Crystal structure of 1-hexynyl(diphenyl)phosphine oxide, $C_{18}H_{19}OP$

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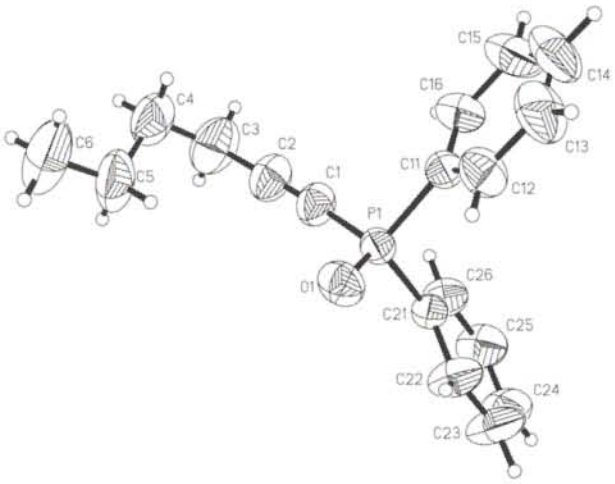

Abstract

$C_{18}H_{19}OP$, monoclinic, *C*1*c*1 (No. 9), *a* = 16.144(3) Å, *b* = 6.239(1) Å, *c* = 16.387(4) Å, β = 106.56(2)°, *V* = 1582.1 Å^3, *Z* = 4, $R_{gt}(F)$ = 0.040, $wR(F^2)$ = 0.097, *T* = 291 K.

Source of material

The compound was prepared by oxidation of 1-hexynyl(diphenyl)-phosphine with 30% hydrogen peroxide solution at 273 K in dichloromethane [1]. The solvent was removed and the residue chromatographed on a silica gel column with diethylether as eluent to give a white solid (5.76 g, 40%), mp 327 K – 328 K. Colourless crystals were grown by slow evaporation from the diethyl ether solution at room temperature.

Discussion

There has been some discussion in the literature on steric and electronic interactions in the related but symmetrical compounds bis(diphenylphosphino)acetylene [2] and bis(diphenylphosphonyl)acetylene [3]. It thus seemed interesting to compare the skeletal geometry of the latter with that of the unsymmetrical title compound. We find that the bond lengths in the P–C–C fragment are both shorter than in the bisphosphonylacetylene, though the deviations from linearity of the acetylene system are slightly larger.

The last three atoms of the hexyl group are disordered with occupancy factors of 0.67 for C4, C5, C6 and 0.33 for C4′, C5′ and C6′.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.15 × 0.35 × 0.55 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	1.67 cm^{-1}
Diffractometer, scan mode:	Nonius Mach3, ω/2θ
$2\theta_{max}$:	54.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1863, 1863
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1544
$N(param)_{refined}$:	209
Programs:	SHELXS-97 [4], SHELXL-97 [5], SHELXTL-Plus [6]

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(3A)	4*a*		0.1090	–0.0188	0.1180	0.151
H(3B)	4*a*		0.0381	0.0957	0.1505	0.151
H(4A)	4*a*	0.67	0.0191	0.1771	0.0124	0.143
H(4B)	4*a*	0.67	0.1143	0.2622	0.0328	0.143
H(5A)	4*a*	0.67	–0.0155	0.4601	0.0945	0.159
H(5B)	4*a*	0.67	0.0798	0.5439	0.1165	0.159
H(6A)	4*a*	0.67	–0.0073	0.7500	0.0139	0.199
H(6B)	4*a*	0.67	–0.0360	0.5487	–0.0446	0.199
H(6C)	4*a*	0.67	0.0596	0.6311	–0.0231	0.199
H(4'1)	4*a*	0.33	–0.0033	0.3269	0.1270	0.147
H(4'2)	4*a*	0.33	–0.0211	0.1782	0.0461	0.147
H(5'1)	4*a*	0.33	0.1277	0.5239	0.0685	0.210
H(5'2)	4*a*	0.33	0.0778	0.4220	–0.0227	0.210
H(6'1)	4*a*	0.33	–0.0177	0.7243	0.0100	0.219
H(6'2)	4*a*	0.33	–0.0158	0.6033	0.0943	0.219
H(6'3)	4*a*	0.33	–0.0645	0.5028	0.0059	0.219
H(12)	4*a*		0.4460	0.6561	0.3480	0.101(5)
H(13)	4*a*		0.5807	0.5577	0.3373	0.101
H(14)	4*a*		0.6049	0.2188	0.2986	0.101
H(15)	4*a*		0.4958	–0.0273	0.2673	0.101
H(16)	4*a*		0.3598	0.0589	0.2802	0.101
H(22)	4*a*		0.3153	0.6643	0.4831	0.101
H(23)	4*a*		0.3192	0.5694	0.6200	0.101
H(24)	4*a*		0.2874	0.2261	0.6508	0.101
H(25)	4*a*		0.2568	–0.0276	0.5455	0.101
H(26)	4*a*		0.2571	0.0573	0.4088	0.101

