Crystal structure of (2-hydroxy-5-methylphenyl)diphenylphosphineoxide, $(C_6H_5)_2P(O)(CH_3C_6H_3OH)$

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Dedicated to our friend and collegue, the late S. J. Rettig

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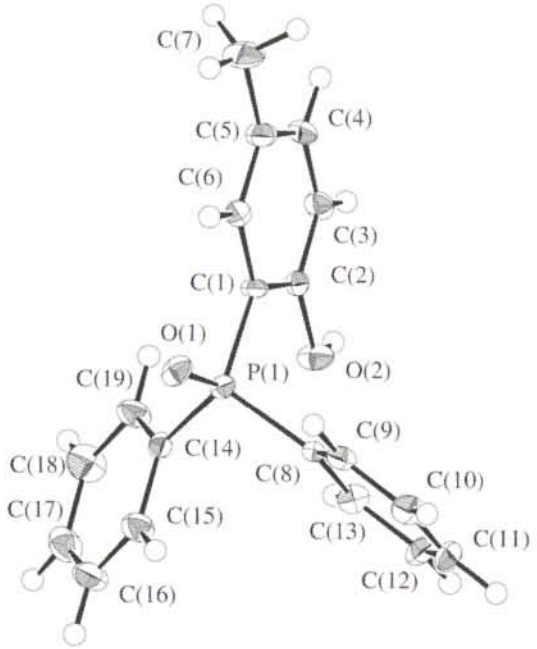

Abstract

$C_{19}H_{17}O_2P$, monoclinic, $P12_1/n1$ (No. 14), $a = 9.366(2)$ Å, $b = 11.693(1)$ Å, $c = 14.5539(6)$ Å, $\beta = 99.7770(9)°$, $V = 1570.8$ Å^3, $Z = 4$, $R_{all}(F) = 0.082$, $wR_{all}(F) = 0.074$, $T = 180$ K.

Source of material

The title compound was synthesized according to [1] starting from 4-methylphenol. The methoxymethylether (mom) protected derivative was ortho-lihiated and subsequently treated with chlorophenylphosphine. Cleavage of the mom-group with anhoydous HCl gave the expected functionalized phosphineoxide. Suitable crystals for the determination of the structure were obtained - as a by-product - through recystallization from methanol.

All non-hydrogen atoms were refined anisotropically. The OH hydrogen was refined isotropically with the O—H bond length restrained to 0.9 Å, other were fixed in calculated positions with d(C—H) = 0.98 Å.

Discussion

As part of a research program to investigate hybrid hard/soft ligands which are phenylphosphines functionalized with phenol groups, we have prepared a series of ligands and their complexes with Re [2, 3]. These ligands are derivatives of triphenylphosphine containing one (*o*-hydroxyphenyl)diphenylphosphine - HPO) and two (bis(*o*-hydroxyphenyl)phenylphosphine · H_2PO_2) phenols [1], as well as a perphenolated dppe analog (*P*,*P*,*P*′,*P*′-tetrakis(*o*-hydroxyphenyl)diphosphinoethane · $H_4P_2O_4$) [3].

In an effort to provide better NMR probes for the study of complexation in solution, we are now examining these same ligands with methyl groups on the rings [4]. As part of this latter effort, we have prepared the phosphineoxide analog of HPO (2-hydroxy-5-methylphenyl)diphenylphosphineoxide, whose crystal structure we report herein.

All determined bond lengths and angles fall in the typical range. The P-atom exhibits a slightly distorted tetrahedral geometry due to a hydrogen bond between O1 and the hydroxy function of a neighbouring molecule.

Table 1. Data collection and handling.

Crystal:	clear block, size 0.10 x 0.20 x 0.30 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	1.79 cm^{-1}
Diffractometer, scan mode:	Rigaku/ADSC CCD, $\chi = -90°$, $0° < \varphi < 190°$, $\Delta\varphi = 0.5°$, $\chi = -90°$, $-23° < \varphi < 18°$, $\Delta\omega = 0.5°$
$2\theta_{max}$:	61°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14463, 4011
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 0\ \sigma(I_{obs})$, 4011
$N(param)_{refined}$:	203
Programs:	teXsan [5], SIR97 [6]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.167(2)	0.144(2)	0.291(2)	0.068(8)
H(2)	4*e*	–0.0647	0.1937	0.2451	0.027
H(3)	4*e*	–0.2225	0.3344	0.1697	0.032
H(4)	4*e*	0.1228	0.5301	0.1302	0.024
H(5)	4*e*	–0.1129	0.6043	0.0836	0.046
H(6)	4*e*	–0.2358	0.5576	0.1381	0.046
H(7)	4*e*	–0.2217	0.5063	0.0381	0.046
H(8)	4*e*	0.4631	0.4058	0.0289	0.025
H(9)	4*e*	0.5787	0.2764	–0.0590	0.030
H(10)	4*e*	0.6360	0.0879	0.0000	0.033
H(11)	4*e*	0.5774	0.0325	0.1446	0.032
H(12)	4*e*	0.4642	0.1644	0.2339	0.028
H(13)	4*e*	0.6640	0.4030	0.2900	0.032
H(14)	4*e*	0.7902	0.3771	0.4439	0.039
H(15)	4*e*	0.6629	0.3447	0.5669	0.041
H(16)	4*e*	0.4077	0.3389	0.5391	0.042
H(17)	4*e*	0.2791	0.3648	0.3852	0.033

