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Crystal structure of 5,17-bis(*N-tert*-butylhydroxyamine)-25,26,27,28-tetrapropoxycalix[4]arene, $C_{48}H_{66}N_2O_6$

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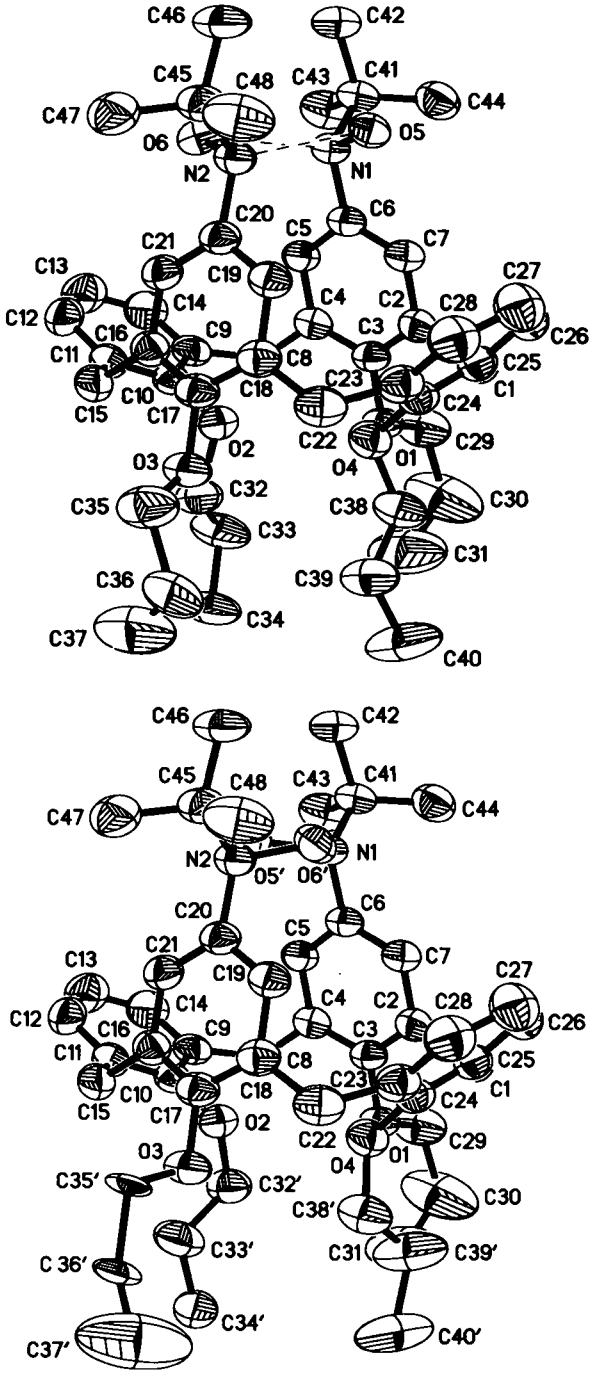

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Abstract

$C_{48}H_{66}N_2O_6$, monoclinic, $P12_1/c1$ (no. 14), a = 14.636(2) Å, b = 16.807(2) Å, c = 19.131(2) Å, β = 102.981(1)°, V = 4585.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.070, $wR_{ref}(F^2)$ = 0.160, T = 295 K.

Source of material

The title compound was prepared starting from the precursor compound 5,17-dibromo-25,26,27,28-tetrapropoxy-calix[4]arene, which was obtained according to [1], in a similar one-step procedure as described in [2] and recrystallized from chloroform/methanol.

Experimental details

Flexible propoxy units and weak intermolecular interactions are the reasons for the large R values and the low N_{gt}/N_{param} ratio which is often observed for substituted calixarenes.

Discussion

We first reported the synthesis of a paramagnetic calix[4]arene with two *N-tert*-butyl nitroxide groups on the upper rims and the study on spin exchange interactions [2]. A similar calix[4]arene with four *N-tert*-butyl nitroxide groups on the upper rims was already reported [3].The calixarene derivatives with *N-tert*-butylhydroxyamine groups are valuable precursors for this kind of paramagnetic calixarenes. Recently we have reported a new crystal structure of calix[4]arene derivative with two *N-tert*-butylhydroxyamine groups and two bromine atoms on the upper rims [4]. As a part of another ongoing investigations of the preparation of paramagnetic calix[4]arene, here we report a crystal structure of another calix[4]arene derivative only with two *N-tert*-butylhydroxyamine groups on the upper rims.

