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X-ray diffraction analysis of $(Ph_2PO)_2$

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ABSTRACT

The title compound (Ph2PO)2 is analyzed by single crystal X-ray diffraction analysis. The crystals are monoclinic, space group P2(1)/n with a = 9.5112(19) Å, b = 11.161(2) Å, c = 9.5487(19) Å, $\alpha = 90°$, $\beta = 91.65(3)°$, $\gamma = 90°$, V = 1013.2(3) Å^3, $Z = 2$, $F(_{000}) = 420$, $Dc = 1.319$ g cm^{-3}, $\mu = 0.232$ mm^{-1}, the final $R = 0.0818$, and $wR = 0.2259$. A total of 7954 reflections were collected, of which 1758 were independent ($R_{int} = 0.0542$). In the crystal packing diagram, intermolecular C—H···O hydrogen bonds stabilize the solid state of the title compound.

KEYWORDS

Crystal structure; diphenyl; phosphine

Introduction

Recently, phosphines have received more and more attention by synthetic chemists possibly because of their wide applications in the research fields of coordination chemistry, inorganic chemistry, and organometallic chemistry as the complexes containing phosphines always have special properties and unique structures [1–10]. Our recent studies have shown that a series of transition-metal complexes with monophosphine or diphosphine ligands were prepared and structurally characterized by spectroscopy and X-ray diffraction analysis [11–14]. In this paper, we present the X-ray crystal structure of the title compound $(Ph_2PO)_2$.

Experimental

Crystal structure determination

The crystal of the title compound with dimensions of 0.20 × 0.18 × 0.12 mm was mounted on a Rigaku Saturn CCD area detector diffractometer with a graphite–monochromated MoKα radiation ($\lambda = 0.71073$ Å) by using phi (ϕ) and scan modes at 293(2) K in the range of 2.81° ≤ θ ≤ 25.01°. The crystal belongs to monoclinic system with space group P2(1)/n and crystal parameters of a = 9.5112(19) Å, b = 11.161(2) Å, c = 9.5487(19) Å, $\alpha = 90°$, $\beta = 91.65(3)°$, $\gamma = 90°$, V = 1013.2(3) A^3, and $D_c = 1.319$ g cm^{-3}. The absorption coefficient, $\mu = 0.232$ mm^{-1}, and Z = 2. A summary of crystal data is presented in Table 1.

The structure was solved by direct methods with SHELXS-97 [15] and refined by the full-matrix least-squares method on F^2 data using SHELXL-97 [16]. The empirical absorption corrections were applied to all intensity data. H atom of N-H was initially located in a difference Fourier map and was refined with the restraint Uiso(H) = 1.2 Ueq(N). Other H atoms were

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Table 1. Crystal data and structure refinement.

Empirical formula	$C_{24}H_{20}O_2P_2$
Formula weight (g/mol)	402.34
Crystal system	Monoclinic
Unit cell dimensions	
a	9.5112(19) Å
b	11.161(2) Å
c	9.5487(19) Å
Unit cell angles	
α	90°
β	91.65(3)°
Γ	90°
Volume ($Å^3$)	1013.2(3)
Z	2
Temperature (K)	293(2)
Space group	P2(1)/n
Wavelength	0.71073 Å
Calculated density	1.319 g cm^{-3}
Absorption coefficient	0.232 mm^{-1}
$F_{(000)}$	420
Crystal size	0.20 × 0.18 × 0.12 mm
Theta (θ) range for data collection ()	2.81°–25.01°
Reflections collected	7954
Independent reflections	1758 [$R_{(int)} = 0.0542$]
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0818$, $wR_2 = 0.2259$

positioned geometrically and refined using a riding model, with d(C—H) = 0.93–0.97 Å and Uiso(H) = 1.2 Ueq(C) or 1.5 Ueq(C methyl). The final full-matrix least-squares refinement gave R = 0.0818 and wR = 0.2259.

Results and discussion

The title compound $(Ph_2PO)_2$ was characterized by single crystal X-ray diffraction analysis. The selected bond lengths and bond angles are listed in Table 2. The structure was solved by direct methods. Anisotropic displacement parameters were applied to all non-hydrogen atoms in full-matrix least-squares refinement based on F^2. The hydrogen atoms were set in calculated positions with a common fixed isotropic thermal parameter.

Table 2. Selected bond lengths and bond angles.

