Crystal structure of 5,11,17,23-tetra(*tert*-butyl)-25,27-di(phenylmethoxy)-26,28-di(diethylacetamido)calix[4]arene, $C_{70}H_{90}N_2O_6$

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Abstract

$C_{70}H_{90}N_2O_6$, orthorhombic, $P2_12_12_1$ (No. 19), $a = 15.4958(9)$ Å, $b = 17.920(1)$ Å, $c = 22.2069(8)$ Å, $V = 6166.4$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.062$, $wR_{\text{all}}(F^2) = 0.150$, $T = 123$ K.

Source of material

In a preliminary step, *p-tert*-butyl calix[4]arene was treated with benzyl bromide (2.5 eq.) and potassium carbonate (14.5 eq.) in boiling acetone under nitrogen for 24 h to yield (80%) the 1,3-dialkylated derivative after precipitation of the crude mixture from methanol. This derivative was further treated in similar conditions with 2-chloro-*N,N*-diethylacetamide (3.0 eq.) in presence of an excess of sodium iodide and refluxed for 6 h. The yield was 45% after chromatography of the crude mixture on silica (Merck Kieselgel no. 11567) with 90:10 dichloromethane:acetone as eluent. After recrystallisation from methanol the title compound was obtained as a white solid melting at 410 K ~ 411 K.

Discussion

The amide derivatives of calix[4]arene and their complexes with alkali metal, transition metal and lanthanide ions have been extensively investigated [1–5]. The title compound is related to diamide derivatives previously described which showed a strong preference for lighter alkali metal cations (Li^+ and Na^+) [6]. The cone conformation and the pseudo- C_2 symmetry of the present molecule were deduced from ¹H-NMR spectrum at 200 MHz in $CDCl_3$. A single AB system was observed for the linking methylene groups at 3.00 and 3.68 ppm ($J = 13.0$ Hz) along with two singlets at 1.02 and 1.12 ppm for the *tert*-butyl groups and two other singlets at 6.95 and 7.02 ppm for the *meta* protons of the phenolic unit of the calixarene. The crystal structure here reported confirms this conformation.

The asymmetric unit comprises one molecule, without any solvent molecule included. The calixarene conformation is a distorted cone, two phenolic rings being more tilted than the two other ones with respect to the mean plane of the four methylenic carbon atoms. The corresponding dihedral angles are 88.91(7)° and 84.12(8)° in the former case (rings associated to O3 and O6, which are nearly parallel to each other) and 51.2(1)° and 43.5(1)° in the latter (rings associated to O1 and O4). Such a situation is common in calix[4]arene with bulky substituents [5, 7] and the resulting pinching of the cone has been thought to prevent inclusion of solvent molecules, otherwise a widespread occurrence [5]. This conformation makes the benzyl substituents (which are nearly parallel to each other within the molecule, with a dihedral angle of 7.8(8)°) more far away from each other than the amide ones. The packing brings the benzyl rings of adjacent molecules nearly perpendicular to each other (dihedral angle 82.0(9)°), with a shortest distance of 3.588(7) Å. The investigation of the complexing properties of this compound is in process. Extraction of alkali picrates from water to dichloromethane revealed a high level of extraction with selectivity for Na^+ , K^+ , Rb^+ .

Table 1. Data collection and handling.

Crystal:	colourless, parallelepiped, size 0.20 × 0.20 × 0.35 mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	0.71 cm ⁻¹
Diffractionmeter, scan mode:	Nonius Kappa-CCD, $\Delta\phi \approx 2^\circ$
$2\theta_{\text{max}}$:	52.22°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	34124, 9391
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 6948
$N(\text{param})_{\text{refined}}$:	719
Programs:	SHELXS-86 [8], SHELXL-93 [9], SHELXTL [10]

