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The crystal structure of dichlorobis(2-(dicyclohexylphosphino)-2',4',6'-tri-*i*-propyl-1,1'-biphenyl) palladium(II)-dichloroform, C₆₈H₁₀₀Cl₈P₂Pd

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.18 × 0.16 × 0.15 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	5.47 mm ⁻¹
Diffractometer, scan mode:	Xcalibur, ω
$\theta_{\max}$, completeness:	67.1°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13103, 6479, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5700
$N(\text{param})_{\text{refined}}$:	364
Programs:	CrysAlis ^{PRO} [1], SHELX [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pd1	1.0000	0.5000	0.0000	0.04090(13)
Cl1	0.83671(14)	0.61644(7)	0.12110(7)	0.0724(3)
P1	0.92615(9)	0.36171(6)	0.07320(5)	0.03562(19)
C1	0.8978(4)	0.2758(3)	-0.0081(2)	0.0437(7)
H1	1.0005	0.2364	-0.0467	0.052*
C2	0.8157(5)	0.3293(3)	-0.0766(3)	0.0615(10)
H2A	0.7085	0.3620	-0.0440	0.074*
H2B	0.8590	0.3824	-0.1053	0.074*
C3	0.8340(6)	0.2487(4)	-0.1510(3)	0.0736(13)
H3A	0.7814	0.2819	-0.1941	0.088*
H3B	0.9411	0.2192	-0.1855	0.088*
C4	0.7713(6)	0.1641(4)	-0.1091(4)	0.0806(14)
H4A	0.7929	0.1115	-0.1576	0.097*
H4B	0.6616	0.1922	-0.0826	0.097*
C5	0.8381(6)	0.1143(4)	-0.0339(3)	0.0742(13)
H5A	0.9444	0.0751	-0.0623	0.089*
H5B	0.7848	0.0665	-0.0039	0.089*
C6	0.8256(5)	0.1946(3)	0.0390(3)	0.0561(9)
H6A	0.8770	0.1608	0.0825	0.067*
H6B	0.7193	0.2278	0.0733	0.067*
C7	1.0784(4)	0.2778(3)	0.1167(2)	0.0408(7)
H7	1.0282	0.2508	0.1740	0.049*
C8	1.1896(5)	0.1851(3)	0.0514(3)	0.0607(10)
H8A	1.2413	0.2089	-0.0064	0.073*
H8B	1.1341	0.1421	0.0376	0.073*
C9	1.3068(6)	0.1216(4)	0.0962(4)	0.0818(15)
H9A	1.2552	0.0930	0.1511	0.098*
H9B	1.3787	0.0644	0.0530	0.098*
C10	1.3921(5)	0.1856(4)	0.1229(4)	0.0823(15)
H10A	1.4528	0.2080	0.0674	0.099*
H10B	1.4605	0.1439	0.1544	0.099*
C11	1.2832(6)	0.2789(4)	0.1859(4)	0.0765(13)
H11A	1.3399	0.3218	0.1983	0.092*
H11B	1.2317	0.2566	0.2446	0.092*

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Abstract

C₆₈H₁₀₀Cl₈P₂Pd, triclinic, $P\bar{1}$ (no. 2), $a = 9.6961(4)$ Å, $b = 13.6069(6)$ Å, $c = 15.1642(5)$ Å, $\alpha = 88.456(3)^\circ$, $\beta = 72.251(3)^\circ$, $\gamma = 72.376(4)^\circ$, $V = 1811.64(12)$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.0483$, $wR_{\text{ref}}(F^2) = 0.1332$, $T = 291$ K.

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The molecular 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.

Source of material

The title compound was prepared from the reaction of PdCl₂(CH₃CN)₂ and 2-(dicyclohexylphosphino)-2',4',6'-tri-*i*-propyl-1,1'-biphenyl according to literature methods [3]. Crystals of the title compound were obtained by recrystallization from chloroform at room temperature.

Experimental details

A suitable crystal was selected and measured on a Xcalibur, Eos, Gemini diffractometer. The crystal was kept at 291.15 K during data collection using the CrysAlisPro program [1]. The

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Table 2 (continued)

