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Crystal structure of *cis*-bis[2-(diphenylphosphinoaminato)-4-methylpyridine]platinum(II), $Pt(C_{18}H_{16}N_2P)_2$

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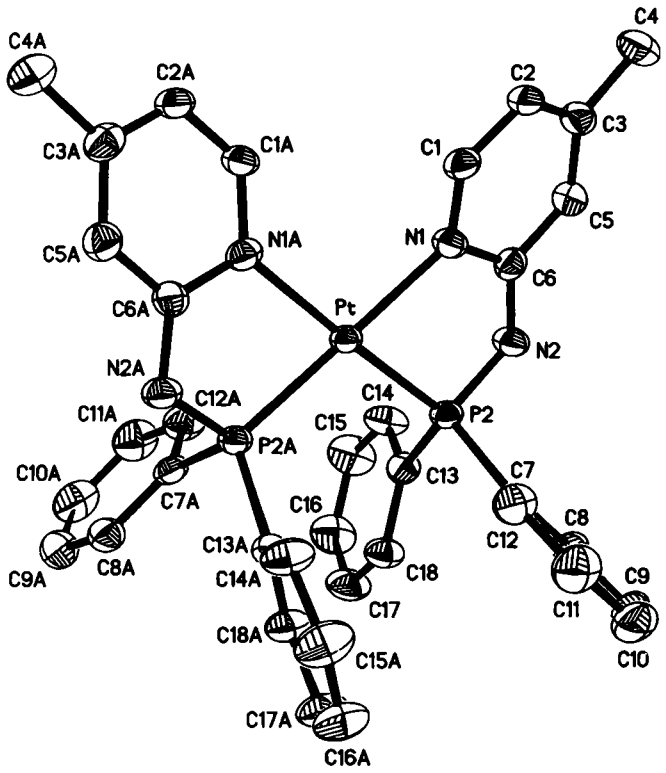

Abstract

$C_{36}H_{32}N_4P_2Pt$, monoclinic, $C12/c1$ (no. 15), a = 22.183(1) Å, b = 10.4130(6) Å, c = 15.2670(9) Å, β = 118.038(5)°, V = 3112.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.024, $wR_{ref}(F^2)$ = 0.069, T = 193 K.

Source of material

A solution of 2-(diphenylphosphinoamino)-4-methylpyridine (150 mg, 0.51 mmol) in toluene (5 mL) was added to [(μ_2-C_2H_4)Pt(PPh_3)$_2$] (381 mg, 0.51 mmol), dissolved in the same solvent (5 mL). The reaction mixture was stirred at room temperature for 2 h. The solvent was removed in vacuo, the residue washed with hexane and dried, to give a yellow powder. ^{31}P NMR measurements revealed it to be a mixture of *cis*/*trans*-1 (in an 1:4 ratio) and $Pt(PPh_3)_n$ (n = 3,4). Crystals of the title compound could be obtained from a C_6D_6 solution at room temperature.

Discussion

Homoleptic platinum and palladium complexes of deprotonated 2-(diphenylphosphinoamino)pyridine have been introduced recently by Woollins et al. [1]. The title compound is a new member of this type of late transition metal complexes. A *cis* arrangement is accomplished with respect to the phosphorus atoms and a square planar coordination is observed. The sum of the angles around Pt is 360°. The chelating angle $N_{pyridine}$–Pt–P is 78.86° and the dihedral angle between the two chelating rings is 7.6°. The five-membered ring Pt-N-C-N-P is nearly planar (0.1035 Å deviation). Both phenyl substituents of the phosphorus atoms are twisted with regard to the five-membered chelating ring with dihedral angles of 72.1° and 62.4°.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.15 × 0.25 × 0.37 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	46.43 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS II, ω
$2\theta_{max}$:	51.46°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	20202, 2942
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2830
$N(param)_{refined}$:	195
Programs:	SIR97 [2], SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	8*f*	0.0340	0.3328	0.8311	0.035
H(2)	8*f*	0.1229	0.4745	0.8884	0.037
H(4A)	8*f*	0.2330	0.5308	0.9162	0.067
H(4B)	8*f*	0.2614	0.4512	0.8536	0.067
H(4C)	8*f*	0.2884	0.4198	0.9687	0.067
H(5)	8*f*	0.2423	0.1951	0.8688	0.038
H(8)	8*f*	0.1848	−0.2802	0.9225	0.041
H(9)	8*f*	0.2153	−0.3767	1.0762	0.052
H(10)	8*f*	0.1564	−0.3228	1.1640	0.059
H(11)	8*f*	0.0699	−0.1707	1.1014	0.057
H(12)	8*f*	0.0396	−0.0718	0.9481	0.045
H(14)	8*f*	0.1197	−0.0809	0.6457	0.045
H(15)	8*f*	0.1112	−0.2262	0.5253	0.057
H(16)	8*f*	0.0766	−0.4359	0.5276	0.055
H(17)	8*f*	0.0498	−0.5018	0.6505	0.051
H(18)	8*f*	0.0574	−0.3554	0.7723	0.039