In the asymmetric unit, there are two conformational forms which are caused by the disorder of the substituted groups on the lower rims. The cone conformation of the calix[4]arene cavity is retained from the precursor, but pinched due to the two O–H···N hydrogen bonds between the two *N-tert*-butyl-hydroxyamine groups on the upper rims. In the first form, the hydrogen bond lengths are 2.06 Å for H5A···N2 and 2.05 Å for H6A···N1, and the angles are 150.0° for O5–H5A···N2 and 150.2° for O6–H6A···N1. In the second form, the hydrogen bond lengths are 2.05 Å for H5A'···N2 and 2.03 Å for H6A'···N1, and the angles are 147.8° for O5'–H5A'···N2 and 151.4° for O6'–H6A'···N1. The four dihedral angles are 34.9° for the phenyl ring C9-C14, 38.2° for the phenyl ring C23-C28, 75.1° for the phenyl ring C16-C21, and 76.8° for the phenyl ring C2-C7 to the reference plane defined by the bridging C atoms (here C1, C8, C15 and C22). The distances from C1, C8, C15 and C22 to the reference plane are 0.057 Å, –0.057 Å, 0.056 Å and –0.057 Å, respectively.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.4 × 0.6 × 0.7 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.72 cm^{-1}
Diffractometer, scan mode:	Bruker P4, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9774, 8079
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3824
$N(param)_{refined}$:	612
Program:	SHELXTL [5]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(5A)	4e	0.650	0.4624	0.4498	0.2675	0.084
H(6A)	4e	0.650	0.4611	0.3169	0.2225	0.091
H(5A')	4e	0.350	0.4532	0.3205	0.2143	0.100
H(6A')	4e	0.350	0.4719	0.4456	0.2752	0.102
H(1A)	4e		0.8216	0.5860	0.2168	0.095
H(1B)	4e		0.7297	0.6371	0.1957	0.095
H(5B)	4e		0.5770	0.3315	0.1156	0.084
H(7A)	4e		0.5816	0.5433	0.2110	0.086
H(8A)	4e		0.7223	0.3154	0.0435	0.094
H(8B)	4e		0.8154	0.3335	0.1003	0.094
H(12A)	4e		0.6738	0.0748	0.2356	0.117
H(13A)	4e		0.6221	0.0790	0.1116	0.135
H(14A)	4e		0.6589	0.1864	0.0485	0.118
H(15A)	4e		0.8459	0.1838	0.3419	0.100
H(15B)	4e		0.7587	0.1314	0.3467	0.100
H(19A)	4e		0.6193	0.4364	0.3984	0.086
H(21A)	4e		0.6004	0.2240	0.3029	0.087
H(22A)	4e		0.7891	0.4532	0.5012	0.105
H(22B)	4e		0.8674	0.4378	0.4585	0.105
H(26A)	4e		0.6686	0.6900	0.2928	0.114
H(27A)	4e		0.6474	0.6834	0.4088	0.132
H(28A)	4e		0.7084	0.5778	0.4810	0.119
H(29A)	4e		0.8109	0.5579	0.0758	0.139
H(29B)	4e		0.8287	0.4766	0.0406	0.139
H(30A)	4e		0.9538	0.5439	0.0389	0.314
H(30B)	4e		0.9563	0.5839	0.1123	0.314
H(31A)	4e		1.0792	0.5212	0.1139	0.349
H(31B)	4e		1.0235	0.4421	0.0916	0.349
