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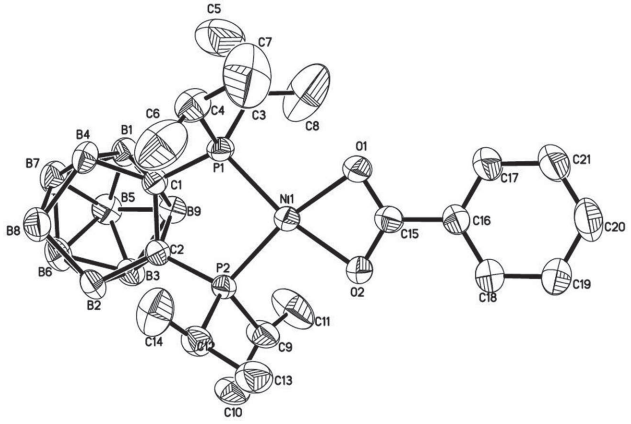
Crystal structure (7,8-bis(diisopropylphosphino)-7,8-dicarba-*nido*-undecaborane-κ^2*P*,*P*′)-(benzoato-κ^2*O*,*O*′)nickel(II), $C_{21}H_{42}B_9NiO_2P_2$



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Abstract

$C_{21}H_{42}B_9NiO_2P_2$, monoclinic, $P2_1/c$, $a = 9.3840(9)$Å, $b = 17.0119(18)$ Å, $c = 19.9631(19)$ Å, $\beta = 112.250(2)°$, $V = 2949.6(5)$Å^3, $Z = 4$, $R_{gt}(F) = 0.0585$, $wR_{ref}(F^2) = 0.1489$, $T = 298$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.40 × 0.30 × 0.23 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	7.8 cm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω
$2\theta_{max}$, completeness:	50°, >97%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14827, 5075, 0.048
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2835
$N(param)_{refined}$:	322
Programs:	Bruker programs [7], SHELX [8]

Source of material

The ligand, 1,2-(P^iPr_2)$_2$-1,2-$C_2B_{10}H_{10}$, was synthesized according to the literature [1]. The title compound was synthesized by the reaction of $NiCl_2$ (1 mmol) and 1,2-(P^iPr_2)$_2$-1,2-$C_2B_{10}H_{10}$ in chloromethane (10 mL) under N_2 atmosphere. The mixture was refluxed for 4 h, then a colourless solution formed. Crystals were obtained by slow evaporation of a dichloromethane-*n*-hexane solution (42.8%, m.p. 423–425 K). **FTIR** (KBr) ν (cm^{-1}): 2989, 2966, 2930, 2872 (C—H); 2614, 2602, 2585, 2556 (B—H); 1071 (C—P).

Experimental details

All H atoms were placed geometrically and treated as riding on their parent atoms, with B—H 1.10, C—H 0.96, with $U_{iso}(H) = 1.2U_{eq}(B)$, $U_{iso}(H) = 1.5U_{eq}(C)$.

Discussion

The environment of the Ni atom is tetra coordinated, in which two positions are occupied by the chelating diphosphine ligand, and the other two come from the oxygen atoms of the benzoato ligand. The mean deviation of only 0.067 Å for the plane P1—P2—Ni—O1—O2 shows the planarity of the coordination. The P1—C1—C2—P2 torsion angle is 12.1(2)° in the free ligand [2, 3]. However, after the ligand is coordinated to Ni, this torsion angle is 2.7(4)° in the complex. The Ni—P bond lengths in the complex are 2.1389(14)Å and 2.1427(13)Å, which are in agreement with [$NiBr_2${1,2-(PPh_2)$_2$-1,2-$C_2B_{10}H_{10}$}]

