Z. Kristallogr. NCS 221 (2006) 171-172 / DOI 10.1524/ncrs.2006.0026

Crystal structure of (diphenylphosphinoyl)phenylmethanol, $C_{19}H_{17}O_2P$

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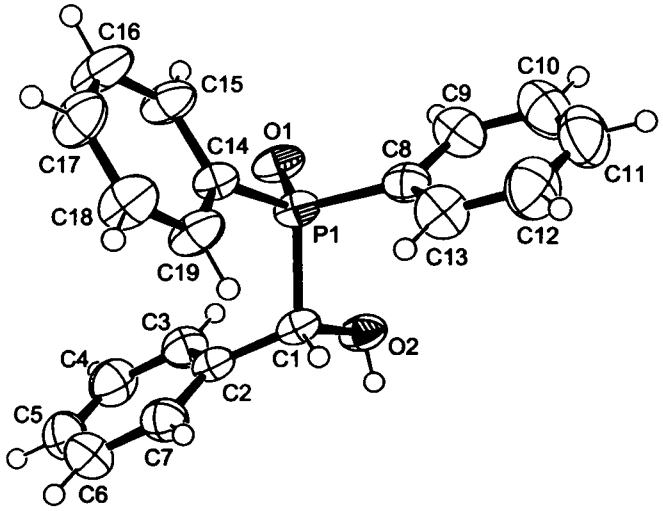
Received March 17, 2006, accepted and available on-line April 23, 2006; CCDC no. 1267/1752



Abstract

$C_{19}H_{17}O_2P$, monoclinic, $P12_1/n1$ (no. 14), a = 11.239(4) Å, b = 6.310(2) Å, c = 22.157(8) Å, β = 92.091(7)°, V = 1570.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.041, $wR_{ref}(F^2)$ = 0.114, T = 293 K.

Source of material

Using PPh_3 (triphenylphosphine) as original material, the title compound was synthesized via one-pot-method instead of the traditional route [1]. Lithium wire (0.3 g, 40 mmol) was added in a solution of PPh_3 (5.2 g, 20 mmol) in THF (60 ml) in argon atmosphere. The mixture was stirred for 8 h, then *t*-butyl chloride (1.9 g, 20 mmol) and benzaldehyde (2 ml, 20 mmol) were added. After stirred for 1 h, saturated NH_4Cl solution (30 ml) was added, then the mixture was extracted with CH_2Cl_2 (3 × 50 ml). The product was purified through column chromatography on silica gel using petroleum ether/ethyl acetate (9:1, *v*/*v*) as eluent. The solution of the target compound in THF was kept in the air for several days, then colorless prismatic crystals were obtained (m.p. 450-452 K).

Discussion

The compound with diphenylphosphinoyl skeleton was known as a kind of good character ligand in many catalytic reactions. The title compound as a ligand was used in hydroformylation reaction [2]. The C—C bond lengths in the three phenyl rings are between 1.366(4) Å to 1.393(3) Å) and the bond angles lie between 118.4(2)° to 120.8(2)°. These show typical aromatic character. The distance of P1—O1 (1.494 Å) is very close to P=O bond length (1.493 Å) reported in the literature [3]. There are no intermolecular π-π stacking interactions in the crystal structure. The molecules are held together as chain by a typical intermolecular O–H···O hydrogen bond (O2–H···O1′=P1′, where d(O2—H) = 0.82 Å, d(H···O1′) = 1.974 Å, d(O2···O1′) = 2.739 Å, ∠O2–H···O1′ = 155.0°. This makes the crystal structure stable and explains the high melting point.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.10 × 0.15 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.79 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6203, 2779
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1840
$N(param)_{refined}$:	199
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	4*e*	0.2848	0.2998	0.2623	0.069
H(1B)	4*e*	0.1541	0.1543	0.2004	0.044
H(3A)	4*e*	0.3352	0.5942	0.1593	0.060
H(4A)	4*e*	0.4729	0.5888	0.0849	0.072
H(5A)	4*e*	0.4901	0.2963	0.0238	0.080
H(6A)	4*e*	0.3706	0.0055	0.0379	0.075
H(7A)	4*e*	0.2335	0.0058	0.1127	0.059
H(9A)	4*e*	−0.0240	0.7121	0.2814	0.070
H(10A)	4*e*	−0.1426	0.6527	0.3621	0.092
H(11A)	4*e*	−0.2273	0.3266	0.3758	0.097
H(12A)	4*e*	−0.1991	0.0585	0.3071	0.088
H(13A)	4*e*	−0.0786	0.1155	0.2261	0.071
H(15A)	4*e*	−0.0697	0.6945	0.0918	0.064
H(16A)	4*e*	−0.1834	0.6159	0.0059	0.081
H(17A)	4*e*	−0.2069	0.2697	−0.0244	0.071
H(18A)	4*e*	−0.1193	0.0024	0.0315	0.069
H(19A)	4*e*	−0.0066	0.0769	0.1177	0.061

