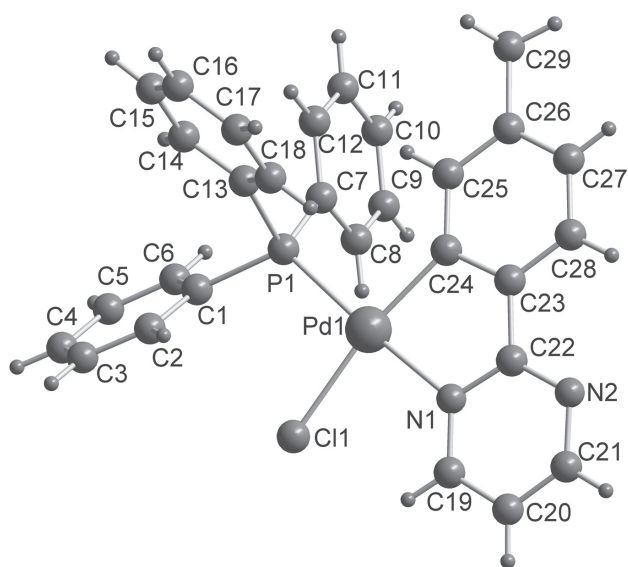


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Crystal structure of (2-*p*-tolylpyrimidine- κ^2C,N)(triphenylphosphane- κP) palladium(II), $C_{29}H_{24}ClN_2PPd$



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Abstract

$C_{29}H_{24}ClN_2PPd$, triclinic, $P\bar{1}$ (no. 2), $a = 9.9885(8)$ Å, $b = 11.5836(8)$ Å, $c = 11.6133(9)$ Å, $\alpha = 76.342(6)^\circ$, $\beta = 89.821(6)^\circ$, $\gamma = 71.435(7)^\circ$, $V = 1233.96(16)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0337$, $wR_{ref}(F^2) = 0.0841$, $T = 291$ 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.

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.20 × 0.18 × 0.18 mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ :	7.83 mm ⁻¹
Diffractometer, scan mode:	Xcalibur, ω
θ_{max} , completeness:	67.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	8906, 4400, 0.031
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 3959
$N(param)_{refined}$:	309
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3]

Source of material

The title compound was obtained from the bridge-splitting reaction of the palladacyclic dimer of 2-*p*-tolylpyrimidine and PPh₃ according to the literature [4] and recrystallized from a dichloromethane/petroleum ether solution at room temperature to give the desired crystals suitable for single-crystal X-ray diffraction.

Experimental details

Data collection and reduction were performed using the CrysAlis^{PRO} software [1]. The structures were solved by direct methods and refined by the least-squares method with Olex2 [2] and SHELXL [3]. All non-hydrogen atoms were refined anisotropically, while hydrogen atoms were placed in geometrically calculated positions and refined using the riding model and appropriate SHELXL AFIX commands.

Comment

Pyrimidine derivatives are an important class of molecules due to their broad area of application in pharmaceuticals and functional materials [5–7]. In addition, pyrimidines as ligands are also used in organometallic chemistry and catalysis [8–10]. For example, cyclometalated pyrimidine complexes have been successfully applied to coupling reactions [11–13]. Here we report the crystal structure of the title cyclopalladated 2-*p*-tolylpyrimidine complex.

The title complex adopts a *trans* configuration of the coordinated PPh₃ ligand to the nitrogen atom of the pyrimidine ligand [14]. The palladium center feature a square planar

