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Crystal structure of 25,27-dipropoxy-26,28-dihydroxycalix[4]arene, $C_{34}H_{36}O_4$

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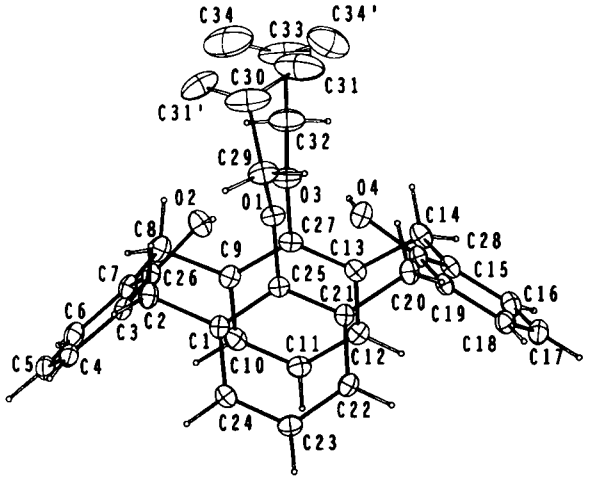

Source of material: The title compound was prepared by selective alkylation of parent 25,26,27,28-tetrahydroxycalix[4]arene according to published procedure (see ref. 1).

The molecule exists in a pinched-cone conformation stabilized by intramolecular hydrogen bonds O2–H···O1 (d(O2···O1) is 2.732(3) Å) and O4–H···O3 (d(O4···O3) is 2.759(2) Å). The methylsubstituents of the propoxy groups are disordered assuming two distinct positions with approximate probability 0.75:0.25. Hydrogen atoms of the disordered parts were not located.

$C_{34}H_{36}O_4$, trigonal, $R\bar{3}$ (No. 148), a =35.633(2) Å, c =11.694(5) Å, V =12858.7 Å^3, Z =18, $R(F)$ =0.047, $R_w(F^2)$ =0.095.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless block, size 0.27 x 0.25 x 0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.76 cm^{-1}
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	2θ/ω
$T_{measurement}$:	293 K
$2\theta_{max}$:	50°
$N(hkl)_{unique}$:	4556
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
$N(param)_{refined}$:	469
Programs:	SHELXS-86, SHELXL-93

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

Atom	Site	x	y	z	U_{iso}
H(1O2)	18*f*	0.201(1)	0.113(1)	0.303(3)	0.16(2)
H(1O4)	18*f*	0.188(2)	0.030(2)	0.185(4)	0.25(2)
H(21)	18*f*	0.2611(7)	0.2247(8)	0.324(2)	0.076(8)
H(22)	18*f*	0.2153(8)	0.1799(7)	0.300(2)	0.072(8)
H(41)	18*f*	0.2996(7)	0.2305(8)	0.490(2)	0.067(7)
H(51)	18*f*	0.3113(8)	0.1976(8)	0.655(2)	0.078(8)
H(61)	18*f*	0.2771(8)	0.1230(8)	0.674(2)	0.074(8)
H(81)	18*f*	0.1873(8)	0.0475(7)	0.530(2)	0.064(7)
H(82)	18*f*	0.2208(8)	0.0525(8)	0.621(2)	0.082(8)
H(101)	18*f*	0.2902(7)	0.0556(7)	0.567(2)	0.069(7)
H(111)	18*f*	0.3233(7)	0.0239(7)	0.446(2)	0.064(7)
H(121)	18*f*	0.2876(7)	–0.0094(7)	0.265(2)	0.068(7)
H(141)	18*f*	0.1825(8)	–0.0284(8)	0.184(2)	0.075(8)
H(142)	18*f*	0.2169(8)	–0.0473(9)	0.166(2)	0.087(8)
H(161)	18*f*	0.2598(7)	–0.0244(7)	0.008(2)	0.062(7)
H(171)	18*f*	0.2927(8)	0.0217(9)	–0.149(2)	0.096(9)
H(181)	18*f*	0.2833(8)	0.0829(8)	–0.176(2)	0.077(8)
H(201)	18*f*	0.2069(8)	0.0995(8)	–0.044(2)	0.074(8)
H(202)	18*f*	0.2457(7)	0.1199(7)	–0.129(2)	0.063(7)
H(221)	18*f*	0.3215(7)	0.1705(7)	–0.051(2)	0.070(7)
H(231)	18*f*	0.3632(8)	0.2258(7)	0.077(2)	0.064(7)
H(241)	18*f*	0.3303(8)	0.2380(8)	0.238(2)	0.084(8)
H(291)	18*f*	0.180(1)	0.147(1)	0.004(3)	0.12(1)
H(292)	18*f*	0.1867(9)	0.177(1)	0.118(2)	0.10(1)
H(321)	18*f*	0.144(1)	–0.027(1)	0.473(3)	0.12(1)
H(322)	18*f*	0.146(1)	–0.056(1)	0.365(3)	0.13(1)