H(31C)	4e		1.0246	0.4809	0.1663	0.349
H(32A)	4e	0.576	0.9383	0.2208	0.2781	0.118
H(32B)	4e	0.576	0.9617	0.3087	0.3030	0.118
H(33A)	4e	0.576	0.9728	0.3383	0.1843	0.163
H(33B)	4e	0.576	0.9642	0.2469	0.1681	0.163
H(34A)	4e	0.576	1.1216	0.2851	0.1930	0.209
H(34B)	4e	0.576	1.1055	0.2246	0.2514	0.209
H(34C)	4e	0.576	1.1139	0.3158	0.2688	0.209
H(32C)	4e	0.424	0.9460	0.3575	0.2285	0.114
H(32D)	4e	0.424	0.9229	0.2848	0.1758	0.114
H(33C)	4e	0.424	0.9775	0.1969	0.2672	0.162
H(33D)	4e	0.424	0.9895	0.2653	0.3244	0.162
H(34D)	4e	0.424	1.1351	0.2316	0.2995	0.211
H(34E)	4e	0.424	1.1106	0.3206	0.2792	0.211
H(34F)	4e	0.424	1.0968	0.2555	0.2191	0.211
H(35A)	4e	0.693	0.9198	0.2177	0.4946	0.153
H(35B)	4e	0.693	0.8841	0.2962	0.5238	0.153
H(36A)	4e	0.693	1.0318	0.3587	0.5076	0.156
H(36B)	4e	0.693	1.0449	0.2968	0.5712	0.156
H(37A)	4e	0.693	1.1423	0.2632	0.4997	0.334
H(37B)	4e	0.693	1.0582	0.2627	0.4325	0.334
H(37C)	4e	0.693	1.0639	0.1982	0.4925	0.334
H(35C)	4e	0.307	0.8670	0.2015	0.4734	0.086
H(35D)	4e	0.307	0.8779	0.2786	0.5216	0.086
H(36C)	4e	0.307	1.0129	0.1831	0.5052	0.097
H(36D)	4e	0.307	1.0248	0.2560	0.4590	0.097
H(37D)	4e	0.307	1.1365	0.2777	0.5417	0.448
H(37E)	4e	0.307	1.0939	0.2315	0.5981	0.448
H(37F)	4e	0.307	1.0628	0.3193	0.5777	0.448
H(38A)	4e	0.526	0.9398	0.5660	0.3142	0.127
H(38B)	4e	0.526	0.9495	0.4921	0.2668	0.127
H(39A)	4e	0.526	1.0247	0.4234	0.3702	0.151
H(39B)	4e	0.526	1.0197	0.5011	0.4152	0.151
H(40A)	4e	0.526	1.1658	0.4933	0.3795	0.264
H(40B)	4e	0.526	1.1107	0.5701	0.3484	0.264
H(40C)	4e	0.526	1.1112	0.4958	0.2990	0.264
H(38C)	4e	0.474	0.9695	0.4253	0.3679	0.130
H(38D)	4e	0.474	0.9533	0.5058	0.4054	0.130
H(39C)	4e	0.474	0.9889	0.5791	0.3159	0.197
H(39D)	4e	0.474	0.9823	0.5044	0.2653	0.197
H(40D)	4e	0.474	1.1417	0.5311	0.3148	0.274
H(40E)	4e	0.474	1.1165	0.4492	0.3449	0.274
H(40F)	4e	0.474	1.1242	0.5259	0.3925	0.274
H(42A)	4e		0.2957	0.3703	0.1351	0.147
H(42B)	4e		0.2478	0.4412	0.0874	0.147
H(42C)	4e		0.2939	0.4552	0.1686	0.147
H(43A)	4e		0.4598	0.4108	0.0290	0.158
H(43B)	4e		0.3505	0.4110	0.0017	0.158
H(43C)	4e		0.3997	0.3424	0.0518	0.158
H(44A)	4e		0.4565	0.5467	0.0790	0.151
H(44B)	4e		0.3940	0.5650	0.1338	0.151
H(44C)	4e		0.3471	0.5501	0.0528	0.151
H(46A)	4e		0.2911	0.3263	0.3666	0.168
H(46B)	4e		0.3158	0.3071	0.2928	0.168
H(46C)	4e		0.3253	0.3943	0.3225	0.168
H(47A)	4e		0.5106	0.2257	0.4189	0.183
H(47B)	4e		0.4339	0.2034	0.3504	0.183
H(47C)	4e		0.4050	0.2199	0.4231	0.183
H(48A)	4e		0.5229	0.3623	0.4656	0.196
H(48B)	4e		0.4179	0.3625	0.4716	0.196
H(48C)	4e		0.4547	0.4288	0.4273	0.196