Bond lengths (Å)			
P1-O1	1.486(4)	P1-O1A	1.615(4)
P1-C7	1.806(7)	P1-C1	1.835(3)
P1-P1A	2.2019(15)	C1-C6	1.380(4)
C1-C2	1.395(4)	C2-C3	1.397(4)
C3-C4	1.342(5)	C4-C5	1.346(5)
C5-C6	1.374(4)	O1A-P1	1.615(4)
C7-C12	1.380(10)	C7-C8	1.416(11)
C8-C9	1.382(10)	C9-C10	1.386(10)
Bond angles (°)			
O1-P1-O1A	89.6(2)	O1-P1-C7	73.6(3)
O1A-P1-C7	123.6(3)	O1-P1-C1	75.03(19)
O1A-P1-C1	121.42(17)	C7-P1-C1	105.8(2)
O1-P1A-P1	47.17(16)	O1A-P1A-P1	42.44(15)
C7-P1A-P1	102.4(2)	C1-P1A-P1	102.01(10)
C6-C1-C2	117.9(3)	C6-C1-P1	127.8(2)
C2-C1-P1	114.3(2)	C1-C2-C3	119.6(3)
C4-C3-C2	120.5(3)	C3-C4-C5	120.4(3)
C4-C5-C6	121.0(3)	C5-C6-C1	120.6(3)

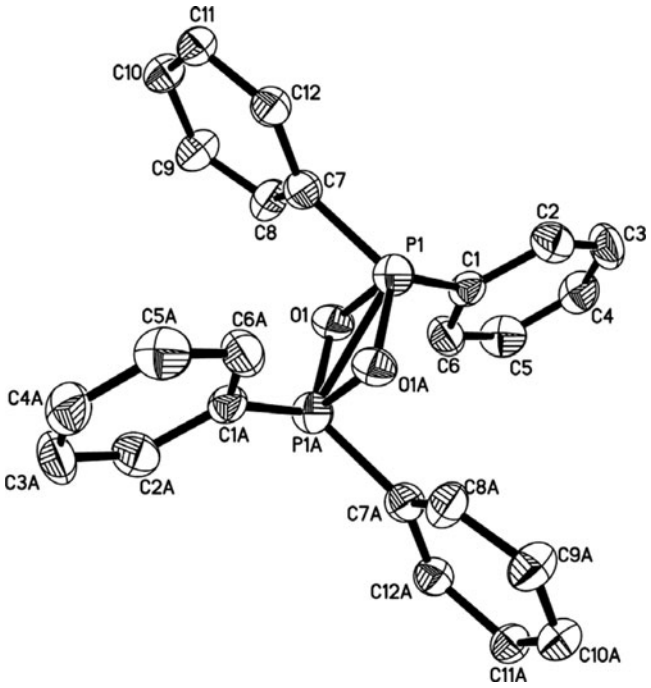

Figure 1. Molecular structure of the title compound.

The molecular structure and the packing view of the title compound are depicted in Figs. 1 and 2, respectively. The title compound crystallizes in monoclinic space group P2(1)/n with two molecules in the unit cell and one molecule in the asymmetric unit. As shown in Fig. 1, the molecular structure comprises two diphenylphosphanyl groups linking together by two oxygen atoms. The molecule is centrosymmetric with the midpoint of P1–P1A as the inversion center. The six-membered rings C1C2C3C4C5C6 and C7C8C9C10C11C12 are almost coplanar with the mean deviations of 0.0057 Å and 0.0341 Å, respectively. The dihedral angle between the two phenyl planes is 111.0°. The bond distances (P1-O1 = 1.486(4) Å, P1-C7 = 1.806(7) Å, P1-C1 = 1.835(3) Å, P1-P1A = 2.2019(15) Å, C1-C6 = 1.380(4) Å, C1-C2 = 1.395(4) Å, and C2-C3 = 1.397(4) Å), and bond angles [O1-P1-O1A = 89.6(2)°, O1-P1-C7 = 73.6(3)°, O1A-P1-C7 = 123.6(3)°, O1-P1-C1 = 75.03(19)°, O1A-P1-C1 = 121.42(17)°, C7-P1-C1 = 105.8(2)°, O1-P1-P1A = 47.17(16)°, and O1A-P1-P1A = 42.44(15)°] are close to other compounds [17–25].

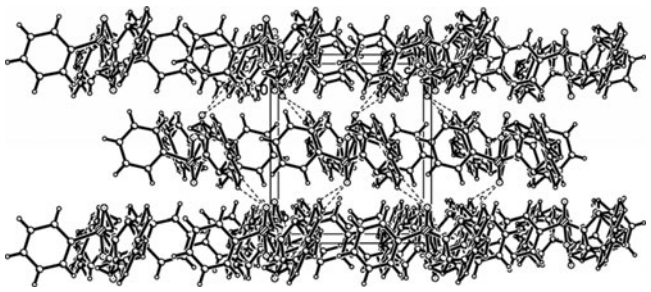

Figure 2. The crystal packing view of the title compound.

As shown in Fig. 2, the crystal packing diagram of the title compound shows that intermolecular C–H···O hydrogen bonds exist to stabilize the solid state.

Conclusions

In summary, the title compound $(Ph_2PO)_2$ has been characterized by X-ray diffraction analysis.

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