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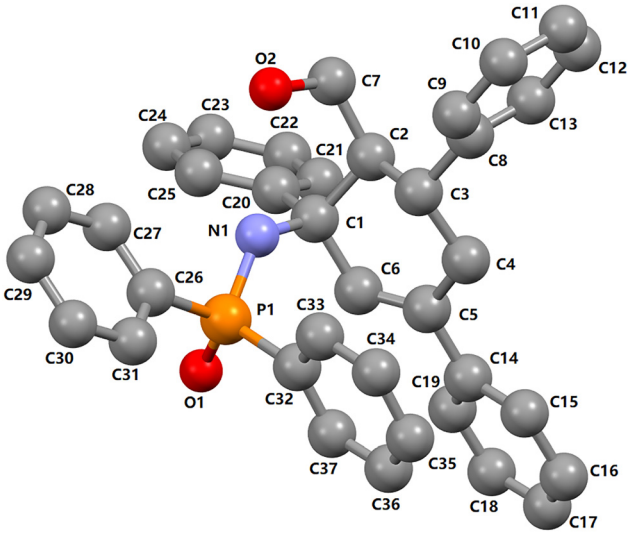
The crystal structure of *N*-(2′-hydroxymethyl-5′-phenyl-3′,4′-dihydro-[1,1′:3′,1″-terphenyl]-1′(2′*H*)-yl)-*P*,*P*-diphenylphosphinic amide, $C_{37}H_{34}NO_2P$



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Abstract

$C_{37}H_{34}NO_2P$, monoclinic, $P2_1/c$ (no. 14), a = 9.3479(3) Å, b = 23.0805(8) Å, c = 13.7819(5) Å, β = 102.462(10)°, V = 2903.44(17) Å^3, Z = 4, $R_{gt}(F)$ = 0.0458, $wR_{ref}(F^2)$ = 0.1309, T = 300(2) K.

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The molecular structure is shown in the figure (Hydrogen atoms are omitted for clarity). 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:	White needle
Size:	0.23 × 0.16 × 0.12 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.13 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	30.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	48573, 8868, 0.029
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 6976
$N(param)_{refined}$:	372
Programs:	Bruker [1], SHELX [2, 3]

Source of material

The title compound was obtained according to the following procedure: to a dry Schlenk tube was added *N*-((1*Z*,2*E*)-1,3-diphenylbut-2-en-1-ylidene)-*P*,*P*-diphenylphosphinic amide (0.2 mmol, 1 eq), cinnamic aldehyde (2 eq), (*R*)-diphenylprolinol trimethyl silyl ether (0.2 eq), K_2HPO_4 (1 eq), KH_2PO_4 (1 eq), and NaCl (1 eq). Under nitrogen atmosphere, the reaction was initiated at 60 °C with 2 mL of MeOH/H_2O (99:1) as solvent. After 36 h, $NaBH_4$ (1.5 eq) was added into the Schlenk tube, and the mixture was then stirred for 1 h under 0 °C. The solvent was removed under reduced pressure to give the crude residue, which was purified via silica gel column chromatography (Hexane/DCM/EtOAc 3:0.8:0.2) to afford the title compound in 34% yield. The white needles were obtained via recrystallization in DCM/MeOH.