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

Atom	Site	x	y	z	<i>U</i> _{iso}
H(1A)	4a	0.9526(3)	0.0705(2)	0.1464(2)	0.042
H(1B)	4a	0.8981(3)	0.0757(2)	0.2056(2)	0.042
H(3A)	4a	0.9998(3)	0.3029(2)	0.2334(2)	0.057
H(3B)	4a	0.9120(3)	0.3074(2)	0.1986(2)	0.057
H(4A)	4a	1.021(2)	0.3817(5)	0.1558(8)	0.110
H(4B)	4a	0.981(1)	0.325(2)	0.1097(5)	0.110
H(4C)	4a	1.071(1)	0.308(2)	0.139(1)	0.110
H(5A)	4a	1.0951(3)	0.1932(3)	0.2120(2)	0.055
H(5B)	4a	1.0453(3)	0.1181(3)	0.2024(2)	0.055
H(6A)	4a	1.075(1)	0.132(2)	0.3039(3)	0.101
H(6B)	4a	0.975(1)	0.136(2)	0.2949(2)	0.101
H(6C)	4a	1.027(3)	0.2096(3)	0.3047(3)	0.101
H(7A)	4a	1.0723(3)	-0.1341(2)	0.1079(2)	0.045
H(7B)	4a	1.0373(3)	-0.1396(2)	0.1742(2)	0.045
H(9)	4a	1.1451(3)	-0.0115(3)	0.0781(2)	0.054
H(10)	4a	1.2495(3)	0.0729(3)	0.1099(2)	0.064
H(11)	4a	1.2755(3)	0.0890(3)	0.2111(2)	0.063
H(12)	4a	1.2028(3)	0.0192(2)	0.2809(2)	0.056
H(13)	4a	1.0986(3)	-0.0649(2)	0.2498(2)	0.046
H(14A)	4a	0.9425(2)	0.0887(2)	0.0064(2)	0.039
H(14B)	4a	0.9243(2)	0.0706(2)	-0.0614(2)	0.039
H(16A)	4a	1.2047(3)	0.0346(3)	-0.0377(2)	0.054
H(16B)	4a	1.2197(3)	0.1193(3)	-0.0234(2)	0.054
H(17A)	4a	1.2785(4)	0.093(2)	-0.1168(4)	0.089
H(17B)	4a	1.200(2)	0.1474(8)	-0.1261(6)	0.089
H(17C)	4a	1.190(2)	0.062(1)	-0.1397(3)	0.089
H(18A)	4a	1.0073(3)	0.1753(2)	-0.0541(2)	0.054
H(18B)	4a	1.1023(3)	0.2039(2)	-0.0610(2)	0.054
H(19A)	4a	1.014(2)	0.181(2)	0.0474(6)	0.100
H(19B)	4a	1.055(2)	0.2557(4)	0.0250(2)	0.100
H(19C)	4a	1.115(1)	0.190(2)	0.0466(6)	0.100
H(20A)	4a	0.7993(3)	0.2339(2)	0.0311(2)	0.042
H(20B)	4a	0.7101(3)	0.2203(2)	-0.0007(2)	0.042
H(22)	4a	0.9007(3)	0.3046(2)	-0.0235(2)	0.061
H(23)	4a	0.9603(4)	0.3469(3)	-0.1129(3)	0.089
H(24)	4a	0.9034(5)	0.3100(3)	-0.2030(3)	0.091
H(25)	4a	0.7825(4)	0.2339(3)	-0.2053(2)	0.070
H(26)	4a	0.7233(3)	0.1921(2)	-0.1166(2)	0.052
H(29)	4a	0.6864(2)	-0.0772(2)	0.2667(2)	0.034
H(31)	4a	0.5538(3)	0.0796(2)	0.1803(2)	0.035
H(34A)	4a	0.574(1)	-0.0816(6)	0.3325(8)	0.062
H(34B)	4a	0.552(2)	-0.1338(5)	0.2779(3)	0.062
H(34C)	4a	0.4782(5)	-0.105(1)	0.320(1)	0.062
H(35A)	4a	0.5314(8)	0.055(1)	0.3278(9)	0.063
H(35B)	4a	0.437(1)	0.0262(6)	0.320(1)	0.063

Table 2. Continued.

