Crystal structure of (*S*)-1-[bis(diphenylphosphino)amino]-2-(methoxymethyl)pyrrolidine, $C_{30}H_{32}N_2OP_2$

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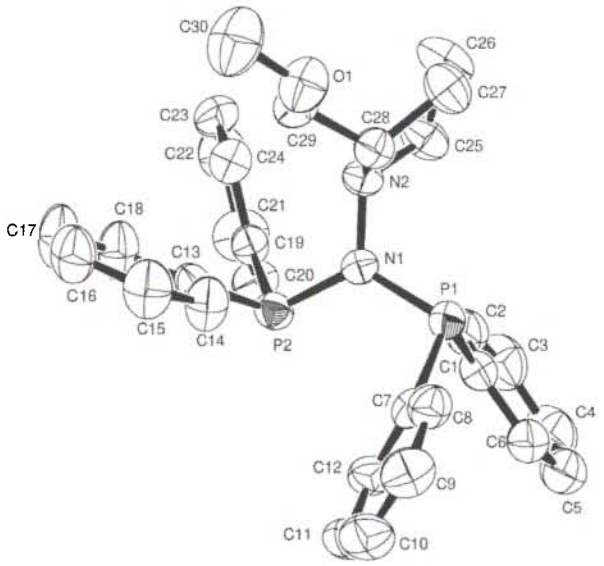

Abstract

$C_{30}H_{32}N_2OP_2$, monoclinic, $P12_11$ (No. 4), $a = 9.405(3)$ Å, $b = 14.531(3)$ Å, $c = 10.578(4)$ Å, $\beta = 110.24(3)°$, $V = 1356.4$ Å^3, $Z = 2$, $R_{gt}(F) = 0.041$, $wR(F) = 0.029$, $T = 293$ K.

Source of material

The title compound was prepared according to the method of Babu et al. [1] and purified by flash chromatography (EtOAc/hexane, 2/8) and recrystallization (MeOH) to give in 29% yield a colorless solid: mp 459 K – 461 K. Crystals suitable for X-ray diffraction were obtained by slow recrystallization from ethanol.

Discussion

$C_{29}H_{30}N_2OP_2$ is a chiral diphosphinoamine, in which the chirality resides on the methoxymethylpyrrolidino group attached to nitrogen. The structure shows a P1–N1–P2 angle of 124.04(9)° and N1–P1, N1–P2 bond lengths of 1.722(2) Å and 1.717(2) Å, respectively. These data indicate partial double bond character for the N1–P1 and N1–P2 bonds and quasi planar geometry around the N1 atom. Diphosphinoamines with quasi planar nitrogens can adopt two different conformations, C_s and C_{2v} [2]. In each case, the lone pairs on N and P are orthogonal. NMR and X-ray diffraction analyses have shown that the C_{2v} conformation is favored when R and R′ groups are relatively small, while the C_s conformation is preferred when the R and/or R′ substituents are large. As expected, R and R′ being rather large, the C_s conformation was found for this ligand in the present investigation.

Table 1. Data collection and handling.