Table 3. Final 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)	18*f*		0.20133(5)	0.12920(5)	0.1422(1)	0.054(1)	0.057(1)	0.060(1)	0.0303(8)	0.0031(7)	0.0184(7)
O(2)	18*f*		0.20445(6)	0.10031(6)	0.3564(2)	0.091(1)	0.059(1)	0.050(1)	0.033(1)	–0.0128(9)	0.0062(8)
O(3)	18*f*		0.17636(5)	0.01136(5)	0.3413(1)	0.052(1)	0.060(1)	0.084(1)	0.0240(9)	0.0037(8)	0.0315(9)
O(4)	18*f*		0.20099(6)	0.05131(5)	0.1308(1)	0.087(1)	0.060(1)	0.055(1)	0.044(1)	0.0149(9)	0.0142(8)
C(1)	18*f*		0.27007(8)	0.18778(7)	0.2028(2)	0.066(2)	0.045(1)	0.045(1)	0.035(1)	0.001(1)	0.006(1)
C(2)	18*f*		0.2493(1)	0.19275(9)	0.3114(2)	0.090(2)	0.059(2)	0.051(2)	0.050(2)	0.004(1)	0.000(1)
C(3)	18*f*		0.25710(7)	0.17202(8)	0.4157(2)	0.064(2)	0.059(2)	0.042(1)	0.039(1)	0.008(1)	–0.001(1)
C(4)	18*f*		0.28564(8)	0.19809(9)	0.5005(2)	0.067(2)	0.066(2)	0.055(2)	0.038(2)	0.006(1)	–0.008(1)
C(5)	18*f*		0.2927(1)	0.1799(1)	0.5965(2)	0.076(2)	0.089(2)	0.051(2)	0.049(2)	–0.007(1)	–0.016(2)
C(6)	18*f*		0.27215(9)	0.1363(1)	0.6084(2)	0.086(2)	0.094(2)	0.040(2)	0.060(2)	0.005(1)	0.004(1)
C(7)	18*f*		0.24268(8)	0.10852(8)	0.5274(2)	0.075(2)	0.069(2)	0.036(1)	0.046(1)	0.012(1)	0.007(1)
C(8)	18*f*		0.2189(1)	0.05990(9)	0.5428(2)	0.090(2)	0.074(2)	0.051(2)	0.045(2)	0.022(2)	0.020(1)
C(9)	18*f*		0.23702(8)	0.03776(7)	0.4689(2)	0.066(2)	0.053(1)	0.044(1)	0.031(1)	0.010(1)	0.016(1)
C(10)	18*f*		0.27584(8)	0.04008(8)	0.4959(2)	0.068(2)	0.061(2)	0.046(2)	0.026(1)	–0.005(1)	0.007(1)
C(11)	18*f*		0.29402(9)	0.02240(8)	0.4257(2)	0.057(2)	0.066(2)	0.058(2)	0.031(1)	–0.001(1)	0.010(1)
C(12)	18*f*		0.27391(8)	0.00296(8)	0.3241(2)	0.064(2)	0.057(2)	0.057(2)	0.032(1)	0.006(1)	0.010(1)
C(13)	18*f*		0.23470(8)	–0.00055(7)	0.2935(2)	0.062(2)	0.041(1)	0.047(1)	0.020(1)	–0.002(1)	0.010(1)
C(14)	18*f*		0.2138(1)	–0.02081(8)	0.1798(2)	0.083(2)	0.046(2)	0.058(2)	0.027(2)	–0.013(1)	0.001(1)
C(15)	18*f*		0.23410(7)	0.00951(7)	0.0785(2)	0.063(2)	0.043(1)	0.046(1)	0.025(1)	–0.015(1)	–0.005(1)
C(16)	18*f*		0.25821(9)	0.00209(9)	–0.0012(2)	0.079(2)	0.060(2)	0.062(2)	0.044(2)	–0.020(1)	–0.016(1)
