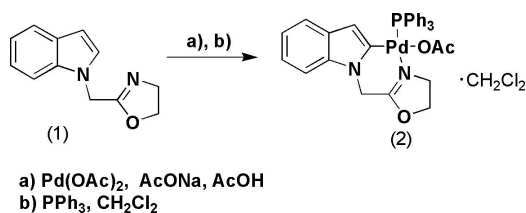


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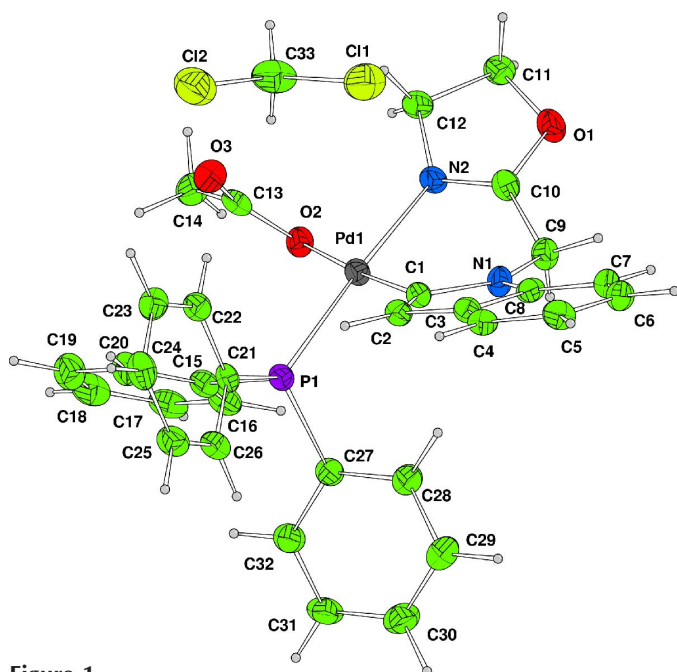
## Key indicators

Single-crystal X-ray study  
*T* = 150 K  
Mean  $\sigma$ (C–C) = 0.005 Å  
*R* factor = 0.036  
*wR* factor = 0.040  
Data-to-parameter ratio = 12.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Acetato[1-(oxazolin-2-ylmethyl- $\kappa$ N)indol-2-yl]-  
(triphenylphosphine)palladium(II) dichloromethane  
solvateThe title compound, [Pd(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)(C<sub>18</sub>H<sub>15</sub>P)(C<sub>12</sub>H<sub>11</sub>N<sub>2</sub>O)]·CH<sub>2</sub>Cl<sub>2</sub>, crystallizes readily from a mixture of dichloromethane and light petroleum. The structure is one of few examples of palladacycles that incorporate the *N*-substituent in five-membered-ring heterocycles [Nonoyama & Nakajima (1998). *Polyhedron*, **18**, 533–543].Received 2 December 2004  
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## Comment

Recently, cyclopalladated oxazoline-based complexes have attracted attention due to their catalytic efficiency and the high potential of the oxazolinyl group as an inducer of chirality (Richards & Stark, 1997; Denmark *et al.*, 1997). A few complexes based on *ortho*-palladation of an aryloxazoline in the arene, ferrocene and [2.2]*paracyclophane* series have been crystallographically characterized (Balavoine *et al.*, 1990; Bölm *et al.*, 2002; Gorunova *et al.*, 2004; Kirsch *et al.*, 2004; Smoliakova *et al.*, 2000). We report here the synthesis and structure of a six-membered indole-fused *ortho*-palladacycle, (2), which incorporates an *N*-methylenioxazoline as a *C,N*-bidentate ligand. We found that the application of an equimolar amount of the weak electrophilic reagent Li<sub>2</sub>PdCl<sub>4</sub> in the reaction with indole-derived oxazoline, (1), in EtOH in the presence of AcONa as a base (Smoliakova *et al.*, 2000) led to the formation of a bis-oxazoline coordination complex with no traces of the desired cyclopalladated compound. On the other hand, one of the most efficient cyclopalladation methods is the use of highly electrophilic Pd(OAc)<sub>2</sub> in AcOH. By applying this method intramolecular C–H bond activation has been achieved and the desired cyclopalladated compound, (2), was obtained in good yield. This is the first crystallographically characterized oxazoline-derived palladacycle with a six-membered chelate ring, but activation of a benzylic *Csp*<sup>3</sup> atom to form a six-ring palladacycle has been reported as an alternative and controllable reaction path in the *para*-cyclophane series (Bölm *et al.*, 2002).

The coordination geometry of the Pd atom closely approaches planarity. The mean plane through the reported atomic positions of the Pd/C/N/O/P atoms intersects those of the fragments C1–C9/N1, N2/O1/C10–C12 and O2/O3/C13/C14 at 39.80 (10), 36.1 (2) and 84.43 (16)°, respectively.



**Figure 1**  
The molecular structure of (2). Displacement ellipsoids are drawn at the 50% probability level.

The solvent interacts with neighbouring molecules of the Pd complex. One of these interactions is apparently through a C—H···O hydrogen bond to the carbonyl O atom [C33···O3 = 3.129 (5) Å], while there is a second short contact between the oxazolonyl O atom and one of the Cl atoms of a second molecule of solvent [O1···Cl1<sup>i</sup> = 3.030 (3) Å; symmetry code: (i)  $-x, -y, 1 - z$ ].