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4e		0.83959(6)	0.46935(6)	0.14534(5)	0.0522(4)	0.1026(7)	0.0718(5)	0.0033(5)	0.0191(4)	0.0148(5)
O(2)	4e		0.83460(6)	0.29635(6)	0.24000(5)	0.0603(5)	0.0836(7)	0.0794(6)	–0.0036(5)	0.0156(4)	–0.0043(5)
O(3)	4e		0.88260(6)	0.30065(7)	0.41998(5)	0.0539(5)	0.1172(8)	0.0782(6)	0.0082(6)	0.0140(4)	0.0166(6)
O(4)	4e		0.85010(6)	0.47897(6)	0.32209(5)	0.0707(5)	0.0881(7)	0.0836(6)	0.0087(5)	0.0199(5)	–0.0063(6)
O(5)	4e	0.650(1)	0.45231(9)	0.47497(9)	0.22979(7)	0.0634(7)	0.085(1)	0.0639(8)	0.0099(8)	0.0213(6)	–0.0065(8)
O(6)	4e	0.650	0.46136(9)	0.29200(9)	0.25943(7)	0.0634(8)	0.087(1)	0.0759(9)	–0.0081(8)	0.0143(7)	–0.0023(8)
O(5')	4e	0.350	0.4538(2)	0.3338(2)	0.1732(2)	0.063(2)	0.092(2)	0.095(2)	–0.003(2)	0.017(1)	0.005(2)
O(6')	4e	0.350	0.4754(2)	0.4310(1)	0.3166(2)	0.074(2)	0.086(2)	0.098(2)	0.020(2)	0.022(2)	0.012(2)
N(1)	4e		0.46817(7)	0.42249(7)	0.17067(6)	0.0508(5)	0.0793(8)	0.0728(7)	0.0035(6)	0.0169(5)	–0.0009(6)
N(2)	4e		0.49392(7)	0.34436(7)	0.32205(6)	0.0545(6)	0.0846(8)	0.0712(7)	0.0042(6)	0.0158(5)	0.0069(6)
C(1)	4e		0.7565(1)	0.58912(9)	0.22020(8)	0.0673(8)	0.077(1)	0.093(1)	–0.0029(8)	0.0171(7)	0.0026(9)
C(2)	4e		0.70387(9)	0.51726(9)	0.18389(7)	0.0608(7)	0.0764(9)	0.0657(8)	0.0060(8)	0.0119(6)	0.0060(8)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(3)	4e		0.74592(8)	0.45926(9)	0.14982(7)	0.0501(6)	0.085(1)	0.0640(7)	0.0023(7)	0.0150(6)	0.0090(8)
C(4)	4e		0.70076(9)	0.38916(9)	0.12442(7)	0.0587(7)	0.085(1)	0.0595(7)	0.0072(8)	0.0132(6)	0.0026(8)
C(5)	4e		0.60850(9)	0.37850(9)	0.13154(7)	0.0541(7)	0.079(1)	0.0765(8)	0.0031(7)	0.0126(6)	−0.0031(8)
C(6)	4e		0.56355(8)	0.43703(9)	0.16201(7)	0.0488(7)	0.084(1)	0.0661(8)	0.0050(7)	0.0134(6)	0.0057(8)
C(7)	4e		0.61147(9)	0.50503(9)	0.18901(7)	0.0552(7)	0.085(1)	0.0756(8)	0.0050(8)	0.0175(6)	−0.0024(8)
C(8)	4e		0.7494(1)	0.32126(9)	0.09436(7)	0.0656(8)	0.100(1)	0.0716(8)	0.0104(8)	0.0181(7)	−0.0092(9)
C(9)	4e		0.73859(9)	0.24490(9)	0.13228(8)	0.0552(7)	0.084(1)	0.0803(9)	0.0122(7)	0.0163(6)	−0.0163(8)
C(10)	4e		0.77662(8)	0.23646(8)	0.20532(7)	0.0509(7)	0.0674(9)	0.0852(9)	0.0064(7)	0.0189(6)	−0.0124(8)
C(11)	4e		0.75089(9)	0.17554(9)	0.24639(8)	0.0627(7)	0.0721(9)	0.099(1)	0.0125(7)	0.0282(7)	−0.0015(8)
