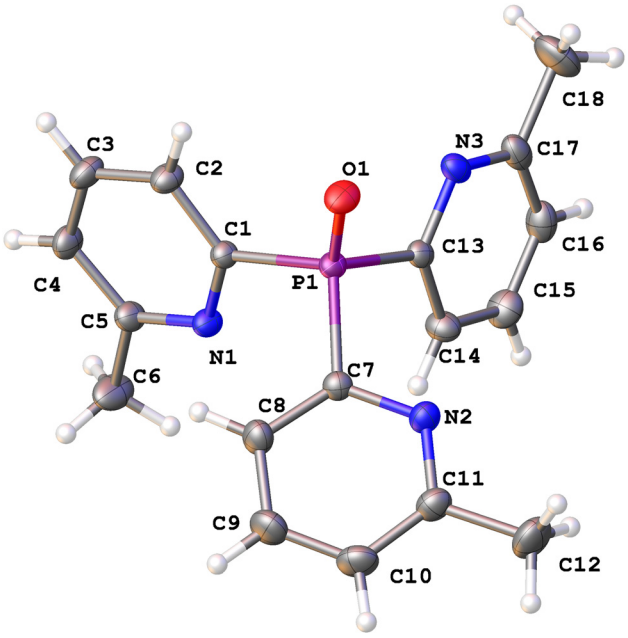
Gui-Ling Wu*

The crystal structure of tris(6-methylpyridin-2-yl) phosphine oxide, $C_{18}H_{18}N_3OP$



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Abstract

$C_{18}H_{18}N_3OP$, monoclinic, $P2_1/n$ (no. 14), a = 13.3987(17) Å, b = 8.7084(11) Å, c = 14.5063(18) Å, β = 95.849(2)° V = 1683.8(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0423, w$R_{ref}(F^2)$ = 0.1311, T = 296(2) K.

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The crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Block, clear light colourless
Size:	0.20 × 0.20 × 0.15 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ:	0.17 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω-scans
θ_{max}, completeness:	30°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17391, 4874, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4110
$N(param)_{refined}$:	211
Programs:	Bruker programs [1], OLEX [2], SHELX [3, 4]

Source of materials

A mixture of 2-bromo-6-methylpyridine (30 mmol), red phosphorus (30 mmol) and powdered KOH (30 mmol), DMSO (20 mL), and H_2O (1 mL) was stirred for 1 h at 120 °C under argon. The mixture was cooled to room temperature, diluted with H_2O (40 mL) and extracted with $CHCl_3$ (3 × 20 mL). The combined extract was washed with H_2O (3 × 20 mL) and dried over Mg_2SO_4. The solvent was removed under reduced pressure and a microcrystalline powder was obtained. The aforementioned solid (4 mmol) was dissolved in toluene (30 mL) and H_2O_2 solution (2.0 mL) was added. The reaction solution was stirred at room temperature for 2 h. After completion of the reaction, the solvent was removed *in vacuo* and the crude product was triturated with isopropanol (10 mL). After drying *in vacuo*, a colorless powder was obtained in 86% yield.

Experimental details

Coordinates of hydrogen atoms were added using standard options of the SHELX system [3, 4]. Their U_{iso} values were set to 1.2U_{eq} of the parent atoms.

Comment

Over the last decade, pyridylphosphine ligands have attracted attention for the design of transition metal complexes which demonstrate extraordinary coordination behavior and catalytic activity. The ability of such ligands to coordinate to a catalytically active transition metal via the phosphorus atom makes them ideal for constructing metallic and heterometallic architectures [5–7]. Tris(2-pyridyl)phosphine exhibits rich

***Corresponding author: Gui-Ling Wu**, Department of Chemistry and Chemical Engineering, Qiannan Normal University for Nationalities, Duyun 558000, P. R. China, E-mail: wuguiling1985@163.com.
https://orcid.org/0000-0002-7584-8829