Atom	Site	x	y	z	<i>U</i> _{iso}
H(35C)	4a	0.476(2)	0.0826(6)	0.2732(2)	0.063
H(36A)	4a	0.444(1)	-0.0022(6)	0.1871(8)	0.060
H(36B)	4a	0.4001(6)	-0.057(2)	0.2324(2)	0.060
H(36C)	4a	0.4737(8)	-0.086(1)	0.1897(8)	0.060
H(37A)	4a	0.8905(3)	-0.0403(2)	0.2142(2)	0.034
H(37B)	4a	0.8353(3)	-0.0973(2)	0.2518(2)	0.034
H(41)	4a	0.8256(2)	-0.2465(2)	0.0279(2)	0.032
H(43)	4a	0.7334(2)	-0.1803(2)	0.1864(2)	0.033
H(45A)	4a	0.5666(5)	-0.2918(8)	0.119(1)	0.093
H(45B)	4a	0.596(1)	-0.211(1)	0.103(1)	0.093
H(45C)	4a	0.6146(7)	-0.243(2)	0.1672(4)	0.093
H(46A)	4a	0.680(1)	-0.3883(8)	0.140(1)	0.082
H(46B)	4a	0.728(2)	-0.3332(5)	0.1836(3)	0.082
H(46C)	4a	0.778(1)	-0.369(1)	0.1294(9)	0.082
H(47A)	4a	0.638(2)	-0.349(2)	0.0378(3)	0.082
H(47B)	4a	0.7368(8)	-0.337(2)	0.0252(6)	0.082
H(47C)	4a	0.671(2)	-0.2722(4)	0.0125(3)	0.082
H(48A)	4a	0.9878(2)	-0.2019(2)	0.0049(2)	0.032
H(48B)	4a	0.9979(2)	-0.1168(2)	0.0202(2)	0.032
H(52)	4a	0.7559(2)	-0.0905(2)	-0.1689(2)	0.035
H(54)	4a	0.9034(2)	-0.2373(2)	-0.0823(2)	0.035
H(56A)	4a	0.7421(9)	-0.169(1)	-0.243(1)	0.100
H(56B)	4a	0.6788(7)	-0.202(2)	-0.1950(3)	0.100
H(56C)	4a	0.714(2)	-0.2533(8)	-0.247(1)	0.100
H(57A)	4a	0.897(1)	-0.1990(5)	-0.241(1)	0.078
H(57B)	4a	0.8696(8)	-0.282(1)	-0.2522(9)	0.078
H(57C)	4a	0.9317(7)	-0.263(2)	-0.1987(3)	0.078
H(58A)	4a	0.8357(6)	-0.3252(8)	-0.128(1)	0.072
H(58B)	4a	0.768(2)	-0.3445(5)	-0.1786(2)	0.072
H(58C)	4a	0.739(1)	-0.3009(5)	-0.121(1)	0.072
H(59A)	4a	0.7433(2)	0.0344(2)	-0.1395(2)	0.033
H(59B)	4a	0.8124(2)	0.0690(2)	-0.0962(2)	0.033
H(63)	4a	0.5389(3)	0.0455(2)	0.0696(2)	0.041
H(65)	4a	0.6195(3)	-0.0360(2)	-0.0876(2)	0.037
H(67A)	4a	0.380(3)	-0.082(2)	0.033(1)	0.174
H(67B)	4a	0.394(3)	0.004(1)	0.038(2)	0.174
H(67C)	4a	0.4555(8)	-0.050(4)	0.0716(4)	0.174
H(68A)	4a	0.4460(7)	-0.1517(8)	-0.045(2)	0.154
H(68B)	4a	0.523(3)	-0.1516(9)	0.0015(9)	0.154
H(68C)	4a	0.539(3)	-0.1295(4)	-0.066(2)	0.154
H(69A)	4a	0.377(2)	-0.042(2)	-0.076(2)	0.141
H(69B)	4a	0.464(1)	-0.019(3)	-0.1072(5)	0.141
H(69C)	4a	0.416(3)	0.0370(9)	-0.064(1)	0.141
H(70A)	4a	0.7230(3)	0.1585(2)	0.0985(2)	0.036
H(70B)	4a	0.6231(3)	0.1644(2)	0.1098(2)	0.036

