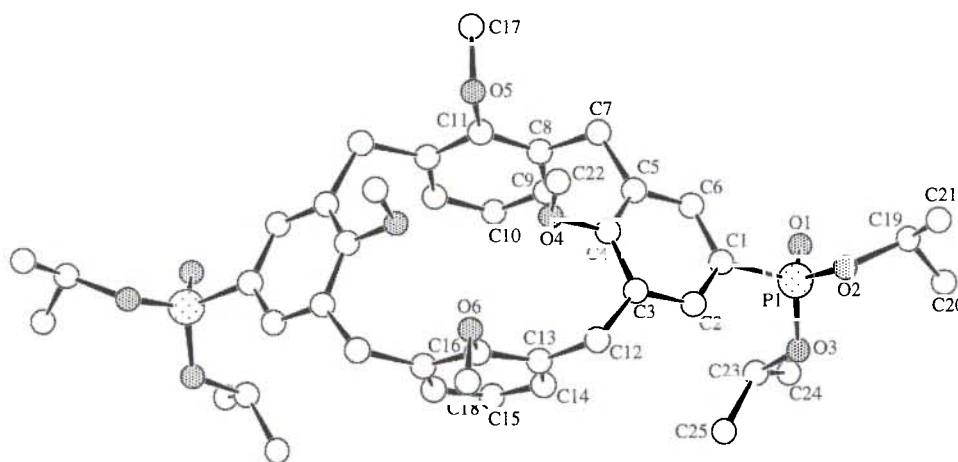


Crystal structure of 11,23-bis(diisopropylphosphono)-25,26,27,28-tetramethoxycalix[4]arene, $C_{44}H_{58}O_{10}P_2$

G. Reck^I, M. Schneider^{II}, J. Gloede^{II} and D. Vollhardt^{* III}^I Bundesanstalt für Materialforschung und -prüfung, Rudower Chaussee 5, D-12484 Berlin, Germany^{II} Institut für Angewandte Chemie e.V., Rudower Chaussee 5, D-12484 Berlin, Germany^{III} Max-Planck-Institut für Kolloid- und Grenzflächenforschung, Rudower Chaussee 5, D-12489 Berlin, Germany

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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, $V = 2327.4$ Å³, $Z = 2$, $R_{gt}(F) = 0.077$, $wR(F^2) = 0.235$, $T = 293$ 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 Å²).

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

* Correspondence author (e-mail: vollh@mpikg.fta-berlin.de)

Table 2. Continued.

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

Table 2. Continued.

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

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

Atom	Site	Occ.	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
P(1)	4b		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)	4b	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)	4b	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)	4b	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)	4b		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)	2a		0	0.9157(4)	0.7318(3)	0.046(2)	0.064(2)	0.064(2)	0	0	-0.006(2)
O(6)	2a		0	0.9170(4)	0.2700(3)	0.066(2)	0.060(2)	0.053(2)	0	0	0.006(2)
C(1)	4b		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)	4b		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)	4b		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)	4b		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)	4b		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)	4b		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)	4b		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)	4b		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)	4b		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)	2a		0	0.3984(6)	0.6824(4)	0.056(3)	0.057(3)	0.059(3)	0	0	-0.013(2)
C(11)	2a		0	0.7418(6)	0.7169(3)	0.055(3)	0.069(3)	0.036(2)	0	0	-0.007(2)
C(12)	4b		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)	4b		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)	4b		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)	2a		0	0.4021(7)	0.3173(5)	0.095(4)	0.058(3)	0.081(4)	0	0	-0.004(3)
C(16)	2a		0	0.7424(5)	0.2869(3)	0.048(2)	0.047(2)	0.030(2)	0	0	0.001(2)
C(17)	2a		0	0.9603(7)	0.8427(4)	0.083(4)	0.088(4)	0.081(4)	0	0	-0.034(3)
C(18)	2a		0	0.9599(8)	0.1573(5)	0.194(9)	0.088(4)	0.052(3)	0	0	0.030(3)
C(19)	4b	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)	4b	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)	4b	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)	4b		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)	4b	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)	4b	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)	4b	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')	4b	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')	4b	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')	4b	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')	4b	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')	4b	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')	4b	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')	4b	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')	4b	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')	4b	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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