## Experimental

A mixture of Pd(OAc)<sub>2</sub> (52 mg, 0.23 mmol) and AcONa (32 mg, 0.23 mmol) was partially dissolved in acetic acid (1 ml). Oxazoline, (1) (50 mg, 0.25 mmol), was dissolved in AcOH (1 ml). The two solutions were combined and stirred at room temperature overnight. The reaction mixture was then stirred at 323 K for 1 h and 24 h at room temperature. The mixture was filtered through Celite, the solvent removed and the yellow solid was dried *in vacuo*. Degassed CH<sub>2</sub>Cl<sub>2</sub> (5 ml) and PPh<sub>3</sub> (66 mg, 0.25 mmol) were then added to the solid and the mixture was stirred at room temperature for 2 h under an argon atmosphere. Addition of light petroleum ether to the solution resulted in precipitation of a yellow solid. Recrystallization of the crude product from dichloromethane and hexane gave the yellow product, (2), with an overall yield of 70%. Full spectroscopic and physical characterization will be reported elsewhere.

### Crystal data

[Pd(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)(C<sub>18</sub>H<sub>15</sub>P)-  
(C<sub>12</sub>H<sub>11</sub>N<sub>2</sub>O)]·CH<sub>2</sub>Cl<sub>2</sub>  
*M<sub>r</sub>* = 711.90  
Triclinic, *P* $\bar{1}$   
*a* = 10.3125 (2) Å  
*b* = 10.9019 (2) Å  
*c* = 16.4409 (5) Å  
 $\alpha$  = 71.7446 (9)°  
 $\beta$  = 75.0748 (9)°  
 $\gamma$  = 62.0330 (10)°  
*V* = 1536.81 (6) Å<sup>3</sup>

*Z* = 2  
*D<sub>x</sub>* = 1.538 Mg m<sup>-3</sup>  
Mo *K* $\alpha$  radiation  
Cell parameters from 20892  
reflections  
 $\theta$  = 5–28°  
 $\mu$  = 0.87 mm<sup>-1</sup>  
*T* = 150 K  
Plate, pale yellow  
0.24 × 0.20 × 0.05 mm

### Data collection

Nonius KappaCCD diffractometer  
 $\omega$  scans  
Absorption correction: multi-scan  
DENZO/SCALEPACK  
(Otwinowski & Minor, 1997)  
*T*<sub>min</sub> = 0.81, *T*<sub>max</sub> = 0.96  
20892 measured reflections

6983 independent reflections  
4899 reflections with *I* > 3 $\sigma$ (*I*)  
*R*<sub>int</sub> = 0.053  
 $\theta$ <sub>max</sub> = 27.5°  
*h* = -13 → 13  
*k* = -14 → 14  
*l* = -21 → 21

### Refinement

Refinement on *F*  
*R* = 0.036  
*wR* = 0.040  
*S* = 1.10  
4899 reflections  
379 parameters

H-atom parameters constrained  
Weighting scheme: see text  
( $\Delta/\sigma$ )<sub>max</sub> = 0.001  
 $\Delta\rho$ <sub>max</sub> = 0.61 e Å<sup>-3</sup>  
 $\Delta\rho$ <sub>min</sub> = -0.49 e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

Pd1—C1	1.995 (3)	Pd1—O2	2.075 (2)
Pd1—N2	2.079 (3)	Pd1—P1	2.2425 (9)
C1—Pd1—N2	87.95 (12)	Pd1—C1—C2	133.7 (2)
C1—Pd1—O2	175.71 (11)	Pd1—C1—N1	118.6 (2)
N2—Pd1—O2	87.79 (10)	Pd1—P1—C15	112.13 (11)
C1—Pd1—P1	92.34 (9)	Pd1—P1—C21	114.12 (11)
N2—Pd1—P1	175.51 (9)	Pd1—P1—C27	116.38 (11)
O2—Pd1—P1	91.87 (7)		
C33···O3	3.129 (5)	O1···Cl1 <sup>i</sup>	3.030 (3)

Symmetry code: (i)  $-x, -y, 1 - z$ .

The weighting scheme was  $w = [1 - \{||F_o| - |F_c||/6\sigma(F_o)\}]^2 / [0.391T_0(x) + 0.126T_1(x) + 0.171T_2(x)]$ , using a second-order Chebyshev polynomial, with  $x = F_c/F_{max}$  (Watkin, 1994; Prince, 1982).

All H atoms were placed in geometrically calculated positions after each refinement cycle, with *X*—H = 1.0 Å; *U*<sub>iso</sub>(H) values were set equal to 1.2*U*<sub>eq</sub> of the connected atom.

Data collection: COLLECT (Nonius, 1998); cell refinement: DENZO/SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO/SCALEPACK; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: CRYSTALS (Betteridge *et al.*, 2003); molecular graphics: CAMERON (Watkin *et al.*, 1996); software used to prepare material for publication: CRYSTALS.

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