Crystal structure of 11,23-bis(diisopropylphosphono)-25,26,27,28-tetramethoxycalix[4]arene, $C_{44}H_{58}O_{10}P_2$

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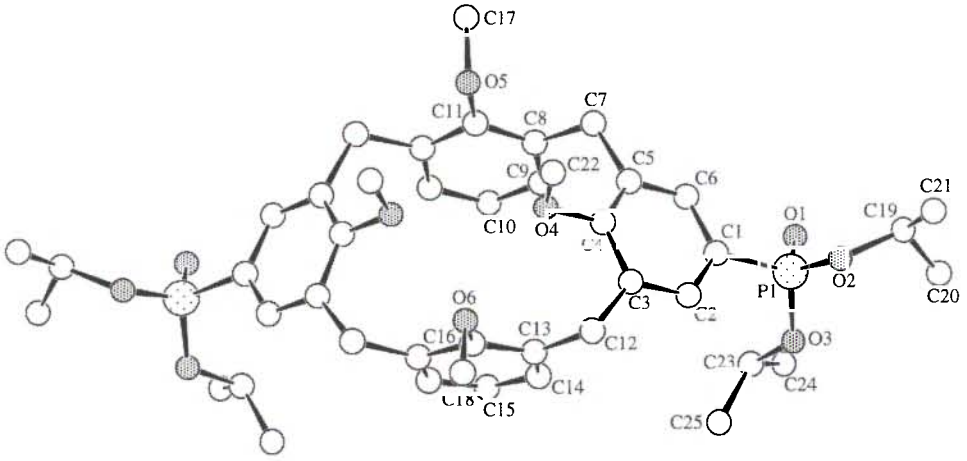

Abstract

$C_{44}H_{58}O_{10}P_2$, orthorhombic, $Pmn2_1$ (No. 31), $a = 23.7325(5)$ Å, $b = 7.9631(3)$ Å, $c = 12.3151(9)$ Å, $V = 2327.4$ Å^3, $Z = 2$, $R_{gt}(F) = 0.077$, $wR(F^2) = 0.235$, $T = 293$ K.

Source of material

The tetraisopropyl *O*-methylated calix[4]arene diphosphonate was synthesized from calix[4]arene in 4 steps [1].

Discussion

The isopropoxy groups connected on the phosphorus atoms are disordered. Because of clarity only a ordered molecule is presented. Hydrogen atoms are left out.

Table 1. Data collection and handling.

Crystal:	colourless, prismatic, size 0.15 × 0.18 × 0.47 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	1.45 cm^{-1}
Diffractometer, scan mode:	Siemens SMART CCD, ω
$2\theta_{max}$:	46.64°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9752, 3303
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2915
$N(param)_{refined}$:	274
Programs:	SHELXS-86 [2], SHELXL-97 [3], CELLGRAPH [4]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1)	4*b*		0.2048	0.5934	0.3371	0.070
H(2)	4*b*		0.2016	0.5800	0.6632	0.081
H(3)	4*b*		0.1001	0.8734	0.7078	0.081
H(4)	4*b*		0.1296	0.7191	0.7635	0.081
H(5)	4*b*		0.0848	0.4307	0.6765	0.077
H(6)	2*a*		0	0.2818	0.6778	0.074
H(7)	4*b*		0.0987	0.8746	0.2915	0.078
H(8)	4*b*		0.1289	0.7240	0.2334	0.078
H(9)	4*b*		0.0830	0.4251	0.3100	0.087
H(10)	2*a*		0	0.2871	0.3306	0.101
H(11)	2*a*		0	1.0804	0.8494	0.109
H(12)	4*b*	0.5	0.0330	0.9154	0.8771	0.109
H(13)	4*b*	0.5	-0.0330	0.9154	0.8771	0.109
H(14)	2*a*		0	1.0799	0.1497	0.145
H(15)	4*b*	0.5	-0.0330	0.9144	0.1233	0.145
H(16)	4*b*	0.5	0.0330	0.9144	0.1233	0.145
H(17)	4*b*	0.612(2)	0.3722	0.5736	0.6077	0.117
H(18)	4*b*	0.612	0.3856	0.3203	0.5191	0.139
H(19)	4*b*	0.612	0.4442	0.4078	0.5347	0.139
H(20)	4*b*	0.612	0.4146	0.4113	0.4210	0.139
H(21)	4*b*	0.612	0.3883	0.8251	0.5349	0.139
H(22)	4*b*	0.612	0.4159	0.7427	0.4323	0.139
H(23)	4*b*	0.612	0.4452	0.7262	0.5460	0.139
H(24)	4*b*		0.0451	1.1054	0.4988	0.132
H(25)	4*b*		0.1030	1.0750	0.4408	0.132
H(26)	4*b*		0.0994	1.0720	0.5679	0.132
H(27)	4*b*	0.612	0.2040	0.2267	0.4663	0.117
H(28)	4*b*	0.612	0.2759	0.0711	0.5302	0.139
H(29)	4*b*	0.612	0.2883	0.0014	0.4136	0.139
H(30)	4*b*	0.612	0.2318	-0.0447	0.4722	0.139
H(31)	4*b*	0.612	0.1888	0.2929	0.2877	0.139