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.28517 (8)	0.30445 (13)	0.01985 (8)	0.0338 (2)
N2	0.36249 (9)	0.72363 (14)	0.16337 (8)	0.0383 (3)
N3	0.24404 (9)	0.72964 (12)	−0.11779 (8)	0.0357 (2)
O1	0.12973 (7)	0.68199 (12)	0.05029 (8)	0.0422 (2)
P1	0.22329 (2)	0.59414 (3)	0.04377 (2)	0.02815 (11)
C1	0.20237 (9)	0.39276 (13)	0.01454 (9)	0.0295 (2)
C2	0.10769 (10)	0.33947 (16)	−0.01556 (10)	0.0379 (3)
H2	0.052390	0.404546	−0.019349	0.045*
C3	0.09743 (11)	0.18528 (18)	−0.04007 (11)	0.0435 (3)
H3	0.034669	0.145247	−0.060366	0.052*
C4	0.18077 (11)	0.09306 (16)	−0.03401 (11)	0.0414 (3)
H4	0.175008	−0.010401	−0.049524	0.050*
C5	0.27455 (10)	0.15616 (15)	−0.00425 (10)	0.0367 (3)
C6	0.36779 (14)	0.0606 (2)	0.00085 (17)	0.0621 (5)
H6A	0.425473	0.126482	0.004285	0.093*
H6B	0.366476	−0.002734	−0.053450	0.093*
H6C	0.371224	−0.003415	0.054976	0.093*
C7	0.30736 (10)	0.59480 (14)	0.15027 (8)	0.0316 (3)
C8	0.30791 (12)	0.47758 (18)	0.21485 (10)	0.0428 (3)
H8	0.267230	0.391623	0.203918	0.051*
C9	0.37124 (14)	0.4924 (2)	0.29667 (11)	0.0539 (4)
H9	0.373590	0.416472	0.341936	0.065*
C10	0.43009 (14)	0.6211 (2)	0.30918 (11)	0.0526 (4)
H10	0.474625	0.631459	0.362336	0.063*
C11	0.42312 (12)	0.73576 (19)	0.24254 (10)	0.0446 (3)
C12	0.48210 (18)	0.8826 (2)	0.25702 (15)	0.0689 (6)
H12A	0.517013	0.902554	0.203683	0.103*
H12B	0.529713	0.872717	0.310812	0.103*
H12C	0.437178	0.965946	0.265807	0.103*
C13	0.29749 (9)	0.65975 (13)	−0.04709 (8)	0.0293 (2)
C14	0.39914 (11)	0.62984 (18)	−0.04539 (10)	0.0406 (3)
H14	0.434084	0.582368	0.005509	0.049*
C15	0.44766 (12)	0.6725 (2)	−0.12168 (12)	0.0486 (4)
H15	0.515656	0.652643	−0.123318	0.058*
C16	0.39368 (14)	0.74414 (18)	−0.19411 (11)	0.0482 (4)
H16	0.424722	0.773580	−0.245820	0.058*
C17	0.29182 (13)	0.77306 (16)	−0.19036 (10)	0.0438 (3)
C18	0.2302 (2)	0.8554 (3)	−0.26712 (14)	0.0754 (6)
H18A	0.192526	0.782029	−0.305905	0.113*
H18B	0.273634	0.912121	−0.303350	0.113*
H18C	0.184853	0.924874	−0.241239	0.113*

coordination capabilities owing to its unique structure. On the other hand, there are many studies on the substitution of pyridine-4-yl substituents in the substituents of tris(2-pyridyl) phosphine [8, 9]. A typical side-product of many reactions are the corresponding Phosphine oxides like the title compound.

The title compound crystallizes in the monoclinic space group $P2_1/n$ (no. 14) with four molecules in the unit cell. The P–O bond distance, 1.4798(9) Å, is comparable with similar structures found in the literature showing typical double bond character [8–10]. The pyridine ring (N1/C1–C5) makes dihedral angles of 60.54(7)° and 80.96(7)° with the other two pyridine rings (N2/C7–C11 and N3/C13–C17), respectively. In addition, the crystal structure exhibits weak C–H$\cdots\pi$ interactions. [C(6)–H(6B)$\cdots$C*g*3′ = 2.75 (4) Å; C*g*3 is the centroid of pyridine ring (N3, C13, C14, C15, C16, C17); ′ = x, −1 + y, z].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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