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Structural analysis of 1,1′-bis(diphenylphosphino)ferrocene dioxide

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ABSTRACT

The title compound 1,1′-bis(diphenylphosphino)ferrocene dioxide has been analyzed by X-ray crystal diffraction analysis. The crystals are monoclinic, space group P2(1)/c with a = *22.461(5)*, b = *10.515(2)*, c = *12.024(2)* Å, $\alpha = 90$, $\beta = 96.28(3)$, $\gamma = 90°$, $V = 2822.7(10)$ *Å*^{3}, $Z = 4$, $F(000) = 1256$, $D_c = 1.422$ *g/cm*^{3}, $\mu = 0.683$ *mm*$^{-1}$, *the final* $R = 0.0514$ *and* $wR = 0.1369$. A total of 22218 reflections were collected, of which 4957 were independent ($R_{int} = 0.0422$). In the crystal packing diagram, intermolecular O–H···O hydrogen bonds between the P=O and H_2O stabilize the solid state of the title compound.

KEYWORDS

Crystal structure; diphosphine; Dppf

Introduction

In recent years, diphosphine ligands such as 1,1-bis(diphenylphosphino)methane, 1,2-bis(diphenylphosphino)ethane, 1,1′-bis(diphenylphosphino)ferrocene and *N*-substituted bis(diphenylphosphanyl)amine, have been attracted much interest due to their widely application in coordination chemistry.[1–15] In this contribution, we unexpectedly obtain the oxidation state of dppf and its structure was characterized by X-ray diffraction analysis.

Experimental

Crystal structure determination

The crystal of the title compound with dimensions of 0.20 mm × 0.18 mm × 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 a phi and scan modes at 113(2) K in the range of $2.58° \leq \theta \leq 25.02°$. The crystal belongs to monoclinic system with space group P2(1)/c and crystal parameters of a = 22.461(5) Å, b = 10.515(2) Å, c = 12.024(2) Å, $\alpha = 90°$, $\beta = 96.28(3)°$, $\gamma = 90°$, $V = 2822.7(10)$ A^3, $D_c = 1.422$ g cm^{-3}, The absorption coefficient $\mu = 0.683$ mm^{-1} and $Z = 4$. A summary of crystal data is presented in Table 1.

The structure was solved by direct methods with SHELXS-97 [16] and refined by the full-matrix least squares method on F^2 data using SHELXL-97 [17]. The empirical absorption corrections were applied to all intensity data. H atom of N-H was initially located in a difference Fourier map and were refined with the restraint Uiso(H) = 1.2Ueq(N). Other H atoms

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Table 1. Crystal data and structure refinement.

Empirical formula	$C_{34}H_{30}FeO_3P_2$
Formula weight	604.37
Crystal system	Monoclinic
Unit cell dimensions	
a (Å)	22.461(5)
b (Å)	10.515(2)
c (Å)	12.024(2)
Unit cell angles (°)	
A	90
β	96.28(3)
Γ	90
Volume ($Å^3$)	2822.7(10)
Z	4
Temperature (K)	113(2)
space group	P2(1)/c
Wavelength (Å)	0.71073
Calculated density (g/cm^3)	1.422
Absorption coefficient (mm^{-1})	0.683
F(000)	1256
Crystal size (mm)	0.20 × 0.18 × 0.12
Theta range for data collection (°)	2.58 –25.02
Reflections collected	22218
Independent reflections	4957 [$R_{(int)} = 0.0422$]
Final R indices [I>2σ(I)]	$R_1 = 0.0514$, w$R_2 = 0.1369$

Table 2. Selected bond lengths (Å) and bond angles (°).

Bond lengths			
Fe(1)-C(13)	2.029(3)	Fe(1)-C(14)	2.034(3)
Fe(1)-C(22)	2.034(3)	Fe(1)-C(18)	2.037(3)
Fe(1)-C(17)	2.042(3)	Fe(1)-C(21)	2.048(3)
Fe(1)-C(19)	2.051(3)	Fe(1)-C(20)	2.055(3)
Fe(1)-C(15)	2.063(3)	Fe(1)-C(16)	2.066(3)
P(1)-O(1)	1.519(2)	P(2)-O(2)	1.526(3)
Bond angles			
C(13)-Fe(1)-C(22)	168.75(12)	C(13)-Fe(1)-C(14)	41.30(12)
C(22)-Fe(1)-C(14)	149.30(12)	C(13)-Fe(1)-C(18)	148.40(12)
C(22)-Fe(1)-C(18)	41.30(13)	C(14)-Fe(1)-C(18)	115.25(13)
C(13)-Fe(1)-C(17)	41.32(11)	C(22)-Fe(1)-C(17)	130.16(12)
C(14)-Fe(1)-C(17)	68.99(12)	C(18)-Fe(1)-C(17)	168.66(12)
O(1)-P(1)-C(13)	113.50(13)	O(1)-P(1)-C(1)	111.94(14)
C(13)-P(1)-C(1)	108.12(13)	C(1)-P(1)-C(7)	104.44(13)
O(2)-P(2)-C(22)	114.21(15)	O(2)-P(2)-C(29)	110.89(16)
C(22)-P(2)-C(29)	105.92(15)	O(2)-P(2)-C(23)	117.77(19)

were positioned geometrically and refined using a riding model, with d(C—H) = 0.93-0.97 Å and Uiso(H) = 1.2Ueq(C) or 1.5Ueq(Cmethyl). The final full-matrix least squares refinement gave $R = 0.0514$ and $wR = 0.1369$.

