21641(8)	0.0304(9)	0.0235(7)	0.0323(9)	0.0074(7)	0.0093(7)	−0.0019(6)
C(25)	2*i*	0.8790(2)	0.46482(9)	0.18576(8)	0.033(1)	0.0235(7)	0.0306(9)	0.0069(7)	0.0042(7)	0.0009(6)
C(26)	2*i*	1.0010(2)	0.48233(9)	0.19789(8)	0.032(1)	0.0228(7)	0.0294(8)	0.0027(7)	0.0077(7)	0.0010(6)
C(27)	2*i*	1.0557(2)	0.43266(9)	0.23999(8)	0.0288(9)	0.0259(8)	0.0276(8)	0.0021(7)	0.0034(7)	−0.0014(6)
C(28)	2*i*	0.6772(2)	0.3795(1)	0.20205(9)	0.029(1)	0.0297(8)	0.041(1)	0.0085(7)	0.0109(8)	0.0057(7)
C(29)	2*i*	0.6402(2)	0.3699(1)	0.0303(1)	0.039(1)	0.043(1)	0.047(1)	0.0151(9)	0.0003(9)	0.0104(8)
C(30)	2*i*	0.7130(2)	0.4297(1)	−0.0100(1)	0.059(2)	0.056(1)	0.050(1)	0.019(1)	0.016(1)	0.017(1)
C(31)	2*i*	0.6446(3)	0.4877(1)	−0.0450(1)	0.091(2)	0.049(1)	0.048(1)	0.028(1)	0.011(1)	0.012(1)
C(32)	2*i*	0.9592(2)	0.2446(1)	0.03661(9)	0.042(1)	0.0337(9)	0.034(1)	0.0128(8)	0.0079(8)	0.0034(7)
C(33)	2*i*	0.9989(2)	0.3370(1)	0.06443(9)	0.039(1)	0.0301(8)	0.038(1)	0.0082(7)	0.0066(8)	0.0062(7)
C(34)	2*i*	1.2442(2)	0.2613(1)	0.20852(9)	0.0246(9)	0.0353(9)	0.042(1)	0.0056(7)	0.0001(8)	0.0043(7)
C(35)	2*i*	1.3113(2)	0.3237(1)	0.16610(9)	0.030(1)	0.0313(8)	0.051(1)	0.0025(7)	0.0104(8)	0.0023(8)
C(36)	2*i*	1.3115(2)	0.2855(1)	0.0950(1)	0.041(1)	0.040(1)	0.056(1)	0.0070(9)	0.016(1)	0.0076(9)
C(37)	2*i*	0.7721(2)	0.3180(1)	0.35473(9)	0.043(1)	0.049(1)	0.040(1)	0.0198(9)	0.0202(9)	0.0146(9)
C(38)	2*i*	0.7068(2)	0.2427(2)	0.3858(1)	0.074(2)	0.065(1)	0.075(2)	0.025(1)	0.043(1)	0.039(1)
C(39)	2*i*	1.0777(2)	0.5494(1)	0.16473(8)	0.034(1)	0.0286(8)	0.0336(9)	0.0036(7)	0.0080(8)	0.0029(7)
C(40)	2*i*	1.0232(2)	0.6090(2)	0.1247(1)	0.045(1)	0.067(1)	0.088(2)	0.017(1)	0.021(1)	0.051(1)
O(8)	2*i*	0.3846(1)	0.74505(6)	0.30683(5)	0.0247(6)	0.0261(5)	0.0334(6)	0.0034(5)	0.0036(5)	0.0075(4)
O(9)	2*i*	0.3248(1)	0.44006(8)	0.32616(7)	0.0334(8)	0.0504(8)	0.0547(8)	−0.0060(6)	0.0008(7)	0.0191(6)
O(10)	2*i*	0.7831(1)	0.67033(7)	0.43730(6)	0.0304(7)	0.0356(6)	0.0413(7)	0.0098(5)	0.0023(6)	0.0064(5)
O(11)	2*i*	0.5991(1)	0.79863(6)	0.42263(5)	0.0343(7)	0.0239(5)	0.0287(6)	0.0058(5)	0.0016(5)	−0.0023(4)
O(12)	2*i*	0.5044(2)	0.78002(8)	0.51395(7)	0.085(1)	0.0436(7)	0.0472(8)	0.0116(8)	0.0329(9)	−0.0017(6)
