

## Crystal Structure of *trans*-Bis[2-(*p*-fluorophenyl)-2-oxazoline- $\kappa^1N$ ]platinum(II) dichloride]

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The title material was synthesised by the treatment of PtCl<sub>2</sub> with 2-(4'-fluorophenyl)-2-oxazoline in a MeOH solution. The crystal system is monoclinic (space group  $P2_1/n$ ;  $Z = 2$ ); the unit-cell dimensions are  $a = 7.7152(7)\text{Å}$ ,  $b = 11.517(1)\text{Å}$ ,  $c = 11.168(1)\text{Å}$  with  $\beta = 110.123(2)^\circ$ ;  $V = 931.8(2)\text{Å}^3$ . The final  $R$  value was 0.0149 (2030 observed reflections:  $I > 2\sigma(I)$ ). The Pt atom coordinates with two  $N$ -bound oxazolines in addition to two (transoidally disposed) chloride atoms; these interactions result in a square planar geometry around the metal center.

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Platinum containing heterocyclic compounds has been used extensively as regio- and enantio-selective catalysts, biomedical agents and in many fundamental studies of coordination chemistry.<sup>1</sup> As part of our continuing investigations into the coordination chemistry of oxazoles, and specifically 2-oxazolines (*i.e.*, 4,5-dihydrooxazoles),<sup>2</sup> we report herein on the synthesis and characterisation of a halide complex of formally Pt<sup>2+</sup> containing the ligand 2-(*p*-fluorophenyl)-2-oxazoline. This ligand was chosen as part of a larger program under development to investigate the medicinal properties of an extensive series of Pt-oxazole systems.

A methanol (10 mL) suspension of PtCl<sub>2</sub> (0.266 g: 1.0 mmol) was treated with 2-(*p*-fluorophenyl)-2-oxazoline (0.454 g: 2.5 mmol), and the resulting mixture was stirred at room temperature for 4 days. The solid that had formed during this time period was filtered off and washed with additional MeOH (10 mL), Et<sub>2</sub>O (2 × 5 mL), and then dried in air to give the *product* in the form of a light-grey coloured powder. Recrystallization (CH<sub>2</sub>Cl<sub>2</sub>/Et<sub>2</sub>O) of this material leads to the isolation of colorless crystals of the title complex. mp 225–227°C (decomp.); *Anal.* calcd (found): C 36.25 (35.34); H 2.70 (2.69); N 4.70 (4.61) %.

A single crystal of the complex of approximate dimensions (0.25 × 0.20 × 0.15 mm) was chosen for a structure determination. A schematic representation of the complex appears in Fig. 1 and the molecular structure is shown in Fig. 2. Crystallographic

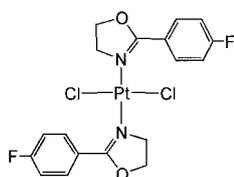


Fig. 1 Schematic representation of the title complex.

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data and the experimental details are given in Table 1. The atomic coordinates and equivalent isotropic displacement parameters for the non-hydrogen atoms appear in Table 2; selected atomic distance and angles are listed in Table 3.

The crystal-structure determination reveals that the central Pt atom is coordinated by two pairs of identical ligands. The coordination sphere contains two transoidally disposed monodentate oxazolines and a pair of chloride atoms. Hence, the title complex is best described as *trans*-bis[2-(*p*-fluorophenyl)-2-oxazoline- $\kappa^1N$ ]platinum(II) chloride. The observed Pt–Cl bond length (~2.30 Å) is typical of that found in other *trans*-[PtCl<sub>2</sub>( $N$ -donor)<sub>2</sub>] complexes, such as [PtCl<sub>2</sub>(8-dqmp)<sub>2</sub>] (2.306(1)Å; CCDC No. 103231).<sup>3</sup> 8-dqmp = diethyl 8-

Table 1 Crystal data and structure refinement for the title complex

CCDC No. 690330
Empirical formula: C <sub>18</sub> H <sub>16</sub> Cl <sub>2</sub> F <sub>2</sub> N <sub>2</sub> O <sub>2</sub> Pt
Formula weight = 596.32
$T = 198\text{ K}$
Crystal system: monoclinic
Space group: $P2_1/n$
$a = 7.7152(7)\text{Å}$
$b = 11.517(1)\text{Å}$
$c = 11.168(1)\text{Å}$
$V = 931.8(1)\text{Å}^3$
$D_{\text{calc}} = 2.125\text{ g/cm}^3$
$\theta$ range for data collection: 2.63 to 27.49°
Reflections collected = 6096
Independent reflections = 2030
Final R indices [ $I > 2\sigma(I)$ ] $R_1 = 0.0149$ , $wR_2 = 0.0365$
$\lambda(\text{Mo } K\alpha) = 0.71073\text{ Å}$
$(\Delta\rho)_{\text{max}} = 0.857\text{ e.Å}^{-3}$
$(\Delta\rho)_{\text{min}} = -0.770\text{ e.Å}^{-3}$
Measurement: Bruker AXS P4/SMART 1000
Structure determination: direct methods (SHELXTL)
Refinement: full-matrix least-squares on $F^2$
Molecular Structure: ORTEP (L. J. Farrugia, <i>J. Appl. Crystallogr.</i> , <b>1997</b> , 30, 565)

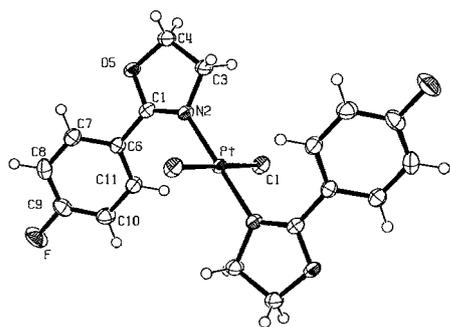


Fig. 2 Molecular structure of the title material with atom labeling.