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Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(32)	4*b*	0.612	0.1729	0.1043	0.3079	0.139
H(33)	4*b*	0.612	0.2298	0.1499	0.2502	0.139
H(17')	4*b*	0.389(2)	0.3646	0.5309	0.3768	0.117
H(18')	4*b*	0.389	0.4028	0.3451	0.5030	0.139
H(19')	4*b*	0.389	0.4268	0.4947	0.5715	0.139
H(20')	4*b*	0.389	0.4519	0.4555	0.4565	0.139
H(21')	4*b*	0.389	0.3687	0.8205	0.4051	0.139
H(22')	4*b*	0.389	0.4307	0.7537	0.3961	0.139

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(23')	4*b*	0.389	0.4050	0.7952	0.5102	0.139
H(27')	4*b*	0.389	0.2159	0.1948	0.5241	0.117
H(28')	4*b*	0.389	0.3036	0.0763	0.4842	0.139
H(29')	4*b*	0.389	0.2692	-0.0579	0.5498	0.139
H(30')	4*b*	0.389	0.3160	0.0480	0.6079	0.139
H(31')	4*b*	0.389	0.1906	0.2397	0.7077	0.139
H(32')	4*b*	0.389	0.2434	0.1374	0.7460	0.139
H(33')	4*b*	0.389	0.1940	0.0461	0.6856	0.139