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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*a*		0.28763(4)	0.4561(1)	0.32750(4)	0.0405(3)	0.0471(3)	0.0436(3)	0.0034(4)	0.0124(2)	0.0029(4)
O(1)	4*a*		0.2760(2)	0.6908(4)	0.3157(2)	0.072(2)	0.050(1)	0.071(2)	0.012(1)	0.027(1)	0.010(1)
C(1)	4*a*		0.2108(2)	0.3003(6)	0.2557(2)	0.049(2)	0.077(2)	0.053(2)	–0.006(1)	0.011(1)	0.002(2)
C(2)	4*a*		0.1564(3)	0.2154(7)	0.2015(2)	0.065(2)	0.082(2)	0.060(2)	–0.012(2)	0.008(2)	–0.002(2)
C(3)	4*a*		0.0890(4)	0.120(1)	0.1316(3)	0.101(3)	0.099(3)	0.083(3)	–0.027(3)	–0.004(3)	–0.017(3)
C(4)	4*a*	0.67	0.0651(8)	0.248(2)	0.0551(6)	0.095(6)	0.103(7)	0.067(4)	0.000(5)	–0.012(4)	–0.021(4)
C(5)	4*a*	0.67	0.0341(8)	0.475(2)	0.0727(6)	0.098(6)	0.102(7)	0.084(5)	0.011(5)	–0.030(5)	–0.008(4)
C(6)	4*a*	0.67	0.011(1)	0.613(2)	–0.0015(8)	0.17(2)	0.100(7)	0.086(7)	–0.006(8)	–0.030(8)	0.012(6)
C(4')	4*a*	0.33	0.023(1)	0.257(3)	0.088(2)	0.056(9)	0.10(1)	0.10(1)	0.005(9)	–0.028(8)	–0.04(1)
C(5')	4*a*	0.33	0.074(1)	0.461(6)	0.034(2)	0.07(1)	0.25(4)	0.09(1)	–0.04(2)	0.011(9)	–0.02(2)
C(6')	4*a*	0.33	–0.015(2)	0.586(7)	0.036(2)	0.13(2)	0.18(3)	0.10(2)	–0.03(2)	–0.02(2)	0.00(2)
C(11)	4*a*		0.3890(2)	0.3658(5)	0.3147(2)	0.046(1)	0.051(2)	0.042(1)	0.003(1)	0.016(1)	0.006(1)
C(12)	4*a*		0.4557(2)	0.5173(7)	0.3321(3)	0.051(2)	0.069(2)	0.086(2)	–0.006(2)	0.025(2)	–0.002(2)
C(13)	4*a*		0.5360(3)	0.458(1)	0.3256(4)	0.051(2)	0.117(4)	0.119(4)	–0.005(2)	0.032(2)	0.016(3)
C(14)	4*a*		0.5505(3)	0.257(1)	0.3023(4)	0.067(2)	0.132(4)	0.116(4)	0.039(3)	0.054(2)	0.050(3)
C(15)	4*a*		0.4855(4)	0.1098(9)	0.2844(3)	0.106(4)	0.086(3)	0.105(3)	0.041(3)	0.065(3)	0.023(3)
C(16)	4*a*		0.4036(2)	0.1611(6)	0.2913(2)	0.074(2)	0.058(2)	0.075(2)	0.007(2)	0.036(2)	–0.003(2)
C(21)	4*a*		0.2850(2)	0.3703(5)	0.4317(2)	0.044(1)	0.055(2)	0.044(1)	0.002(1)	0.016(1)	–0.001(1)
C(22)	4*a*		0.3038(3)	0.5237(6)	0.4954(2)	0.115(3)	0.057(2)	0.057(2)	–0.005(2)	0.033(2)	–0.009(2)
C(23)	4*a*		0.3055(4)	0.4669(9)	0.5770(3)	0.147(5)	0.079(3)	0.054(2)	0.004(3)	0.041(2)	–0.015(2)
C(24)	4*a*		0.2873(3)	0.2625(8)	0.5958(2)	0.113(3)	0.094(3)	0.054(2)	0.021(3)	0.040(2)	0.012(2)
C(25)	4*a*		0.2691(3)	0.1119(7)	0.5328(3)	0.109(3)	0.069(2)	0.074(2)	0.003(2)	0.047(2)	0.019(2)
C(26)	4*a*		0.2685(2)	0.1623(6)	0.4508(2)	0.074(2)	0.059(2)	0.060(2)	–0.007(2)	0.029(2)	–0.002(1)

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