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)	4*e*	0.05381(5)	0.47606(9)	0.18238(3)	0.0360(3)	0.0298(4)	0.0458(4)	−0.0005(3)	−0.0093(3)	−0.0008(3)
O(1)	4*e*	0.0881(1)	0.7046(2)	0.18009(7)	0.0446(9)	0.0289(9)	0.063(1)	−0.0013(7)	−0.0172(8)	0.0008(7)
O(2)	4*e*	0.2317(1)	0.3791(2)	0.24971(7)	0.047(1)	0.0437(9)	0.047(1)	0.0091(8)	−0.0165(8)	−0.0039(8)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4e	0.1824(2)	0.2993(3)	0.1942(1)	0.039(1)	0.029(1)	0.041(1)	0.001(1)	–0.011(1)	0.001(1)
C(2)	4e	0.2688(2)	0.3003(3)	0.1438(1)	0.038(1)	0.035(1)	0.041(1)	0.005(1)	–0.007(1)	0.004(1)
C(3)	4e	0.3419(2)	0.4750(4)	0.1350(1)	0.049(2)	0.044(2)	0.055(2)	–0.000(1)	–0.001(1)	0.003(1)
C(4)	4e	0.4242(2)	0.4718(5)	0.0904(1)	0.054(2)	0.062(2)	0.066(2)	–0.007(1)	0.006(2)	0.013(2)
C(5)	4e	0.4348(3)	0.2972(5)	0.0540(1)	0.062(2)	0.085(2)	0.055(2)	0.013(2)	0.016(2)	0.012(2)
C(6)	4e	0.3634(3)	0.1240(5)	0.0625(1)	0.069(2)	0.061(2)	0.058(2)	0.012(2)	0.007(2)	–0.008(2)
C(7)	4e	0.2810(2)	0.1244(4)	0.1072(1)	0.050(2)	0.044(2)	0.052(2)	0.002(1)	–0.003(1)	–0.004(1)
C(8)	4e	–0.0391(2)	0.4210(4)	0.2451(1)	0.033(1)	0.045(2)	0.048(2)	0.002(1)	–0.009(1)	–0.003(1)
C(9)	4e	–0.0588(2)	0.5798(5)	0.2864(1)	0.051(2)	0.063(2)	0.061(2)	–0.004(1)	0.000(1)	–0.015(2)
C(10)	4e	–0.1295(3)	0.5443(6)	0.3347(1)	0.064(2)	0.103(3)	0.065(2)	0.002(2)	0.007(2)	–0.025(2)
C(11)	4e	–0.1805(3)	0.3502(7)	0.3426(1)	0.056(2)	0.122(3)	0.065(2)	–0.002(2)	0.014(2)	0.005(2)
C(12)	4e	–0.1631(3)	0.1897(5)	0.3020(1)	0.058(2)	0.086(2)	0.078(2)	–0.016(2)	0.010(2)	0.015(2)
C(13)	4e	–0.0915(2)	0.2244(4)	0.2533(1)	0.058(2)	0.058(2)	0.063(2)	–0.006(1)	0.003(2)	–0.004(1)
C(14)	4e	–0.0274(2)	0.3947(3)	0.1146(1)	0.034(1)	0.032(1)	0.044(1)	–0.001(1)	–0.006(1)	0.003(1)
C(15)	4e	–0.0800(2)	0.5539(4)	0.0801(1)	0.065(2)	0.033(1)	0.060(2)	0.005(1)	–0.023(1)	–0.003(1)
C(16)	4e	–0.1476(2)	0.5073(4)	0.0284(1)	0.086(2)	0.046(2)	0.067(2)	0.011(2)	–0.036(2)	0.006(1)
C(17)	4e	–0.1618(2)	0.3015(4)	0.0104(1)	0.067(2)	0.059(2)	0.049(2)	–0.006(1)	–0.023(1)	–0.003(1)
C(18)	4e	–0.1094(2)	0.1423(4)	0.0438(1)	0.073(2)	0.038(2)	0.062(2)	–0.007(1)	–0.020(2)	–0.004(1)
C(19)	4e	–0.0421(2)	0.1866(4)	0.0955(1)	0.062(2)	0.035(1)	0.056(2)	–0.002(1)	–0.019(1)	0.004(1)

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