O(13)	2*i*	0.7126(1)	0.70915(7)	0.21018(6)	0.0394(8)	0.0293(6)	0.0398(7)	0.0066(5)	0.0165(6)	0.0111(5)
O(14)	2*i*	0.7172(2)	0.61167(9)	0.12435(7)	0.073(1)	0.0566(9)	0.0447(8)	0.0211(8)	0.0293(8)	0.0075(7)
C(41)	2*i*	0.4572(2)	0.80645(9)	0.27560(8)	0.0245(9)	0.0252(7)	0.0312(9)	0.0035(6)	0.0017(7)	0.0087(6)
C(42)	2*i*	0.4942(2)	0.8906(1)	0.30707(8)	0.0297(9)	0.0248(8)	0.0349(9)	0.0052(7)	0.0009(7)	0.0056(7)
C(43)	2*i*	0.5653(2)	0.9512(1)	0.27446(9)	0.039(1)	0.0249(8)	0.047(1)	0.0049(7)	0.0035(9)	0.0070(7)
C(44)	2*i*	0.5977(2)	0.9300(1)	0.21213(9)	0.040(1)	0.0326(9)	0.047(1)	0.0050(8)	0.0100(9)	0.0170(8)
C(45)	2*i*	0.5626(2)	0.8455(1)	0.18260(9)	0.041(1)	0.0340(9)	0.036(1)	0.0106(8)	0.0082(8)	0.0113(7)
C(46)	2*i*	0.4934(2)	0.7821(1)	0.21395(8)	0.0290(9)	0.0274(8)	0.0312(9)	0.0055(7)	0.0000(7)	0.0085(7)
C(47)	2*i*	0.4654(2)	0.6883(1)	0.18459(8)	0.036(1)	0.0324(8)	0.0246(8)	0.0072(7)	0.0019(7)	0.0030(6)
C(48)	2*i*	0.5222(2)	0.63169(9)	0.22828(8)	0.035(1)	0.0224(7)	0.0255(8)	0.0048(7)	0.0033(7)	−0.0007(6)
C(49)	2*i*	0.4548(2)	0.56598(9)	0.25883(8)	0.0287(9)	0.0245(7)	0.0286(8)	0.0020(7)	0.0026(7)	−0.0003(6)
C(50)	2*i*	0.5078(2)	0.51757(9)	0.30227(8)	0.034(1)	0.0204(7)	0.0261(8)	0.0020(7)	0.0063(7)	−0.0007(6)
C(51)	2*i*	0.6300(2)	0.53646(9)	0.31668(8)	0.0299(9)	0.0240(7)	0.0279(8)	0.0079(7)	0.0079(7)	0.0026(6)
C(52)	2*i*	0.7003(2)	0.60104(9)	0.28710(8)	0.0296(9)	0.0237(7)	0.0318(8)	0.0073(7)	0.0076(7)	0.0015(6)
C(53)	2*i*	0.6439(2)	0.64498(9)	0.24186(8)	0.0316(9)	0.0235(7)	0.0301(8)	0.0067(7)	0.0102(7)	0.0035(6)
C(54)	2*i*	0.8324(2)	0.6237(1)	0.30442(9)	0.0281(9)	0.0298(8)	0.041(1)	0.0090(7)	0.0111(8)	0.0073(7)
C(55)	2*i*	0.8752(2)	0.7141(1)	0.34024(9)	0.0198(9)	0.0305(8)	0.050(1)	0.0045(7)	0.0036(8)	0.0076(7)
C(56)	2*i*	0.8444(2)	0.7350(1)	0.40376(9)	0.0220(9)	0.0311(8)	0.046(1)	0.0046(7)	−0.0023(8)	0.0033(7)
C(57)	2*i*	0.8688(2)	0.8202(1)	0.4331(1)	0.029(1)	0.0343(9)	0.060(1)	0.0062(8)	−0.0120(9)	−0.0037(8)
C(58)	2*i*	0.9305(2)	0.8839(1)	0.3994(1)	0.033(1)	0.033(1)	0.086(2)	0.0034(8)	−0.008(1)	−0.005(1)
C(59)	2*i*	0.9690(2)	0.8643(1)	0.3389(1)	0.027(1)	0.0316(9)	0.103(2)	−0.0004(8)	0.000(1)	0.021(1)