C(12)	4e		0.6918(1)	0.1168(1)	0.2100(1)	0.0812(9)	0.071(1)	0.147(1)	−0.0044(9)	0.0369(9)	−0.013(1)
C(13)	4e		0.6591(1)	0.1199(1)	0.1356(1)	0.097(1)	0.098(1)	0.140(2)	−0.012(1)	0.020(1)	−0.038(1)
C(14)	4e		0.6819(1)	0.1840(1)	0.0979(1)	0.088(1)	0.098(1)	0.107(1)	0.003(1)	0.0160(9)	−0.033(1)
C(15)	4e		0.7782(1)	0.17981(9)	0.32659(8)	0.0675(8)	0.078(1)	0.111(1)	0.0091(8)	0.0324(7)	0.0172(9)
C(16)	4e		0.73311(9)	0.25136(9)	0.35456(7)	0.0602(7)	0.0773(9)	0.0756(8)	0.0053(7)	0.0230(6)	0.0132(8)
C(17)	4e		0.78495(9)	0.30991(9)	0.39720(7)	0.0563(7)	0.096(1)	0.0656(8)	0.0046(8)	0.0195(6)	0.0112(8)
C(18)	4e		0.74412(9)	0.37995(9)	0.41415(7)	0.0624(7)	0.090(1)	0.0621(8)	−0.0002(8)	0.0161(6)	0.0035(8)
C(19)	4e		0.64786(9)	0.38951(9)	0.38858(7)	0.0614(7)	0.086(1)	0.0716(8)	0.0064(8)	0.0208(6)	0.0010(8)
C(20)	4e		0.59437(9)	0.33072(9)	0.34907(7)	0.0533(7)	0.085(1)	0.0701(8)	0.0014(7)	0.0209(6)	0.0092(8)
C(21)	4e		0.63663(9)	0.26266(9)	0.33102(7)	0.0604(7)	0.082(1)	0.0786(9)	−0.0037(8)	0.0221(6)	0.0017(8)
C(22)	4e		0.8010(1)	0.4484(1)	0.45354(8)	0.0744(9)	0.116(1)	0.0687(9)	0.002(1)	0.0111(7)	−0.0097(9)
C(23)	4e		0.7749(1)	0.5251(1)	0.41262(8)	0.0644(8)	0.091(1)	0.0749(9)	−0.0039(8)	0.0114(7)	−0.0202(8)
C(24)	4e		0.79433(9)	0.53486(9)	0.34536(8)	0.0569(7)	0.0741(9)	0.0830(9)	−0.0044(7)	0.0145(7)	−0.0183(8)
C(25)	4e		0.75173(9)	0.59342(9)	0.29757(8)	0.0606(8)	0.0736(9)	0.090(1)	−0.0056(8)	0.0153(7)	−0.0114(8)
C(26)	4e		0.6965(1)	0.6491(1)	0.32282(9)	0.082(1)	0.080(1)	0.122(1)	0.0050(9)	0.0217(9)	−0.010(1)
C(27)	4e		0.6822(1)	0.6445(1)	0.3919(1)	0.105(1)	0.109(1)	0.123(1)	0.012(1)	0.038(1)	−0.033(1)
C(28)	4e		0.7201(1)	0.5818(1)	0.43535(9)	0.094(1)	0.111(1)	0.100(1)	−0.001(1)	0.0327(9)	−0.029(1)
C(29)	4e		0.8497(1)	0.5105(1)	0.08218(8)	0.085(1)	0.177(2)	0.093(1)	0.009(1)	0.0351(8)	0.047(1)
C(30)	4e		0.9486(1)	0.5331(2)	0.0877(1)	0.136(1)	0.409(3)	0.282(2)	0.085(2)	0.134(1)	0.231(2)
C(31)	4e		1.0241(2)	0.4915(2)	0.1169(1)	0.111(2)	0.399(4)	0.198(2)	−0.019(2)	0.056(2)	0.098(3)
C(32)	4e	0.576(2)	0.9331(1)	0.2758(2)	0.2623(1)	0.063(2)	0.129(3)	0.099(2)	−0.009(2)	0.008(1)	0.022(2)
C(33)	4e	0.576	0.9860(2)	0.2859(3)	0.2055(2)	0.069(2)	0.204(4)	0.140(3)	0.040(2)	0.036(2)	0.009(3)
C(34)	4e	0.576	1.0915(2)	0.2770(3)	0.2321(2)	0.057(2)	0.211(4)	0.148(3)	0.024(2)	0.021(2)	0.028(3)