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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}
Ni1	0.34307(7)	0.28071(3)	0.10394(3)	0.0607(2)
P1	0.28534(15)	0.27175(7)	0.19755(7)	0.0589(3)
P2	0.18322(14)	0.18891(7)	0.05171(6)	0.0540(3)
O2	0.4179(4)	0.3065(2)	0.02805(18)	0.0749(10)
O1	0.4989(4)	0.3618(2)	0.1339(2)	0.0850(11)
B1	−0.0261(7)	0.2217(3)	0.2069(3)	0.0678(16)
H1	−0.0395	0.2717	0.2390	0.081*
C2	0.0709(5)	0.1619(2)	0.1043(2)	0.0539(11)
C1	0.1276(5)	0.2037(3)	0.1821(2)	0.0583(12)
C12	0.2795(6)	0.0999(3)	0.0390(3)	0.0738(14)
H12	0.1989	0.0629	0.0110	0.089*
C16	0.6126(6)	0.4054(3)	0.0524(3)	0.0743(15)
B2	0.0083(7)	0.0730(3)	0.1049(3)	0.0696(16)
H2	0.0660	0.0289	0.0836	0.083*
C15	0.5083(6)	0.3568(3)	0.0728(3)	0.0687(14)
B3	−0.1291(7)	0.1497(4)	0.0683(4)	0.0740(17)
H3	−0.2074	0.1525	0.0111	0.089*
C9	0.0655(8)	0.2200(3)	−0.0400(3)	0.0887(18)
H9	0.1456	0.2313	−0.0590	0.106*
B4	0.1126(8)	0.1452(4)	0.2451(4)	0.0762(18)
H4	0.2200	0.1368	0.2926	0.091*
B5	−0.1845(7)	0.1833(4)	0.1373(4)	0.0813(19)
H5	−0.3009	0.2067	0.1251	0.098*
C3	0.2306(7)	0.3677(3)	0.2245(3)	0.0898(17)
H3A	0.1185	0.3663	0.1992	0.108*
C13	0.3789(7)	0.1164(3)	−0.0054(3)	0.0934(18)
H13A	0.4529	0.1565	0.0181	0.140*
H13B	0.3143	0.1338	−0.0530	0.140*
H13C	0.4316	0.0692	−0.0090	0.140*
C4	0.4489(7)	0.2349(4)	0.2749(3)	0.0870(17)
H4A	0.4193	0.2339	0.3170	0.104*
C10	−0.0223(7)	0.1547(3)	−0.0912(3)	0.0931(18)
H10A	−0.0968	0.1325	−0.0743	0.140*
H10B	0.0485	0.1146	−0.0926	0.140*
H10C	−0.0739	0.1759	−0.1388	0.140*
B6	−0.1502(8)	0.0813(4)	0.1300(4)	0.089(2)
H6	−0.2448	0.0384	0.1120	0.106*
B9	−0.0441(7)	0.2355(4)	0.1162(3)	0.0683(16)
H9A	−0.0630	0.2934	0.0897	0.082*
C11	−0.0085(9)	0.2925(4)	−0.0500(4)	0.127(2)
H11A	−0.0479	0.3059	−0.1005	0.191*
H11B	0.0634	0.3320	−0.0230	0.191*
H11C	−0.0920	0.2897	−0.0334	0.191*
C17	0.7083(8)	0.4580(4)	0.1001(4)	0.111(2)
H17	0.7049	0.4625	0.1459	0.133*
C18	0.6189(8)	0.3985(3)	−0.0153(4)	0.101(2)
H18	0.5560	0.3625	−0.0484	0.121*
C5	0.2444(10)	0.3738(5)	0.2984(4)	0.155(3)
H5A	0.3511	0.3711	0.3298	0.232*
H5B	0.1893	0.3314	0.3094	0.232*
H5C	0.2021	0.4231	0.3055	0.232*
C14	0.3769(8)	0.0590(4)	0.1089(4)	0.120(2)
H14A	0.4149	0.0103	0.0980	0.180*
H14B	0.3153	0.0489	0.1369	0.180*
H14C	0.4620	0.0921	0.1361	0.180*
C19	0.7179(9)	0.4448(4)	−0.0337(4)	0.119(2)
H19	0.7222	0.4403	−0.0793	0.143*
C6	0.5041(9)	0.1581(5)	0.2668(4)	0.147(3)
H6A	0.5575	0.1610	0.2343	0.220*
H6B	0.4185	0.1227	0.2476	0.220*
H6C	0.5730	0.1394	0.3131	0.220*
B7	−0.0819(8)	0.1268(4)	0.2177(4)	0.090(2)
H7	−0.1301	0.1142	0.2589	0.108*
C20	0.8101(8)	0.4977(4)	0.0150(5)	0.116(2)
H20	0.8753	0.5301	0.0019	0.139*
C21	0.8071(9)	0.5035(4)	0.0834(5)	0.123(3)
H21	0.8723	0.5381	0.1173	0.148*
C7	0.5872(10)	0.2842(6)	0.2929(5)	0.202(5)
H7A	0.5622	0.3376	0.2995	0.303*
H7B	0.6657	0.2654	0.3367	0.303*
H7C	0.6242	0.2818	0.2542	0.303*
C8	0.2684(13)	0.4353(5)	0.1931(6)	0.199(4)
H8A	0.2548	0.4241	0.1439	0.299*
H8B	0.2023	0.4780	0.1940	0.299*
H8C	0.3736	0.4496	0.2200	0.299*
B8	0.0250(8)	0.0532(4)	0.1976(4)	0.087(2)
H8	0.0822	−0.0020	0.2219	0.104*

[4, 5]. The average bond lengths of C—P in the complexes (1.809(4)Å) are different to the P–C distance 1.894(3)Å in 1,2-$(P^iPr_2)_2$-1,2-$C_2B_{10}H_{10}$ [6].

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