C(17)	18*f*		0.27631(9)	0.02887(9)	–0.0944(2)	0.081(2)	0.078(2)	0.058(2)	0.049(2)	–0.002(1)	–0.010(1)
C(18)	18*f*		0.27009(8)	0.06336(9)	–0.1088(2)	0.068(2)	0.064(2)	0.044(1)	0.031(1)	–0.002(1)	–0.001(1)
C(19)	18*f*		0.24575(7)	0.07232(7)	–0.0328(2)	0.059(1)	0.048(1)	0.038(1)	0.026(1)	–0.010(1)	–0.004(1)
C(20)	18*f*		0.2380(1)	0.10990(8)	–0.0511(2)	0.082(2)	0.061(2)	0.035(1)	0.040(2)	–0.005(1)	0.004(1)
C(21)	18*f*		0.26421(7)	0.14792(7)	0.0280(2)	0.061(2)	0.046(1)	0.039(1)	0.030(1)	0.001(1)	0.0091(9)
C(22)	18*f*		0.30808(8)	0.17534(8)	0.0108(2)	0.070(2)	0.059(2)	0.047(1)	0.038(2)	0.013(1)	0.013(1)
C(23)	18*f*		0.33278(9)	0.20849(8)	0.0856(2)	0.053(2)	0.056(2)	0.067(2)	0.024(1)	0.005(1)	0.014(1)
C(24)	18*f*		0.31368(8)	0.21410(8)	0.1826(2)	0.066(2)	0.046(1)	0.057(2)	0.028(1)	–0.008(1)	0.001(1)
C(25)	18*f*		0.24564(7)	0.15581(7)	0.1230(2)	0.051(1)	0.042(1)	0.046(1)	0.025(1)	0.002(1)	0.013(1)
C(26)	18*f*		0.23562(8)	0.12758(8)	0.4314(2)	0.063(2)	0.060(2)	0.038(1)	0.036(1)	0.003(1)	–0.001(1)
C(27)	18*f*		0.21614(7)	0.01570(7)	0.3688(2)	0.051(1)	0.047(1)	0.055(2)	0.022(1)	0.001(1)	0.019(1)
C(28)	18*f*		0.22763(7)	0.04443(7)	0.0605(2)	0.059(1)	0.047(1)	0.039(1)	0.027(1)	–0.006(1)	–0.004(1)
C(29)	18*f*		0.17461(9)	0.1457(1)	0.0962(3)	0.066(2)	0.085(2)	0.096(3)	0.047(2)	0.002(2)	0.030(2)
C(30)	18*f*		0.1289(1)	0.1173(2)	0.1363(6)	0.068(3)	0.148(5)	0.229(6)	0.052(3)	0.010(3)	0.073(4)
C(31)	18*f*	0.74(1)	0.1087(2)	0.0752(2)	0.0994(8)	0.094(5)	0.102(6)	0.35(1)	0.029(4)	–0.023(5)	0.028(6)
C(31')	18*f*	0.26	0.1194(5)	0.1086(7)	0.244(1)	0.11(1)	0.19(2)	0.14(2)	0.09(1)	0.07(1)	0.08(2)
C(32)	18*f*		0.1398(1)	–0.0281(1)	0.3835(4)	0.059(2)	0.082(2)	0.135(3)	0.021(2)	0.009(2)	0.051(2)
C(33)	18*f*		0.0987(1)	–0.0291(2)	0.3436(8)	0.067(3)	0.165(5)	0.278(8)	0.036(3)	0.016(4)	0.103(5)
C(34)	18*f*	0.71(1)	0.0908(2)	0.0027(3)	0.381(1)	0.120(6)	0.165(8)	0.38(2)	0.098(6)	0.027(7)	0.047(8)
C(34')	18*f*	0.29	0.0935(5)	–0.0235(6)	0.236(1)	0.14(1)	0.16(2)	0.15(2)	0.03(1)	–0.05(1)	0.02(1)

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