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
O(1)	4a	0.8278(2)	0.0602(1)	0.1331(1)	0.028(2)	0.035(2)	0.034(1)	-0.003(1)	0.006(1)	-0.002(1)
O(2)	4a	0.8489(2)	0.2125(2)	0.1350(1)	0.044(2)	0.037(2)	0.046(2)	0.002(2)	-0.002(1)	0.004(1)
O(3)	4a	0.9701(2)	-0.0698(1)	0.1229(1)	0.020(1)	0.026(1)	0.036(1)	-0.001(1)	-0.002(1)	-0.002(1)
O(4)	4a	0.8989(2)	-0.0115(1)	-0.0071(1)	0.027(1)	0.024(1)	0.031(1)	-0.003(1)	0.002(1)	-0.002(1)
O(5)	4a	1.0721(2)	-0.0283(2)	-0.0368(1)	0.033(2)	0.035(2)	0.077(2)	0.009(2)	0.013(2)	0.005(2)
O(6)	4a	0.7864(2)	0.1308(1)	-0.0013(1)	0.025(1)	0.022(1)	0.041(1)	0.002(1)	0.004(1)	0.004(1)
N(1)	4a	0.9708(2)	0.2084(2)	0.1896(2)	0.034(2)	0.032(2)	0.048(2)	0.000(2)	0.001(2)	-0.005(2)
N(2)	4a	1.0951(2)	0.0952(2)	-0.0410(2)	0.025(2)	0.029(2)	0.052(2)	-0.001(2)	0.004(2)	-0.001(2)
C(1)	4a	0.9002(3)	0.0908(2)	0.1637(2)	0.028(2)	0.032(2)	0.043(2)	-0.007(2)	0.001(2)	-0.003(2)
C(2)	4a	0.9037(3)	0.1754(2)	0.1604(2)	0.031(2)	0.032(2)	0.032(2)	-0.003(2)	0.010(2)	-0.002(2)
C(3)	4a	0.9712(3)	0.2900(2)	0.1959(2)	0.052(3)	0.039(3)	0.051(3)	-0.004(2)	0.005(2)	-0.011(2)
C(4)	4a	1.0148(4)	0.3299(3)	0.1456(3)	0.084(4)	0.044(3)	0.092(4)	-0.010(3)	0.032(3)	-0.010(3)
C(5)	4a	1.0411(3)	0.1676(3)	0.2197(2)	0.032(3)	0.041(3)	0.064(3)	0.001(2)	-0.003(2)	-0.002(2)
C(6)	4a	1.0284(4)	0.1607(3)	0.2869(2)	0.064(4)	0.075(4)	0.064(3)	0.013(3)	-0.022(3)	-0.007(3)
C(7)	4a	1.0489(3)	-0.1055(2)	0.1412(2)	0.029(2)	0.030(2)	0.055(2)	0.008(2)	-0.013(2)	0.001(2)
C(8)	4a	1.1128(2)	-0.0479(2)	0.1607(2)	0.021(2)	0.030(2)	0.041(2)	0.001(2)	-0.004(2)	-0.002(2)
C(9)	4a	1.1568(3)	-0.0059(3)	0.1189(2)	0.038(3)	0.051(3)	0.045(2)	-0.006(2)	0.001(2)	0.004(2)
C(10)	4a	1.2188(3)	0.0451(3)	0.1380(2)	0.040(3)	0.049(3)	0.071(3)	-0.008(3)	0.011(2)	0.015(2)
C(11)	4a	1.2346(3)	0.0543(3)	0.1985(2)	0.035(3)	0.042(3)	0.082(3)	-0.007(2)	-0.020(2)	-0.009(2)