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt	4*e*	0	0.04983(1)	¾	0.0258(1)	0.0163(1)	0.0202(1)	0	0.00965(8)	0
C(1)	8*f*	0.0766(2)	0.3045(3)	0.8375(2)	0.036(2)	0.020(2)	0.027(2)	0.005(1)	0.011(1)	0.001(1)
C(2)	8*f*	0.1289(2)	0.3896(3)	0.8710(3)	0.039(2)	0.018(2)	0.029(2)	–0.000(1)	0.011(1)	–0.001(1)
C(3)	8*f*	0.1923(2)	0.3501(4)	0.8797(2)	0.040(2)	0.027(2)	0.025(2)	–0.006(2)	0.013(1)	0.000(1)
C(4)	8*f*	0.2487(3)	0.4463(4)	0.9069(4)	0.043(2)	0.030(2)	0.056(3)	–0.011(2)	0.019(2)	–0.005(2)
C(5)	8*f*	0.1997(2)	0.2240(4)	0.8619(3)	0.033(2)	0.029(2)	0.031(2)	–0.002(1)	0.013(1)	0.000(1)
C(6)	8*f*	0.1450(2)	0.1350(3)	0.8333(2)	0.031(2)	0.026(2)	0.022(1)	0.002(1)	0.010(1)	0.002(1)
C(7)	8*f*	0.1090(2)	–0.1678(3)	0.9203(2)	0.033(2)	0.019(2)	0.024(2)	–0.004(1)	0.009(1)	–0.004(1)
C(8)	8*f*	0.1613(2)	–0.2578(4)	0.9587(3)	0.040(2)	0.027(2)	0.028(2)	0.002(2)	0.011(1)	0.001(1)
C(9)	8*f*	0.1794(2)	–0.3154(4)	1.0498(3)	0.051(2)	0.031(2)	0.033(2)	0.004(2)	0.006(2)	0.007(2)
C(10)	8*f*	0.1446(3)	–0.2828(4)	1.1021(3)	0.074(3)	0.040(2)	0.026(2)	–0.007(2)	0.018(2)	0.004(2)
C(11)	8*f*	0.0932(3)	–0.1929(5)	1.0649(3)	0.064(3)	0.048(3)	0.033(2)	–0.002(2)	0.026(2)	–0.002(2)
C(12)	8*f*	0.0750(2)	–0.1342(4)	0.9736(3)	0.048(2)	0.034(2)	0.032(2)	0.001(2)	0.020(2)	–0.002(2)
C(13)	8*f*	0.0887(2)	–0.2034(3)	0.7205(2)	0.030(2)	0.024(2)	0.026(2)	0.002(1)	0.013(1)	–0.002(1)
C(14)	8*f*	0.1051(2)	–0.1663(4)	0.6469(3)	0.054(2)	0.025(2)	0.043(2)	–0.007(2)	0.031(2)	–0.004(2)
C(15)	8*f*	0.1003(3)	–0.2526(5)	0.5757(3)	0.068(3)	0.047(3)	0.041(2)	–0.009(2)	0.038(2)	–0.011(2)
C(16)	8*f*	0.0798(2)	–0.3769(4)	0.5771(3)	0.063(3)	0.037(2)	0.044(2)	–0.003(2)	0.031(2)	–0.014(2)
C(17)	8*f*	0.0638(2)	–0.4159(4)	0.6495(3)	0.060(3)	0.022(2)	0.046(2)	–0.005(2)	0.025(2)	–0.007(2)
C(18)	8*f*	0.0683(2)	–0.3287(3)	0.7220(3)	0.045(2)	0.022(2)	0.033(2)	–0.003(1)	0.021(2)	–0.003(1)
N(1)	8*f*	0.0813(2)	0.1813(3)	0.8125(2)	0.032(1)	0.021(1)	0.025(1)	–0.004(1)	0.012(1)	–0.001(1)
N(2)	8*f*	0.1554(2)	0.0098(3)	0.8261(2)	0.032(2)	0.020(2)	0.036(2)	–0.002(1)	0.017(1)	–0.002(1)
P(2)	8*f*	0.08968(4)	–0.08206(9)	0.80628(6)	0.0276(4)	0.0177(4)	0.0234(4)	0.0011(3)	0.0112(3)	–0.0001(3)

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