Atom	x	y	z	U _{iso} */U _{eq}
C12	1.1655(5)	0.3422(3)	0.1420(3)	0.0593(10)
H12A	1.0944	0.3997	0.1851	0.071*
H12B	1.2164	0.3703	0.0866	0.071*
C13	0.7436(4)	0.4038(2)	0.1673(2)	0.0378(7)
C14	0.6270(4)	0.4632(3)	0.1339(3)	0.0536(9)
H14	0.6513	0.4775	0.0717	0.064*
C15	0.4780(5)	0.5011(4)	0.1893(3)	0.0670(12)
H15	0.4029	0.5384	0.1643	0.080*
C16	0.4412(5)	0.4835(4)	0.2821(3)	0.0712(12)
H16	0.3411	0.5084	0.3207	0.085*
C17	0.5542(4)	0.4287(4)	0.3166(3)	0.0606(10)
H17	0.5284	0.4176	0.3795	0.073*
C18	0.7064(4)	0.3885(3)	0.2627(2)	0.0407(7)
C19	0.8127(4)	0.3336(3)	0.3156(2)	0.0434(7)
C20	0.8338(4)	0.2293(3)	0.3315(2)	0.0505(8)
C21	0.9264(5)	0.1839(3)	0.3872(3)	0.0642(11)
H21	0.9407	0.1146	0.3988	0.077*
C22	0.9966(5)	0.2384(4)	0.4252(3)	0.0648(11)
C23	0.9689(5)	0.3406(4)	0.4109(3)	0.0603(10)
H23	1.0121	0.3787	0.4382	0.072*
C24	0.8791(4)	0.3904(3)	0.3573(2)	0.0485(8)
C25	0.7565(6)	0.1669(3)	0.2943(3)	0.0653(11)
H25	0.7210	0.2052	0.2456	0.078*
C26	0.8615(7)	0.0606(4)	0.2505(4)	0.0871(16)
H26A	0.8863	0.0174	0.2981	0.131*
H26B	0.9531	0.0674	0.2071	0.131*
H26C	0.8112	0.0300	0.2187	0.131*
C27	0.6146(8)	0.1569(5)	0.3707(5)	0.108(2)
H27A	0.6442	0.1256	0.4224	0.163*
H27B	0.5695	0.1146	0.3464	0.163*
H27C	0.5421	0.2242	0.3909	0.163*
C28	1.0964(6)	0.1866(5)	0.4848(4)	0.0833(15)
H28	1.1453	0.2368	0.4948	0.100*
C29	1.2222(8)	0.0941(6)	0.4365(6)	0.137(3)
H29A	1.2960	0.0756	0.4691	0.205*
H29B	1.2696	0.1088	0.3743	0.205*
H29C	1.1827	0.0377	0.4345	0.205*
C30	1.0032(7)	0.1710(6)	0.5781(4)	0.109(2)
H30A	0.9290	0.2356	0.6061	0.163*
H30B	1.0678	0.1452	0.6161	0.163*
H30C	0.9523	0.1219	0.5728	0.163*
C31	0.8448(5)	0.5067(3)	0.3526(3)	0.0578(9)
H31	0.8088	0.5260	0.2989	0.069*
C32	0.9818(6)	0.5458(4)	0.3412(4)	0.0800(14)
H32A	0.9507	0.6196	0.3389	0.120*
H32B	1.0606	0.5146	0.2848	0.120*
H32C	1.0196	0.5277	0.3930	0.120*
C33	0.7157(6)	0.5618(4)	0.4397(4)	0.0870(15)
H33A	0.6248	0.5458	0.4419	0.131*
H33B	0.6972	0.6351	0.4379	0.131*
H33C	0.7442	0.5390	0.4939	0.131*
C34	0.5670(7)	0.8115(5)	0.2742(5)	0.108(2)
H34	0.6399	0.7502	0.2352	0.130*
Cl2	0.6500(3)	0.84638(19)	0.3463(2)	0.1669(11)
Cl3	0.5251(4)	0.9106(2)	0.20218(18)	0.1961(14)
Cl4	0.4119(3)	0.7802(3)	0.3357(3)	0.2417(19)

structure was solved with the SHELXS structure solution program using Patterson Method and refined with the SHELXL [2] refinement package using least-squares minimisation. Hydrogen atoms were refined using the appropriate riding models provided by the SHELXL program.

Comment

Palladium-catalyzed coupling reactions provide an extremely powerful method for the formation of carbon-carbon or carbon-heteroatom bonds in organic synthesis [3–6]. Great progress has been obtained for coupling reactions by using Pd catalysts with bulky electron-rich phosphines or *N*-heterocyclic carbenes ligands [7–9]. Among them, Pd complexes derived from bulky monophosphinobiaryl ligands are highly effective catalysts for this transformation [10–13]. Here we report the crystal structure of the title bis(monophosphinobiaryl) Pd complex. It has a *trans* square planar geometry, including a center of inversion about the palladium atom, in which the sterically demanding biaryl portion of the ligands point away from the crowded Pd center. The Pd–P [2.3464(8) Å] and Pd–Cl [2.2924(9) Å] bond lengths of the title complex are similar to those of related Pd complexes [11, 12, 14–16]. All bond lengths and angles are within normal ranges and in excellent agreement with those reported for the structure of the unsolvated complex [17]. CHCl₃ molecules are present in the crystal and form intermolecular C–H···Cl hydrogen bonds [18] (Cl···H = 2.450 Å) by acting as hydrogen-bond donors. The ambient-temperature data indicates there is some evidence of disorder in the solvent molecule, which has not been modelled.

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