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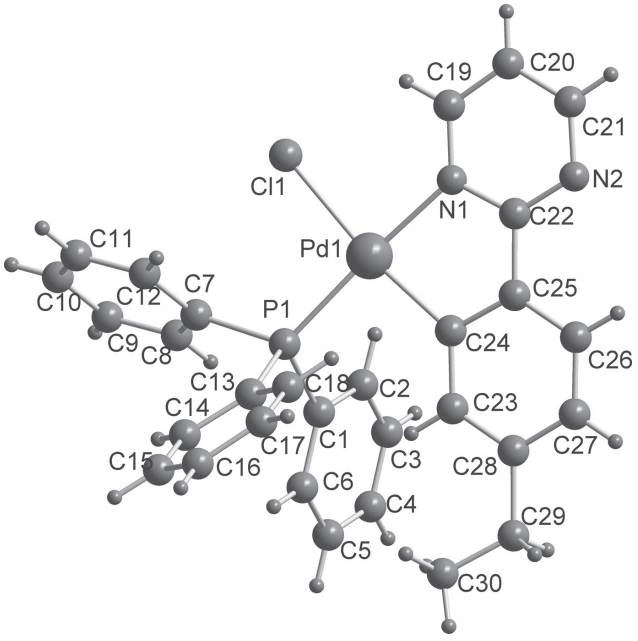
Crystal structure of chlorido (2-(4-ethylphenyl) pyrimidine-k^2*C*,*N*)(triphenylphosphane-k*P*) palladium(II), $C_{30}H_{26}ClN_2PPd$



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Abstract

$C_{30}H_{26}ClN_2PPd$, triclinic, $P\bar{1}$ (no. 2), $a = 9.1278(5)$ Å, $b = 9.5892(7)$ Å, $c = 15.5255(10)$ Å, $\alpha = 79.827(6)°$, $\beta = 89.131(5)°$, $\gamma = 78.358(5)°$, $V = 1309.74(14)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0342$, $wR_{ref}(F^2) = 0.0706$, $T = 291$ 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 × 0.17 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.89 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10890, 5353, 0.028
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4548
$N(param)_{refined}$:	317
Programs:	CrysAlis^{PRO} [1], SHELX [2]

Source of material

The title compound was prepared from the bridge-splitting reaction of the corresponding palladacyclic dimer of 2-(4-ethylphenyl)pyrimidine and PPh_3 according to the literature [3]. Crystals of the title compound were obtained by recrystallization from a dichloromethane/petroleum ether solution at room temperature.

Experimental details

A suitable crystal was selected and measured on an Xcalibur, Eos, Gemini diffractometer. The crystal was kept at 291.15 K during data collection using the CrysAlis^{Pro} program [1]. The 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 a constrained model and the appropriate AFIX commands.

Comment

Palladacycles have received considerable attention due to their unique physical properties and their extensively applications as highly efficient precatalysts [4–6]. Among them, a wide variety of phosphines adducts of palladacycles have been successfully used in coupling reactions [7–9]. The cyclopalladation via C—H bond activation is usually achieved by using various directing groups, such as amine, imine, pyridine, and oxazoline groups. However, there have been a few reports which demonstrated that pyrimidine can act as a directing group for cyclopalladation [10, 11]. Here we report