C(32')	4e	0.424	0.9256(2)	0.3025(2)	0.2245(2)	0.067(2)	0.122(3)	0.095(3)	−0.002(2)	0.018(2)	0.021(3)
C(33')	4e	0.424	0.9936(2)	0.2526(3)	0.2757(2)	0.071(2)	0.154(4)	0.179(5)	0.027(3)	0.026(3)	0.034(4)
C(34')	4e	0.424	1.0932(2)	0.2663(3)	0.2676(3)	0.071(2)	0.108(4)	0.248(6)	0.011(3)	0.046(3)	0.010(4)
C(35)	4e	0.693(2)	0.9220(2)	0.2753(2)	0.4924(1)	0.082(2)	0.197(3)	0.093(2)	−0.013(2)	−0.000(1)	0.048(2)
C(36)	4e	0.693	1.0247(2)	0.3033(2)	0.5196(1)	0.102(2)	0.165(3)	0.105(2)	0.033(2)	−0.012(2)	0.023(2)
C(37)	4e	0.693	1.0765(2)	0.2527(3)	0.4831(2)	0.115(3)	0.308(6)	0.214(4)	0.011(4)	−0.027(3)	0.008(4)
C(35')	4e	0.307	0.8991(2)	0.2521(3)	0.4830(2)	0.025(2)	0.114(4)	0.067(3)	0.017(2)	−0.010(2)	0.031(3)
C(36')	4e	0.307	1.0054(2)	0.2405(3)	0.5024(2)	0.032(2)	0.103(4)	0.100(3)	0.026(2)	−0.002(2)	−0.006(3)
C(37')	4e	0.307	1.0811(4)	0.2698(6)	0.5599(3)	0.142(7)	0.53(2)	0.214(8)	0.07(1)	0.006(7)	−0.17(1)
C(38)	4e	0.526(2)	0.9411(2)	0.5083(2)	0.3136(2)	0.065(2)	0.143(3)	0.114(2)	0.024(2)	0.030(2)	0.001(2)
C(39)	4e	0.526	1.0231(2)	0.4811(2)	0.3683(2)	0.070(2)	0.172(4)	0.126(3)	−0.004(2)	0.002(2)	0.008(3)
C(40)	4e	0.526	1.1109(2)	0.5130(3)	0.3468(2)	0.079(2)	0.308(6)	0.132(3)	−0.061(3)	0.003(2)	0.058(4)
C(38')	4e	0.474	0.9466(2)	0.4794(2)	0.3595(2)	0.077(2)	0.151(4)	0.098(2)	0.019(2)	0.023(2)	0.014(3)
C(39')	4e	0.474	1.0009(2)	0.5224(3)	0.3147(2)	0.080(2)	0.300(7)	0.110(3)	−0.034(4)	0.016(2)	0.031(4)
C(40')	4e	0.474	1.1057(2)	0.5056(4)	0.3445(3)	0.075(3)	0.298(6)	0.175(4)	−0.029(4)	0.027(3)	−0.128(4)
C(41)	4e		0.39213(9)	0.4461(1)	0.10678(7)	0.0524(7)	0.099(1)	0.0717(8)	0.0098(8)	0.0105(6)	0.0008(9)
C(42)	4e		0.29867(9)	0.4264(1)	0.12630(9)	0.0539(8)	0.130(2)	0.108(1)	0.002(1)	0.0140(8)	−0.007(1)
C(43)	4e		0.4014(1)	0.3981(1)	0.04129(8)	0.085(1)	0.145(2)	0.080(1)	0.013(1)	0.0059(9)	−0.026(1)
C(44)	4e		0.3980(1)	0.5353(1)	0.09170(9)	0.082(1)	0.111(1)	0.106(1)	0.018(1)	0.0160(9)	0.023(1)
C(45)	4e		0.4339(1)	0.3223(1)	0.37409(8)	0.0616(8)	0.118(1)	0.0863(9)	0.0077(9)	0.0286(7)	0.019(1)
C(46)	4e		0.3320(1)	0.3391(1)	0.33538(9)	0.0532(8)	0.163(2)	0.124(1)	0.008(1)	0.0291(8)	0.016(1)
C(47)	4e		0.4471(1)	0.2348(1)	0.3934(1)	0.087(1)	0.147(2)	0.140(1)	0.000(1)	0.0430(9)	0.055(1)
C(48)	4e		0.4597(1)	0.3737(1)	0.44077(9)	0.099(1)	0.213(2)	0.088(1)	0.026(1)	0.0370(9)	−0.013(1)

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