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}
P(1)	4*b*		0.27572(3)	0.4530(1)	0.5001(2)	0.0366(4)	0.0715(5)	0.1226(7)	0.0031(3)	-0.0005(7)	0.0035(9)
O(1)	4*b*	0.612(2)	0.2781(2)	0.3485(7)	0.6070(4)	0.106(2)	0.167(3)	0.138(3)	0.063(2)	0.035(2)	0.072(3)
O(2)	4*b*	0.612	0.3223(1)	0.5792(5)	0.4772(4)	0.036(1)	0.094(2)	0.109(3)	-0.010(1)	-0.023(2)	0.063(3)
O(3)	4*b*	0.612	0.2751(1)	0.3318(4)	0.4062(4)	0.031(2)	0.032(2)	0.155(3)	-0.022(1)	0.007(2)	-0.031(2)
O(4)	4*b*		0.06884(7)	0.8689(3)	0.4989(3)	0.0433(9)	0.091(1)	0.069(1)	0.0180(9)	-0.004(2)	-0.009(2)
O(5)	2*a*		0	0.9157(4)	0.7318(3)	0.046(2)	0.064(2)	0.064(2)	0	0	-0.006(2)
O(6)	2*a*		0	0.9170(4)	0.2700(3)	0.066(2)	0.060(2)	0.053(2)	0	0	0.006(2)
C(1)	4*b*		0.2125(1)	0.5754(3)	0.5001(3)	0.032(1)	0.067(2)	0.076(2)	-0.003(1)	0.002(2)	0.017(3)
C(2)	4*b*		0.1879(1)	0.6248(4)	0.4022(2)	0.045(2)	0.063(2)	0.053(2)	-0.006(2)	0.013(1)	0.001(2)
C(3)	4*b*		0.1394(1)	0.7184(4)	0.3997(2)	0.052(2)	0.062(2)	0.049(2)	-0.004(2)	0.012(1)	-0.000(2)
C(4)	4*b*		0.1166(1)	0.7713(3)	0.4999(3)	0.034(1)	0.072(2)	0.063(2)	-0.000(1)	0.007(2)	-0.021(2)
C(5)	4*b*		0.1381(1)	0.7166(5)	0.5978(3)	0.023(1)	0.092(2)	0.062(2)	-0.003(2)	0.003(1)	-0.005(2)
C(6)	4*b*		0.1865(1)	0.6178(5)	0.5979(3)	0.033(2)	0.076(2)	0.079(2)	0.000(2)	-0.005(2)	0.005(2)
C(7)	4*b*		0.1069(2)	0.7536(4)	0.7019(3)	0.066(2)	0.066(2)	0.056(2)	0.001(2)	-0.002(2)	-0.010(2)
C(8)	4*b*		0.0508(1)	0.6594(4)	0.7032(2)	0.047(2)	0.068(2)	0.026(1)	0.001(2)	-0.002(1)	0.011(1)
C(9)	4*b*		0.0509(1)	0.4872(4)	0.6859(3)	0.052(2)	0.071(2)	0.054(2)	0.014(2)	-0.003(2)	-0.009(2)
C(10)	2*a*		0	0.3984(6)	0.6824(4)	0.056(3)	0.057(3)	0.059(3)	0	0	-0.013(2)
C(11)	2*a*		0	0.7418(6)	0.7169(3)	0.055(3)	0.069(3)	0.036(2)	0	0	-0.007(2)
C(12)	4*b*		0.1064(1)	0.7552(5)	0.2961(3)	0.033(2)	0.099(3)	0.048(2)	-0.002(2)	0.009(1)	0.008(2)
C(13)	4*b*		0.0516(1)	0.6595(4)	0.2942(3)	0.046(2)	0.073(2)	0.057(2)	0.004(2)	0.004(2)	0.027(2)
C(14)	4*b*		0.0495(2)	0.4854(5)	0.3077(3)	0.067(2)	0.071(2)	0.062(2)	0.010(2)	-0.002(2)	0.002(2)
C(15)	2*a*		0	0.4021(7)	0.3173(5)	0.095(4)	0.058(3)	0.081(4)	0	0	-0.004(3)
C(16)	2*a*		0	0.7424(5)	0.2869(3)	0.048(2)	0.047(2)	0.030(2)	0	0	0.001(2)
C(17)	2*a*		0	0.9603(7)	0.8427(4)	0.083(4)	0.088(4)	0.081(4)	0	0	-0.034(3)
C(18)	2*a*		0	0.9599(8)	0.1573(5)	0.194(9)	0.088(4)	0.052(3)	0	0	0.030(3)
C(19)	4*b*	0.612	0.3787(2)	0.5785(7)	0.5291(6)	0.054(2)	0.090(2)	0.125(3)	-0.016(2)	0.017(3)	-0.027(3)
C(20)	4*b*	0.612	0.4086(3)	0.4140(7)	0.498(1)	0.078(2)	0.115(2)	0.128(2)	-0.016(2)	-0.001(2)	-0.004(3)
C(21)	4*b*	0.612	0.4096(2)	0.7308(7)	0.509(1)	0.078(2)	0.115(2)	0.128(2)	-0.016(2)	-0.001(2)	-0.004(3)
C(22)	4*b*		0.0800(2)	1.0447(4)	0.5019(7)	0.086(2)	0.091(2)	0.127(3)	0.031(2)	0.021(4)	0.019(4)
C(23)	4*b*	0.612	0.2326(3)	0.1899(8)	0.4139(6)	0.054(2)	0.090(2)	0.125(3)	-0.016(2)	0.017(3)	-0.027(3)
C(24)	4*b*	0.612	0.2595(3)	0.0416(8)	0.4616(6)	0.078(2)	0.115(2)	0.128(2)	-0.016(2)	-0.001(2)	-0.004(3)
C(25)	4*b*	0.612	0.2033(3)	0.184(1)	0.3050(6)	0.078(2)	0.115(2)	0.128(2)	-0.016(2)	-0.001(2)	-0.004(3)
O(1')	4*b*	0.389(2)	0.2861(2)	0.3880(7)	0.3858(5)	0.031(2)	0.032(2)	0.155(3)	-0.022(1)	0.007(2)	-0.031(2)
O(2')	4*b*	0.389	0.3253(2)	0.5756(7)	0.5232(6)	0.036(1)	0.094(2)	0.109(3)	-0.010(1)	-0.023(2)	0.063(3)
O(3')	4*b*	0.389	0.2789(4)	0.335(1)	0.5923(7)	0.106(2)	0.167(3)	0.138(3)	0.063(2)	0.035(2)	0.072(3)
C(19')	4*b*	0.389	0.3756(3)	0.573(1)	0.4484(9)	0.054(2)	0.090(2)	0.125(3)	-0.016(2)	0.017(3)	-0.027(3)
C(20')	4*b*	0.389	0.4182(3)	0.457(1)	0.500(2)	0.078(2)	0.115(2)	0.128(2)	-0.016(2)	-0.001(2)	-0.004(3)
C(21')	4*b*	0.389	0.3970(4)	0.752(1)	0.439(1)	0.078(2)	0.115(2)	0.128(2)	-0.016(2)	-0.001(2)	-0.004(3)
C(23')	4*b*	0.389	0.2438(3)	0.182(1)	0.5822(8)	0.054(2)	0.090(2)	0.125(3)	-0.016(2)	0.017(3)	-0.027(3)
C(24')	4*b*	0.389	0.2871(4)	0.050(1)	0.553(1)	0.078(2)	0.115(2)	0.128(2)	-0.016(2)	-0.001(2)	-0.004(3)
C(25')	4*b*	0.389	0.2153(4)	0.148(2)	0.6903(8)	0.078(2)	0.115(2)	0.128(2)	-0.016(2)	-0.001(2)	-0.004(3)

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