Crystal:	colorless, monoclinic prism, size 0.27 × 0.28 × 0.30 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	1.85 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω
$2\theta_{max}$:	59.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4299, 4133
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 1\ \sigma(I_{obs})$, 3243
$N(param)_{refined}$:	315
Programs:	SIR [3], TEXSAN [4], ORTEPII [5]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2a	0.8312	0.8230	0.4691	0.072
H(2)	2a	0.6881	0.8925	0.5796	0.145
H(3)	2a	0.5075	1.0070	0.4785	0.097
H(4)	2a	0.4191	1.0278	0.2407	0.105
H(5)	2a	0.5307	0.9309	0.1078	0.104
H(6)	2a	0.7710	0.8144	–0.0899	0.065
H(7)	2a	0.5962	0.7720	–0.2889	0.093
H(8)	2a	0.3627	0.7076	–0.2772	0.076
H(9)	2a	0.2976	0.6941	–0.0983	0.099
H(10)	2a	0.4987	0.7349	0.1165	0.065
H(11)	2a	0.7796	0.5922	0.0507	0.074
H(12)	2a	0.8505	0.4635	–0.0582	0.082
H(13)	2a	0.9135	0.3252	0.0546	0.085
H(14)	2a	0.9354	0.3193	0.2765	0.079
H(15)	2a	0.8943	0.4472	0.3936	0.077
H(16)	2a	0.6903	0.6614	0.5398	0.067
H(17)	2a	0.7961	0.6401	0.7717	0.083
H(18)	2a	1.0432	0.5949	0.8727	0.074
H(19)	2a	1.1822	0.5561	0.7305	0.057
H(20)	2a	1.0876	0.5746	0.4981	0.058
H(21)	2a	1.1185	0.7713	0.5203	0.074
H(22)	2a	1.0430	0.8559	0.3883	0.074
H(23)	2a	1.3106	0.8572	0.4556	0.102
H(24)	2a	1.3318	0.7451	0.4625	0.106
H(25)	2a	1.3235	0.7790	0.2429	0.072
H(26)	2a	1.1852	0.8531	0.2218	0.094
H(27)	2a	1.0441	0.7294	0.1330	0.056
H(28)	2a	1.1071	0.5713	0.2151	0.080
H(29)	2a	1.2697	0.6164	0.3181	0.080
H(30)	2a	1.3446	0.5278	0.0320	0.136
H(31)	2a	1.2717	0.4825	0.1262	0.136
H(32)	2a	1.4155	0.5310	0.1950	0.143