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*	0.37113(6)	0.40264(4)	0.20447(4)	0.0152(3)	0.0149(2)	0.0207(3)	0.0008(2)	0.0049(2)	0.0007(2)
O(1)	4*e*	0.3970(2)	0.51938(9)	0.16680(9)	0.0253(9)	0.0134(6)	0.0331(9)	0.0003(6)	0.0102(8)	0.0026(6)
O(2)	4*e*	0.2187(2)	0.1938(1)	0.2661(1)	0.0182(9)	0.0161(7)	0.045(1)	0.0006(6)	0.0047(8)	0.0097(7)
C(1)	4*e*	0.1800(2)	0.3763(1)	0.1962(1)	0.010(1)	0.0174(9)	0.021(1)	0.0022(7)	0.0009(9)	0.0019(8)
C(2)	4*e*	0.1232(2)	0.2755(1)	0.2281(1)	0.017(1)	0.0182(9)	0.015(1)	0.0016(8)	0.0017(9)	–0.0022(8)
C(3)	4*e*	–0.0249(2)	0.2623(2)	0.2201(1)	0.016(1)	0.028(1)	0.024(1)	–0.0050(8)	0.006(1)	–0.0030(9)
C(4)	4*e*	–0.1176(2)	0.3462(2)	0.1767(2)	0.012(1)	0.034(1)	0.033(1)	–0.0008(9)	0.001(1)	–0.0098(9)
C(5)	4*e*	–0.0652(2)	0.4469(2)	0.1428(1)	0.018(1)	0.031(1)	0.023(1)	0.0062(9)	0.000(1)	–0.0020(9)
C(6)	4*e*	0.0835(2)	0.4601(1)	0.1534(1)	0.019(1)	0.0181(9)	0.021(1)	0.0008(8)	0.001(1)	–0.0008(8)
C(7)	4*e*	–0.1678(3)	0.5366(2)	0.0966(2)	0.029(2)	0.042(1)	0.040(2)	0.016(1)	–0.008(1)	0.007(1)
C(8)	4*e*	0.4524(2)	0.2973(1)	0.1382(1)	0.011(1)	0.0168(9)	0.019(1)	0.0008(8)	0.0010(9)	–0.0036(7)
C(9)	4*e*	0.4870(2)	0.3286(2)	0.0530(1)	0.015(1)	0.027(1)	0.019(1)	0.0013(9)	0.0000(9)	0.0018(8)
C(10)	4*e*	0.5545(2)	0.2531(2)	0.0012(1)	0.021(1)	0.038(1)	0.018(1)	0.001(1)	0.008(1)	–0.0006(9)
C(11)	4*e*	0.5882(2)	0.1423(2)	0.0361(2)	0.024(1)	0.031(1)	0.030(1)	0.002(1)	0.009(1)	–0.0145(9)
C(12)	4*e*	0.5543(2)	0.1100(2)	0.1209(1)	0.026(1)	0.020(1)	0.036(1)	0.0061(9)	0.009(1)	–0.0015(9)
C(13)	4*e*	0.4870(2)	0.1871(2)	0.1732(1)	0.023(1)	0.0241(9)	0.023(1)	0.0014(9)	0.006(1)	0.0033(8)
C(14)	4*e*	0.4595(2)	0.3845(1)	0.3235(1)	0.019(1)	0.0147(9)	0.020(1)	–0.0001(8)	0.0040(8)	–0.0021(8)
C(15)	4*e*	0.6102(2)	0.3892(2)	0.3412(1)	0.019(1)	0.033(1)	0.027(1)	–0.003(1)	0.005(1)	–0.002(1)
C(16)	4*e*	0.6841(2)	0.3743(2)	0.4315(2)	0.020(1)	0.032(1)	0.042(1)	–0.001(1)	–0.005(1)	–0.002(1)
C(17)	4*e*	0.6096(3)	0.3555(2)	0.5035(2)	0.037(1)	0.032(1)	0.028(1)	–0.000(1)	–0.008(1)	0.002(1)
C(18)	4*e*	0.4605(3)	0.3519(2)	0.4874(2)	0.035(1)	0.047(1)	0.023(1)	–0.003(1)	0.007(1)	0.003(1)
C(19)	4*e*	0.3852(2)	0.3668(2)	0.3970(2)	0.017(1)	0.040(1)	0.026(1)	–0.003(1)	0.005(1)	–0.003(1)

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