Table 2 Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ).  $U_{\text{eq}}$  is defined as 1/3 of the trace of the orthogonalized  $U_{ij}$  tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
Pt	0	5000	5000	20(1)
Cl	2974(1)	4492(1)	6201(1)	31(1)
C(1)	1015(3)	7338(2)	4264(2)	22(1)
N(2)	738(3)	6675(2)	5099(2)	22(1)
C(3)	843(4)	7373(2)	6230(2)	27(1)
C(4)	1227(4)	8600(2)	5859(3)	29(1)
O(5)	1263(2)	8468(2)	4564(2)	28(1)
C(6)	1093(3)	7028(2)	3004(2)	23(1)
C(7)	608(3)	7866(2)	2043(2)	27(1)
C(8)	674(4)	7610(3)	851(3)	32(1)
C(9)	1255(4)	6528(3)	660(3)	33(1)
C(10)	1767(4)	5685(3)	1586(3)	32(1)
C(11)	1686(3)	5942(2)	2773(3)	28(1)
F	1371(3)	6271(2)	-502(2)	51(1)

quinolylmethylphosphonate- $\kappa^1N$ . The two symmetry related oxazoline ligands are coordinated *via* the *N*-atoms (Pt-N bond length  $\sim 2.00$  \AA), as expected.<sup>2</sup> This latter interaction displays typical Pt-oxazoline bond lengths, and can be compared with complexes such as the cisoidal isomer of  $[\text{PtCl}_2(2\text{-diethylamino-2-oxazoline})_2]$  (CCDC No. 199088).<sup>4</sup> The aromatic groups appending the coordinated heterocyclic ligands of the title material are tilted out of the plane of the oxazoline rings with a torsion angle ( $\angle\text{O5-C1-C6-C7}$ ) of approximately  $27.5^\circ$ . A similar situation is observed with the structurally related complex *trans*- $[\text{PdCl}_2(2\text{-phenyl-2-oxazoline})_2]$  ( $\sim 28.2^\circ$ : identified as the complementary O1-C9-C1-C6 torsion angle in CCDC No. 138520).<sup>5</sup> Crystallographic symmetry imposes Cl-Pt-Cl and N-Pt-N angles of  $180^\circ$ , and therefore the geometry about the metal centre is close to true square planar in nature. The only deviations from this geometry occur because of the differences between the Pt-N and Pt-Cl bond lengths and the corresponding Cl-Pt-N angles; these latter values are slightly

Table 3 Bond lengths [ $\text{\AA}$ ] and angles [ $^\circ$ ] for the crystal of the title complex

Pt-N(2)	2.004(2)	N(2)-Pt-Cl#1	89.68(6)
Pt-Cl	2.2996(7)	N(2)-Pt-Cl	90.32(6)
C(1)-N(2)	1.279(3)	N(2)-C(1)-O(5)	116.2(2)
C(1)-O(5)	1.341(3)	N(2)-C(1)-C(6)	128.6(2)
C(1)-C(6)	1.473(3)	O(5)-C(1)-C(6)	115.1(2)
N(2)-C(3)	1.476(3)	C(1)-N(2)-C(3)	109.0(2)
C(3)-C(4)	1.530(4)	C(1)-N(2)-Pt	130.56(17)
C(4)-O(5)	1.464(3)	C(3)-N(2)-Pt	120.15(15)
C(6)-C(11)	1.387(4)	N(2)-C(3)-C(4)	103.25(19)
C(6)-C(7)	1.395(4)	O(5)-C(4)-C(3)	104.1(2)
C(7)-C(8)	1.382(4)	C(1)-O(5)-C(4)	107.33(19)
C(9)-F	1.363(3)	C(11)-C(6)-C(7)	120.0(2)
C(9)-C(10)	1.373(4)	C(11)-C(6)-C(1)	121.4(2)
C(10)-C(11)	1.381(4)	C(7)-C(6)-C(1)	118.6(2)
		C(8)-C(7)-C(6)	120.3(3)
		C(9)-C(8)-C(7)	117.9(3)
		F-C(9)-C(8)	118.6(3)
		F-C(9)-C(10)	117.9(3)
		C(8)-C(9)-C(10)	123.6(3)
		C(9)-C(10)-C(11)	118.4(3)
		C(10)-C(11)-C(6)	119.9(3)

Symmetry transformations used to generate equivalent atoms:  
#1  $-x, -y+1, -z+1$

offset from the idealised right angle.

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