C(60)	2*i*	0.9401(2)	0.7795(1)	0.3089(1)	0.026(1)	0.040(1)	0.073(1)	0.0070(8)	0.009(1)	0.0163(9)
C(61)	2*i*	0.8208(2)	0.8452(1)	0.4979(1)	0.047(1)	0.048(1)	0.054(1)	0.018(1)	−0.023(1)	−0.0162(9)
C(62)	2*i*	0.7462(2)	0.9107(1)	0.4855(1)	0.046(1)	0.041(1)	0.047(1)	0.0149(9)	−0.012(1)	−0.0122(8)
C(63)	2*i*	0.7851(2)	0.9979(1)	0.5078(1)	0.047(1)	0.046(1)	0.069(2)	0.009(1)	−0.018(1)	−0.023(1)
C(64)	2*i*	0.7196(2)	1.0571(1)	0.4907(1)	0.055(1)	0.0317(9)	0.059(1)	0.0052(9)	−0.002(1)	−0.0164(9)
C(65)	2*i*	0.6149(2)	1.0311(1)	0.44938(9)	0.047(1)	0.0282(8)	0.045(1)	0.0097(8)	0.0069(9)	−0.0017(8)
C(66)	2*i*	0.5733(2)	0.9445(1)	0.42526(8)	0.035(1)	0.0295(8)	0.0311(9)	0.0079(7)	0.0056(8)	−0.0020(7)
C(67)	2*i*	0.6395(2)	0.8865(1)	0.44573(8)	0.038(1)	0.0252(8)	0.0321(9)	0.0068(7)	0.0013(8)	−0.0055(6)
C(68)	2*i*	0.4638(2)	0.9145(1)	0.37631(8)	0.031(1)	0.0274(8)	0.0380(9)	0.0078(7)	0.0040(8)	0.0016(7)
C(69)	2*i*	0.2620(2)	0.7384(1)	0.28605(9)	0.0252(9)	0.0348(9)	0.041(1)	0.0045(7)	0.0003(8)	0.0072(7)
C(70)	2*i*	0.1903(2)	0.6768(1)	0.32749(9)	0.028(1)	0.0306(8)	0.052(1)	0.0014(7)	0.0081(8)	0.0025(8)
C(71)	2*i*	0.1901(2)	0.7147(1)	0.3987(1)	0.036(1)	0.039(1)	0.054(1)	0.0082(8)	0.0114(9)	0.0047(8)
C(72)	2*i*	0.4310(2)	0.4501(1)	0.33479(8)	0.034(1)	0.0262(8)	0.0334(9)	0.0014(7)	0.0065(8)	0.0004(7)
C(73)	2*i*	0.4841(2)	0.3935(1)	0.3786(1)	0.046(1)	0.059(1)	0.080(2)	0.016(1)	0.022(1)	0.044(1)
C(74)	2*i*	0.8589(2)	0.6269(1)	0.47624(9)	0.039(1)	0.043(1)	0.041(1)	0.0130(9)	−0.0016(9)	0.0065(8)
C(75)	2*i*	0.7851(2)	0.5652(1)	0.5150(1)	0.054(1)	0.057(1)	0.047(1)	0.017(1)	0.010(1)	0.017(1)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(76)	2*i*	0.8555(2)	0.5086(1)	0.5500(1)	0.084(2)	0.048(1)	0.049(1)	0.023(1)	0.007(1)	0.013(1)
C(77)	2*i*	0.5308(2)	0.7502(1)	0.46255(8)	0.038(1)	0.0344(9)	0.0306(9)	0.0120(8)	0.0064(8)	0.0043(7)
C(78)	2*i*	0.4953(2)	0.6589(1)	0.43389(9)	0.037(1)	0.0292(8)	0.0361(9)	0.0090(7)	0.0054(8)	0.0041(7)
C(79)	2*i*	0.7443(2)	0.6848(1)	0.14946(9)	0.050(1)	0.046(1)	0.040(1)	0.0229(9)	0.0236(9)	0.0177(9)
C(80)	2*i*	0.8145(2)	0.7598(2)	0.1212(1)	0.074(2)	0.061(1)	0.082(2)	0.028(1)	0.049(2)	0.042(1)

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