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

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Pd1	0.75900(3)	0.82567(2)	0.80146(2)	0.03241(10)
Cl1	0.57819(12)	0.82255(11)	0.93271(11)	0.0602(3)
P1	0.72495(9)	0.67552(8)	0.72113(8)	0.0320(2)
N1	0.7994(3)	0.9666(3)	0.8685(3)	0.0367(7)
N2	0.9434(4)	1.0973(3)	0.8417(4)	0.0536(9)
C1	0.5891(4)	0.6072(3)	0.7807(3)	0.0352(8)
C2	0.4498(4)	0.6851(4)	0.7715(4)	0.0480(10)
H2	0.4277	0.7709	0.7383	0.058*
C3	0.3428(4)	0.6369(4)	0.8112(4)	0.0537(11)
H3	0.2496	0.6902	0.8044	0.064*
C4	0.3740(5)	0.5111(5)	0.8602(4)	0.0591(12)
H4	0.3024	0.4785	0.8870	0.071*
C5	0.5118(5)	0.4329(4)	0.8697(4)	0.0619(12)
H5	0.5331	0.3473	0.9038	0.074*
C6	0.6188(4)	0.4794(4)	0.8295(4)	0.0472(10)
H6	0.7112	0.4249	0.8350	0.057*
C7	0.8840(4)	0.5371(3)	0.7422(3)	0.0347(8)
C8	0.9489(4)	0.4875(4)	0.8553(4)	0.0530(11)
H8	0.9137	0.5269	0.9154	0.064*
C9	1.0662(5)	0.3794(4)	0.8810(4)	0.0574(12)
H9	1.1085	0.3461	0.9581	0.069*
C10	1.1194(4)	0.3221(4)	0.7936(4)	0.0537(11)
H10	1.1984	0.2498	0.8109	0.064*
C11	1.0574(5)	0.3700(4)	0.6808(4)	0.0607(12)
H11	1.0946	0.3307	0.6212	0.073*
C12	0.9390(5)	0.4775(4)	0.6543(4)	0.0505(10)
H12	0.8965	0.5094	0.5771	0.061*
C13	0.6650(4)	0.7275(4)	0.5632(3)	0.0374(8)
C14	0.6190(4)	0.6514(4)	0.5078(4)	0.0523(10)
H14	0.6243	0.5709	0.5501	0.063*
C15	0.5656(5)	0.6943(6)	0.3906(4)	0.0671(14)
H15	0.5379	0.6417	0.3535	0.081*
C16	0.5529(5)	0.8150(5)	0.3279(4)	0.0689(15)
H16	0.5149	0.8445	0.2494	0.083*
C17	0.5966(5)	0.8904(5)	0.3819(4)	0.0666(14)
H17	0.5892	0.9713	0.3395	0.080*
C18	0.6520(4)	0.8485(4)	0.4995(4)	0.0496(10)
H18	0.6804	0.9015	0.5355	0.060*
C19	0.7340(4)	1.0196(4)	0.9519(4)	0.0477(10)
H19	0.6618	0.9939	0.9886	0.057*
C20	0.7720(5)	1.1120(4)	0.9847(4)	0.0558(11)
H20	0.7285	1.1485	1.0442	0.067*
C21	0.8762(5)	1.1480(4)	0.9261(4)	0.0583(12)
H21	0.9018	1.2116	0.9462	0.070*
C22	0.9032(4)	1.0064(3)	0.8160(4)	0.0419(9)
C23	0.9722(4)	0.9398(3)	0.7286(4)	0.0415(9)
C24	0.9234(4)	0.8449(3)	0.7051(3)	0.0353(8)
C25	0.9952(4)	0.7810(4)	0.6246(4)	0.0429(9)
H25	0.9656	0.7183	0.6070	0.051*
C26	1.1100(4)	0.8066(4)	0.5690(4)	0.0477(10)
C27	1.1522(4)	0.9017(4)	0.5915(4)	0.0508(10)
H27	1.2266	0.9213	0.5526	0.061*
C28	1.0855(4)	0.9681(4)	0.6713(4)	0.0487(10)

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H28	1.1156	1.0314	0.6870	0.058*
C29	1.1881(5)	0.7302(5)	0.4862(5)	0.0680(14)
H29A	1.2263	0.6434	0.5285	0.102*
H29B	1.2638	0.7601	0.4561	0.102*
H29C	1.1239	0.7384	0.4211	0.102*

coordination geometry. The Pd–P [2.2707(9) Å] and Pd–N [2.110(3) Å] bond lengths of the title complex are similar to those of related palladacycles [8, 15–17]. The chelate ring of the palladacycle Pd1–N1–C22–C23–C24 is virtually planar, with the greatest deviation from the fitted least-squares mean plane being 0.023(3) Å for N1. The title complex exists as a dimer in the crystal due to intermolecular hydrogen bonds between chlorine atom and the adjacent C–H group of PPH₃ (Cl···H = 2.886 Å) [17–19].

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