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Pd1	0.74506(2)	0.88360(2)	0.684667(14)	0.03093(8)
Cl1	0.56160(8)	1.02502(8)	0.76113(5)	0.04411(19)
P1	0.83848(7)	0.72944(7)	0.80782(5)	0.03113(17)
N1	0.6750(2)	1.0244(2)	0.56613(15)	0.0344(5)
N2	0.7341(3)	1.0641(3)	0.41588(17)	0.0517(7)
C1	0.8547(3)	0.5390(3)	0.80088(18)	0.0345(6)
C2	0.7373(3)	0.5011(3)	0.7601(2)	0.0459(8)
H2	0.6580	0.5729	0.7349	0.055*
C3	0.7374(4)	0.3584(4)	0.7568(2)	0.0598(9)
H3	0.6573	0.3344	0.7305	0.072*
C4	0.8548(5)	0.2524(4)	0.7919(2)	0.0633(10)
H4	0.8552	0.1564	0.7885	0.076*
C5	0.9724(4)	0.2865(4)	0.8321(2)	0.0640(10)
H5	1.0520	0.2139	0.8563	0.077*
C6	0.9722(3)	0.4302(3)	0.8367(2)	0.0475(8)
H6	1.0518	0.4533	0.8640	0.057*
C7	0.7382(3)	0.7399(3)	0.91071(18)	0.0339(6)
C8	0.6775(3)	0.6281(3)	0.95437(19)	0.0429(7)
H8	0.6806	0.5463	0.9293	0.052*
C9	0.6116(3)	0.6348(4)	1.0350(2)	0.0548(9)
H9	0.5729	0.5570	1.0639	0.066*
C10	0.6034(3)	0.7533(4)	1.0719(2)	0.0579(9)
H10	0.5586	0.7579	1.1258	0.070*
C11	0.6617(4)	0.8670(4)	1.0292(2)	0.0639(10)
H11	0.6563	0.9488	1.0544	0.077*
C12	0.7280(4)	0.8616(3)	0.9493(2)	0.0532(9)
H12	0.7663	0.9399	0.9209	0.064*
C13	1.0210(3)	0.7584(3)	0.83757(19)	0.0344(6)
C14	1.0815(3)	0.7078(3)	0.9215(2)	0.0509(8)
H14	1.0307	0.6542	0.9631	0.061*
C15	1.2178(4)	0.7371(4)	0.9435(2)	0.0560(9)
H15	1.2582	0.7027	0.9997	0.067*
C16	1.2936(3)	0.8171(3)	0.8824(2)	0.0486(8)
H16	1.3859	0.8346	0.8969	0.058*
C17	1.2325(3)	0.8705(3)	0.8006(2)	0.0486(8)
H17	1.2822	0.9265	0.7597	0.058*
C18	1.0972(3)	0.8416(3)	0.7782(2)	0.0413(7)
H18	1.0566	0.8787	0.7223	0.050*
C19	0.5615(3)	1.1377(3)	0.5519(2)	0.0440(8)
H19	0.5029	1.1625	0.5986	0.053*
C20	0.5294(3)	1.2186(3)	0.4701(2)	0.0530(9)
H20	0.4503	1.2979	0.4600	0.064*
C21	0.6193(4)	1.1773(4)	0.4036(2)	0.0572(9)
H21	0.5994	1.2305	0.3474	0.069*
C22	0.7585(3)	0.9901(3)	0.4971(2)	0.0377(7)
C23	1.0091(3)	0.6646(3)	0.62065(19)	0.0391(7)
H23	1.0226	0.6125	0.6774	0.047*
C24	0.8946(3)	0.7864(3)	0.60401(18)	0.0337(6)
C25	0.8779(3)	0.8621(3)	0.51721(19)	0.0375(7)
C26	0.9742(3)	0.8157(3)	0.4520(2)	0.0502(8)
H26	0.9628	0.8672	0.3951	0.060*
C27	1.0856(3)	0.6943(4)	0.4723(2)	0.0537(9)
H27	1.1489	0.6641	0.4288	0.064*
C28	1.1045(3)	0.6170(3)	0.5563(2)	0.0468(8)
C29	1.2286(4)	0.4835(4)	0.5768(3)	0.0664(11)
H29A	1.1988	0.4072	0.5520	0.080*
H29B	1.3170	0.5054	0.5463	0.080*
C30	1.2715(6)	0.4259(6)	0.6679(3)	0.146(3)
H30A	1.3637	0.3560	0.6709	0.219*
H30B	1.1946	0.3805	0.6962	0.219*
H30C	1.2847	0.5033	0.6968	0.219*

the crystal structure of the title PPh_3-adduct of cyclopalladated 2-(4-ethylphenyl)pyrimidine complex.

The title complex has a *trans*-geometry in the solid state and the Pd atom is in a slightly distorted square-planar environment provided by a chloride, a P atom, and the C atom and the N atom of the 2-(4-ethylphenyl)pyrimidine ligand. The Pd—P [2.2607(8) Å] and Pd—N [2.104(2) Å] bond lengths of the title complex are similar to those of the related palladacycles [3, 11, 12]. The pyrimidyl and phenyl ring are approximately coplanar. In the crystal there exist C—H$\cdots$N (N$\cdots$H = 2.717 Å) and C—H$\cdots$Cl (Cl$\cdots$H = 2.895 Å) hydrogen bonds [13–15], which are attributed to construct the 1D lamellar structure.

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