Table 3. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(12)	4a	1.1911(3)	0.0133(2)	0.2401(2)	0.047(3)	0.037(2)	0.055(3)	-0.001(2)	-0.018(2)	-0.003(2)
C(13)	4a	1.1293(3)	-0.0375(2)	0.2214(2)	0.037(2)	0.034(2)	0.043(2)	-0.003(2)	-0.005(2)	0.001(2)
C(14)	4a	0.9481(2)	0.0506(2)	-0.0243(2)	0.029(2)	0.031(2)	0.037(2)	0.001(2)	0.002(2)	-0.008(2)
C(15)	4a	1.0433(3)	0.0346(2)	-0.0340(2)	0.028(2)	0.031(2)	0.036(2)	0.004(2)	0.000(2)	-0.001(2)
C(16)	4a	1.1890(3)	0.0850(3)	-0.0495(2)	0.028(2)	0.049(3)	0.058(3)	-0.006(2)	0.000(2)	0.008(2)
C(17)	4a	1.2170(3)	0.0981(3)	-0.1139(2)	0.042(3)	0.076(3)	0.060(3)	0.006(3)	0.008(2)	-0.003(3)
C(18)	4a	1.0647(3)	0.1724(2)	-0.0369(2)	0.041(3)	0.032(2)	0.063(3)	-0.004(2)	0.006(2)	-0.005(2)
C(19)	4a	1.0619(4)	0.2025(3)	0.0262(2)	0.056(3)	0.065(3)	0.079(3)	-0.020(3)	0.023(3)	-0.035(3)
C(20)	4a	0.7715(3)	0.2103(2)	-0.0030(2)	0.041(2)	0.024(2)	0.040(2)	0.002(2)	0.008(2)	0.000(2)
C(21)	4a	0.8071(3)	0.2417(2)	-0.0604(2)	0.028(2)	0.025(2)	0.046(2)	0.005(2)	0.005(2)	0.006(2)
C(22)	4a	0.8772(3)	0.2894(2)	-0.0600(2)	0.037(3)	0.042(3)	0.073(3)	-0.005(2)	-0.008(2)	0.017(2)
C(23)	4a	0.9129(4)	0.3151(3)	-0.1137(3)	0.038(3)	0.072(4)	0.112(5)	0.002(3)	0.016(3)	0.051(4)
C(24)	4a	0.8786(5)	0.2937(3)	-0.1672(3)	0.093(5)	0.060(4)	0.074(4)	0.036(4)	0.041(4)	0.031(3)
C(25)	4a	0.8068(4)	0.2477(3)	-0.1687(2)	0.091(4)	0.041(3)	0.044(3)	0.016(3)	0.009(3)	0.003(2)
C(26)	4a	0.7718(3)	0.2226(2)	-0.1155(2)	0.060(3)	0.030(2)	0.040(2)	0.005(2)	0.000(2)	0.003(3)
C(27)	4a	0.7561(2)	0.0400(2)	0.1671(2)	0.027(2)	0.034(2)	0.025(2)	-0.003(2)	0.004(2)	0.000(2)
C(28)	4a	0.7596(2)	-0.0192(2)	0.2082(2)	0.024(2)	0.032(2)	0.024(2)	0.005(2)	-0.001(2)	-0.007(2)
C(29)	4a	0.6842(2)	-0.0385(2)	0.2389(2)	0.031(2)	0.032(2)	0.021(2)	0.002(2)	0.000(2)	-0.005(2)
C(30)	4a	0.6059(3)	-0.0024(2)	0.2297(2)	0.031(2)	0.029(2)	0.022(2)	-0.001(2)	0.000(2)	-0.006(2)
C(31)	4a	0.6054(3)	0.0545(2)	0.1873(2)	0.025(2)	0.033(2)	0.031(2)	0.001(2)	0.001(2)	-0.001(2)