Results and discussion

The title compound 1,1′-bis(diphenylphosphino)ferrocene dioxide was confirmed 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 nonhydrogen atoms in full-matrix least-square refinements based on F^2. The hydrogen atoms were set in calculated positions with a common fixed isotropic thermal parameter.

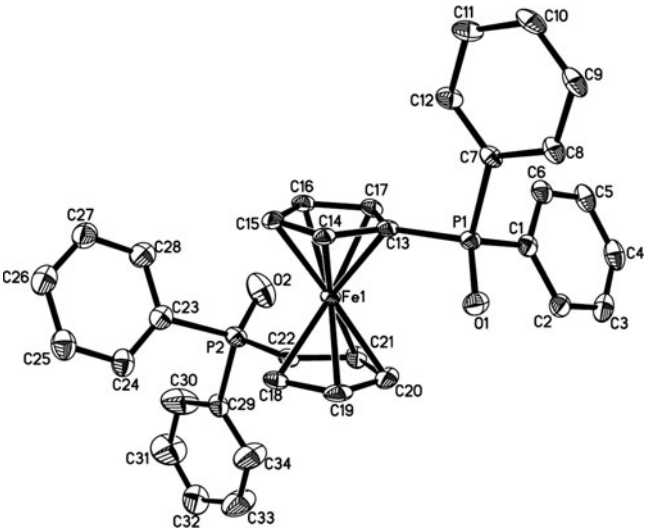

Figure 1. Molecular structure of the title compound.

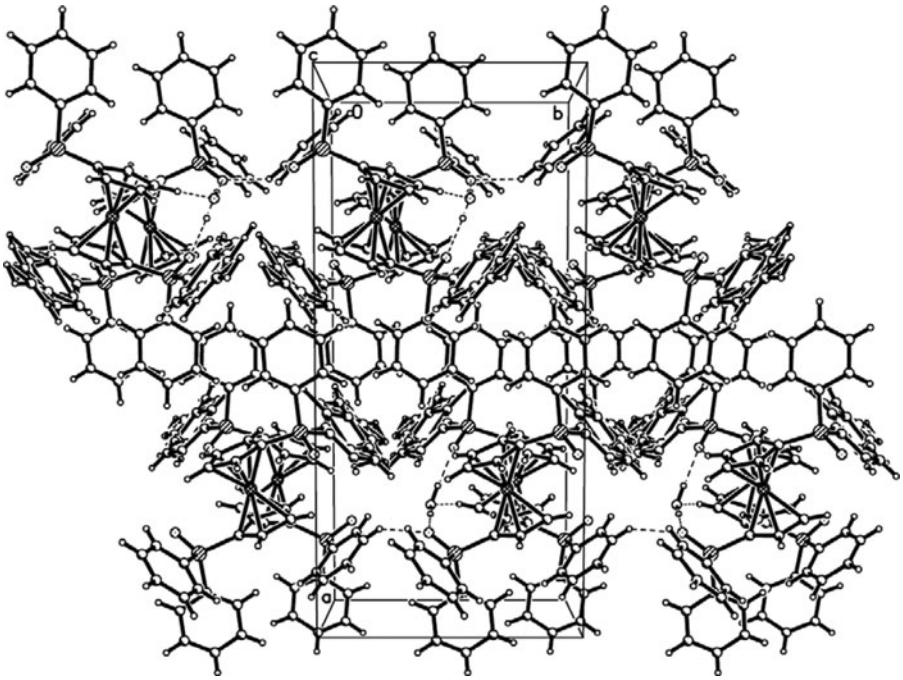

Figure 2. The crystal packing view of the title compound.

The molecular structure and the packing view of the title compound are displayed in Figs. 1 and 2, respectively. The title compound crystallizes in monoclinic space group P2(1)/c with four molecules in the unit cell and one molecule in the asymmetric unit. As shown in Fig. 1, the molecular structure consists of a ferrocene unit with two diphenylphosphino groups. The orientation of the two diphenylphosphine resides in an opposite position in order to reduce the steric repulsion. The five-membered rings C13C14C15C16C17 and C18C19C20C21C22 are almost parallel to each other with the dihedral angle of 2.3°. The bond distances [Fe(1)-C(13) = 2.029(3) Å, Fe(1)-C(22) = 2.034(3) Å, Fe(1)-C(14) = 2.034(3) Å, Fe(1)-C(18) = 2.037(3)

Å, P(1)-O(1) = 1.519(2) Å, P(1)-C(13) = 1.785(3) Å, P(1)-C(1) = 1.798(3) Å, and P(2)-O(2) = 1.526(3) Å] and bond angles [C(13)-Fe(1)-C(22) = 168.75(12)°, C(13)-Fe(1)-C(14) = 41.30(12)°, C(22)-Fe(1)-C(14) = 149.30(12)°, C(13)-Fe(1)-C(18) = 148.40(12)°, O(1)-P(1)-C(13) = 113.50(13)°, O(1)-P(1)-C(1) = 111.94(14)°, O(2)-P(2)-C(22) = 114.21(15)°, and O(2)-P(2)-C(23) = 117.77(19)°] are close to other similar compounds [18–28].

As shown in Fig. 2, the crystal packing diagram of the title compound reveals that intermolecular O–H···O hydrogen bonds between the P = O and H_2O existing to stabilize the solid state.

Conclusions

In summary, the title compound 1,1′-bis(diphenylphosphino)ferrocene dioxide has been characterized by X-ray diffraction analysis.

Acknowledgment

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Supplemental Data

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 1034404 for the title compound. Copies of the data can be obtained free of charge at http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk.

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