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P(1)	2*a*	0.80160(6)	0.8184	0.18406(5)	0.0441(3)	0.0391(3)	0.0476(3)	–0.0013(3)	0.0108(2)	0.0038(3)
P(2)	2*a*	0.77587(5)	0.63412(5)	0.30974(5)	0.0406(2)	0.0449(3)	0.0394(2)	–0.0046(3)	0.0090(2)	0.0021(3)
O(1)	2*a*	1.2494(2)	0.6165(1)	0.1212(1)	0.0853(9)	0.066(1)	0.0687(8)	0.0117(9)	0.0452(7)	0.0015(8)
N(1)	2*a*	0.8796(2)	0.7205(1)	0.2726(1)	0.0358(8)	0.0395(9)	0.0402(8)	0.0009(7)	0.0080(6)	0.0029(7)
N(2)	2*a*	1.0424(2)	0.7153(1)	0.3251(1)	0.0367(8)	0.0479(9)	0.0349(8)	–0.0030(8)	0.0090(6)	–0.0023(8)
C(1)	2*a*	0.6920(2)	0.8700(1)	0.2797(2)	0.044(1)	0.039(1)	0.055(1)	–0.006(1)	0.0102(9)	–0.005(1)
C(2)	2*a*	0.7418(2)	0.8636(2)	0.4186(2)	0.058(1)	0.052(1)	0.064(1)	–0.002(1)	0.022(1)	–0.010(1)
C(3)	2*a*	0.6761(3)	0.9153(2)	0.4940(2)	0.082(1)	0.066(2)	0.085(2)	–0.011(1)	0.043(1)	–0.018(1)
C(4)	2*a*	0.5575(3)	0.9735(2)	0.4287(3)	0.072(2)	0.070(2)	0.114(2)	–0.007(1)	0.046(1)	–0.037(2)
C(5)	2*a*	0.5066(3)	0.9806(2)	0.2918(3)	0.057(1)	0.054(1)	0.116(2)	0.001(1)	0.026(1)	–0.017(1)
C(6)	2*a*	0.5732(2)	0.9297(2)	0.2163(2)	0.052(1)	0.048(1)	0.087(2)	–0.001(1)	0.020(1)	–0.004(1)
C(7)	2*a*	0.6512(2)	0.7776(1)	0.0331(2)	0.047(1)	0.044(1)	0.043(1)	0.009(1)	0.0114(8)	0.0090(9)
C(8)	2*a*	0.6767(2)	0.7870(2)	–0.0885(2)	0.055(1)	0.057(1)	0.050(1)	0.003(1)	0.0159(9)	0.011(1)
C(9)	2*a*	0.5653(3)	0.7610(2)	–0.2093(2)	0.077(2)	0.085(2)	0.045(1)	0.007(1)	0.019(1)	0.008(1)
C(10)	2*a*	0.4326(3)	0.7251(2)	–0.2083(2)	0.065(1)	0.088(2)	0.045(1)	–0.003(1)	–0.002(1)	–0.001(1)
C(11)	2*a*	0.4056(2)	0.7147(2)	–0.0892(2)	0.049(1)	0.072(2)	0.054(1)	–0.006(1)	0.003(1)	0.000(1)
C(12)	2*a*	0.5140(2)	0.7414(1)	0.0302(2)	0.046(1)	0.060(1)	0.044(1)	–0.001(1)	0.0100(9)	0.006(1)
C(13)	2*a*	0.8305(2)	0.5336(1)	0.2338(2)	0.048(1)	0.043(1)	0.041(1)	–0.013(1)	0.0095(9)	–0.0019(9)
C(14)	2*a*	0.8157(3)	0.5387(2)	0.0983(2)	0.091(2)	0.050(1)	0.039(1)	–0.001(1)	0.014(1)	–0.001(1)
C(15)	2*a*	0.8473(3)	0.4647(2)	0.0327(2)	0.105(2)	0.067(2)	0.039(1)	–0.004(2)	0.018(1)	–0.008(1)
C(16)	2*a*	0.8945(3)	0.3823(2)	0.0988(2)	0.096(2)	0.051(1)	0.058(1)	–0.005(1)	0.022(1)	–0.016(1)
C(17)	2*a*	0.9053(3)	0.3753(2)	0.2301(2)	0.122(2)	0.043(1)	0.059(1)	0.000(1)	0.021(1)	–0.002(1)
C(18)	2*a*	0.8736(3)	0.4503(2)	0.2968(2)	0.103(2)	0.047(1)	0.041(1)	–0.004(1)	0.022(1)	0.002(1)
C(19)	2*a*	0.8708(2)	0.6141(1)	0.4903(2)	0.046(1)	0.039(1)	0.0378(9)	–0.0039(9)	0.0125(8)	0.0002(9)
C(20)	2*a*	0.7868(2)	0.6332(2)	0.5730(2)	0.050(1)	0.061(1)	0.052(1)	0.005(1)	0.0214(8)	0.009(1)
C(21)	2*a*	0.8509(2)	0.6230(2)	0.7117(2)	0.074(1)	0.087(2)	0.048(1)	0.005(1)	0.0314(9)	0.000(1)
C(22)	2*a*	0.9982(3)	0.5947(2)	0.7692(2)	0.081(2)	0.067(2)	0.043(1)	–0.005(1)	0.017(1)	0.004(1)
C(23)	2*a*	1.0815(2)	0.5752(2)	0.6895(2)	0.051(1)	0.055(1)	0.053(1)	0.006(1)	0.004(1)	0.007(1)
C(24)	2*a*	1.0194(2)	0.5852(1)	0.5511(2)	0.054(1)	0.049(1)	0.046(1)	0.003(1)	0.0200(9)	0.001(1)
C(25)	2*a*	1.1103(2)	0.7883(2)	0.4247(2)	0.050(1)	0.066(1)	0.036(1)	–0.014(1)	0.0076(8)	–0.010(1)
C(26)	2*a*	1.2672(2)	0.7991(2)	0.4177(2)	0.054(1)	0.094(2)	0.067(1)	–0.026(1)	0.019(1)	–0.018(1)
C(27)	2*a*	1.2368(2)	0.7919(2)	0.2676(2)	0.066(1)	0.069(2)	0.068(1)	–0.017(1)	0.035(1)	0.000(1)
C(28)	2*a*	1.1188(2)	0.7142(1)	0.2238(2)	0.048(1)	0.048(1)	0.0337(9)	0.001(1)	0.0132(8)	0.0041(9)
C(29)	2*a*	1.1909(2)	0.6210(2)	0.2272(2)	0.056(1)	0.059(1)	0.052(1)	0.009(1)	0.0251(8)	0.008(1)
C(30)	2*a*	1.3247(3)	0.5336(2)	0.1214(3)	0.132(2)	0.081(2)	0.107(2)	0.023(2)	0.072(1)	–0.002(2)

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