C(32)	4a	0.6780(2)	0.0760(2)	0.1546(2)	0.025(2)	0.027(2)	0.030(2)	0.002(2)	-0.002(2)	-0.004(2)
C(33)	4a	0.5212(2)	-0.0241(2)	0.2613(2)	0.027(2)	0.034(2)	0.026(2)	0.002(2)	0.002(2)	0.001(2)
C(34)	4a	0.5325(3)	-0.0924(2)	0.3017(2)	0.036(2)	0.053(3)	0.035(2)	0.000(2)	0.003(2)	0.007(2)
C(35)	4a	0.4882(3)	0.0409(3)	0.2990(2)	0.030(2)	0.053(3)	0.043(2)	-0.003(2)	0.008(2)	-0.006(2)
C(36)	4a	0.4534(3)	-0.0443(2)	0.2131(2)	0.030(2)	0.049(3)	0.041(2)	-0.008(2)	-0.003(2)	0.007(2)
C(37)	4a	0.8381(3)	-0.0700(2)	0.2141(2)	0.030(2)	0.029(2)	0.026(2)	0.000(2)	-0.003(2)	0.000(2)
C(38)	4a	0.8396(2)	-0.1241(2)	0.1619(2)	0.028(2)	0.023(2)	0.028(2)	0.005(2)	0.000(2)	0.004(2)
C(39)	4a	0.9025(2)	-0.1202(2)	0.1162(2)	0.022(2)	0.019(2)	0.038(2)	0.000(2)	-0.001(2)	0.004(2)
C(40)	4a	0.8966(2)	-0.1652(2)	0.0647(2)	0.025(2)	0.023(2)	0.029(2)	0.004(2)	-0.001(2)	0.001(2)
C(41)	4a	0.8295(2)	-0.2162(2)	0.0619(2)	0.028(2)	0.023(2)	0.030(2)	0.000(2)	-0.003(2)	-0.003(2)
C(42)	4a	0.7679(2)	-0.2249(2)	0.1065(2)	0.029(2)	0.026(2)	0.029(2)	0.000(2)	-0.002(2)	0.005(2)
C(43)	4a	0.7745(2)	-0.1767(2)	0.1560(2)	0.024(2)	0.030(2)	0.029(2)	-0.001(2)	0.000(2)	0.006(2)
C(44)	4a	0.6969(3)	-0.2846(2)	0.1031(2)	0.026(2)	0.031(2)	0.042(2)	-0.004(2)	0.001(2)	0.003(2)
C(45)	4a	0.6104(3)	-0.2546(3)	0.1252(3)	0.027(3)	0.061(3)	0.097(4)	-0.005(2)	0.000(3)	-0.010(3)
C(46)	4a	0.7231(3)	-0.3497(3)	0.1426(2)	0.044(3)	0.045(3)	0.075(3)	-0.014(2)	-0.009(2)	0.020(2)
C(47)	4a	0.6846(3)	-0.3133(3)	0.0386(2)	0.050(3)	0.056(3)	0.057(3)	-0.032(3)	-0.007(2)	-0.006(2)
C(48)	4a	0.9564(2)	-0.1558(2)	0.0117(2)	0.027(2)	0.025(2)	0.029(2)	0.001(2)	0.000(2)	-0.003(2)
C(49)	4a	0.9057(2)	-0.1360(2)	-0.0441(1)	0.020(2)	0.028(2)	0.024(2)	0.000(2)	0.002(2)	-0.001(2)
C(50)	4a	0.8752(2)	-0.0632(2)	-0.0516(2)	0.026(2)	0.030(2)	0.026(2)	0.001(2)	0.003(2)	-0.002(2)
C(51)	4a	0.8159(2)	-0.0455(2)	-0.0970(2)	0.024(2)	0.030(2)	0.030(2)	0.003(2)	0.004(2)	0.003(2)