Experimental details

A Bruker APEX–II CCD diffractometer was employed to perform the diffraction experiments of a single crystal selected carefully. The data was collected using APEX 2 [1] data collection software at 300 K. The structure was solved and refined using the Bruker SHELXTL Software Package [2, 3]. Coordinates of all hydrogen atoms were refined without any constraints or restraints.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
P1	0.56110 (3)	0.25128 (2)	0.59713 (2)	0.02851 (8)
O1	0.59335 (11)	0.23536 (4)	0.70377 (7)	0.0392 (2)
O2	0.79206 (14)	0.24872 (4)	0.37462 (8)	0.0464 (3)
H2	0.7372 (19)	0.2644 (6)	0.3229 (17)	0.070*
N1	0.70491 (11)	0.25796 (4)	0.54820 (8)	0.0303 (2)
H1	0.6749	0.2663	0.4863	0.036*
C1	0.83957 (12)	0.29181 (5)	0.58658 (8)	0.0278 (2)
C2	0.88036 (13)	0.32603 (5)	0.49880 (8)	0.0297 (2)
H2A	0.9723	0.3463	0.5261	0.036*
C3	0.76388 (14)	0.37312 (5)	0.46416 (9)	0.0325 (2)
H3	0.6684	0.3538	0.4474	0.039*
C4	0.75934 (19)	0.41587 (6)	0.54889 (10)	0.0448 (3)
H4A	0.8340	0.4451	0.5495	0.054*
H4B	0.6651	0.4353	0.5347	0.054*
C5	0.78232 (13)	0.38952 (5)	0.65068 (9)	0.0319 (2)
C6	0.81453 (13)	0.33367 (5)	0.66584 (9)	0.0302 (2)
H6	0.8222	0.3193	0.7298	0.036*
C7	0.90975 (15)	0.28699 (6)	0.41500 (10)	0.0379 (3)
H7A	0.9296	0.3113	0.3620	0.045*
H7B	0.9969	0.2642	0.4403	0.045*
C8	0.78408 (15)	0.40530 (5)	0.37238 (9)	0.0342 (2)
C9	0.90389 (19)	0.44098 (7)	0.37350 (13)	0.0516 (4)
H9	0.9740	0.4455	0.4321	0.062*
C10	0.9207 (3)	0.46992 (8)	0.28859 (16)	0.0683 (5)
H10	1.0018	0.4936	0.2907	0.082*
C11	0.8189 (3)	0.46387 (8)	0.20193 (14)	0.0699 (6)
H11	0.8305	0.4834	0.1451	0.084*
C12	0.7001 (3)	0.42913 (9)	0.19910 (13)	0.0704 (6)
H12	0.6304	0.4252	0.1401	0.084*
C13	0.68234 (19)	0.39958 (7)	0.28360 (11)	0.0505 (4)
H13	0.6013	0.3757	0.2805	0.061*
C14	0.77056 (14)	0.42820 (5)	0.73505 (10)	0.0350 (3)
C15	0.8421 (2)	0.41496 (8)	0.83125 (12)	0.0576 (4)
H15	0.8956	0.3808	0.8431	0.069*
C16	0.8361 (3)	0.45118 (10)	0.91033 (14)	0.0758 (6)
H16	0.8854	0.4411	0.9741	0.091*
C17	0.7582 (3)	0.50176 (9)	0.89502 (15)	0.0708 (6)
H17	0.7539	0.5261	0.9481	0.085*
C18	0.6869 (2)	0.51589 (7)	0.80129 (16)	0.0646 (5)
H18	0.6339	0.5503	0.7904	0.078*
C19	0.69195 (19)	0.47969 (6)	0.72125 (13)	0.0495 (4)
H19	0.6421	0.4901	0.6578	0.059*
C20	0.96627 (13)	0.25165 (5)	0.63421 (9)	0.0299 (2)
C21	1.09640 (15)	0.27582 (6)	0.68732 (10)	0.0401 (3)
H21	1.1047	0.3159	0.6931	0.048*
C22	1.21365 (16)	0.24137 (7)	0.73160 (11)	0.0463 (3)
H22	1.2995	0.2582	0.7670	0.056*
C23	1.20252 (17)	0.18199 (7)	0.72292 (12)	0.0492 (4)
H23	1.2815	0.1586	0.7515	0.059*
C24	1.07467 (18)	0.15738 (7)	0.67197 (13)	0.0507 (4)
H24	1.0670	0.1173	0.6668	0.061*
C25	0.95680 (16)	0.19179 (6)	0.62802 (11)	0.0414 (3)
H25	0.8706	0.1745	0.5941	0.050*
C26	0.45234 (13)	0.31694 (5)	0.57847 (9)	0.0324 (2)
C27	0.43792 (15)	0.35074 (6)	0.65915 (11)	0.0402 (3)
H27	0.4812	0.3389	0.7233	0.048*
C28	0.35907 (18)	0.40219 (7)	0.64452 (13)	0.0515 (4)
H28	0.3510	0.4249	0.6988	0.062*
C29	0.29288 (19)	0.41965 (7)	0.55007 (14)	0.0568 (4)
H29	0.2392	0.4539	0.5407	0.068*
C30	0.3059 (2)	0.38666 (8)	0.46969 (13)	0.0578 (4)
H30	0.2610	0.3986	0.4059	0.069*
C31	0.38600 (17)	0.33539 (6)	0.48345 (11)	0.0457 (3)
H31	0.3952	0.3133	0.4287	0.055*
C32	0.45469 (14)	0.19817 (5)	0.51586 (9)	0.0333 (2)
C33	0.30286 (16)	0.19866 (7)	0.50195 (11)	0.0443 (3)
H33	0.2571	0.2273	0.5317	0.053*
C34	0.21953 (19)	0.15660 (8)	0.44398 (13)	0.0566 (4)
H34	0.1180	0.1573	0.4347	0.068*
C35	0.2854 (2)	0.11405 (7)	0.40040 (12)	0.0554 (4)
H35	0.2286	0.0862	0.3612	0.066*
C36	0.4360 (2)	0.11230 (7)	0.41438 (13)	0.0552 (4)
H36	0.4807	0.0830	0.3854	0.066*
C37	0.52041 (17)	0.15431 (6)	0.47158 (12)	0.0463 (3)
H37	0.6219	0.1532	0.4805	0.056*

Comment

Phosphinic amides, a family of phosphorus–nitrogen compounds, have drawn attention for their coordination chemistry [4–8]. Additionally, a series of studies have been performed on structural features of phosphinic amides, such as the geometry of phosphorus or nitrogen atom, and the hydrogen bond patterns [9–12].

The asymmetric unit of the title structure contains one crystallographically independent molecule. The phosphorus atom displays a distorted tetrahedral configuration with characteristic bond angles in the range of 101.53(6)° (N1–P1–C32) to 115.02(6)° (O1–P1–N1). Due to the p–π conjugation effect, the P=O bond length is slightly longer than normal phosphorus–oxygen double bond length (1.4815(9) Å vs 1.45 Å), while the P–N bond length is shorter than the standard phosphorus–nitrogen single bond length (1.6361(11) Å vs 1.77 Å). The nitrogen atom also shows a characteristic non-planar geometry with the bond-angle sum of 343.68(8)°. The geometric parameters shown above are in accordance with literature data of compounds with the similar skeleton [11, 12]. However, the hydrogen bond pattern of the title compound is distinguished from the reported phosphinic amides. There is an intramolecular hydrogen bond between N1–H1 and O2 with a donor-acceptor distance of 2.697 Å, and the molecules are aggregated through intermolecular hydrogen bond between O2–H2A and O1–P1, rather than the literature

deduced hydrogen bond between N1–H1 and O1–P1. The possible functions of this kind of phosphinic amide deserves further studies in detail.

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

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