C(52)	4a	0.7941(2)	-0.1017(2)	-0.1378(2)	0.025(2)	0.033(2)	0.030(2)	0.003(2)	-0.001(2)	0.004(2)
C(53)	4a	0.8267(2)	-0.1735(2)	-0.1341(2)	0.024(2)	0.031(2)	0.031(2)	-0.003(2)	-0.001(2)	0.001(2)
C(54)	4a	0.8818(2)	-0.1891(2)	-0.0862(2)	0.023(2)	0.032(2)	0.032(2)	0.003(2)	0.002(2)	0.000(2)
C(55)	4a	0.8050(3)	-0.2346(2)	-0.1803(2)	0.033(2)	0.031(2)	0.038(2)	0.001(2)	-0.007(2)	-0.002(2)
C(56)	4a	0.7278(4)	-0.2129(3)	-0.2200(2)	0.070(4)	0.056(3)	0.074(3)	0.017(3)	-0.040(3)	-0.027(3)
C(57)	4a	0.8833(3)	-0.2456(3)	-0.2219(2)	0.058(3)	0.048(3)	0.050(3)	-0.010(3)	0.009(2)	-0.012(2)
C(58)	4a	0.7852(3)	-0.3080(2)	-0.1491(2)	0.058(3)	0.039(2)	0.046(2)	-0.023(2)	-0.002(2)	-0.003(2)
C(59)	4a	0.7703(2)	0.0293(2)	-0.1004(2)	0.025(2)	0.030(2)	0.027(2)	0.005(2)	0.002(2)	0.002(2)
C(60)	4a	0.7016(2)	0.0375(2)	-0.0513(2)	0.025(2)	0.029(2)	0.027(2)	0.005(2)	0.002(2)	0.006(2)
C(61)	4a	0.7129(2)	0.0861(2)	-0.0027(2)	0.025(2)	0.023(2)	0.032(2)	0.005(2)	0.002(2)	0.006(2)
C(62)	4a	0.6533(2)	0.0868(2)	0.0445(2)	0.027(2)	0.027(2)	0.029(2)	0.002(2)	-0.002(2)	0.004(2)
C(63)	4a	0.5796(3)	0.0439(2)	0.0389(2)	0.028(2)	0.044(2)	0.030(2)	0.000(2)	0.003(2)	0.003(2)
C(64)	4a	0.5632(3)	-0.0015(2)	-0.0106(2)	0.030(2)	0.045(2)	0.033(2)	-0.009(2)	-0.003(2)	0.007(2)
C(65)	4a	0.6273(3)	-0.0045(2)	-0.0547(2)	0.034(2)	0.033(2)	0.026(2)	-0.001(2)	-0.001(2)	0.002(2)
C(66)	4a	0.4797(3)	-0.0455(3)	-0.0179(2)	0.035(2)	0.051(3)	0.035(2)	-0.014(2)	-0.002(2)	0.003(2)
C(67)	4a	0.4219(6)	-0.0432(5)	0.0357(3)	0.111(3)	0.124(3)	0.113(3)	-0.011(2)	0.005(2)	-0.006(2)
C(68)	4a	0.4986(4)	-0.1272(4)	-0.0332(4)	0.071(5)	0.075(5)	0.163(7)	-0.029(4)	-0.020(4)	0.019(4)
C(69)	4a	0.4297(4)	-0.0145(4)	-0.0714(3)	0.062(4)	0.107(5)	0.112(5)	-0.024(4)	-0.028(4)	0.011(4)
C(70)	4a	0.6702(3)	0.1299(2)	0.1024(2)	0.029(2)	0.027(2)	0.035(2)	0.003(2)	0.002(2)	0.003(2)

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