



University of  
**Salford**  
MANCHESTER

# trans-Bis(tert-butylamine)dichloropalladium(II)

Boag, NM and Clapham, S

<http://dx.doi.org/10.1107/S1600536805030047>

<b>Title</b>	trans-Bis(tert-butylamine)dichloropalladium(II)
<b>Authors</b>	Boag, NM and Clapham, S
<b>Type</b>	Article
<b>URL</b>	This version is available at: <a href="http://usir.salford.ac.uk/id/eprint/408/">http://usir.salford.ac.uk/id/eprint/408/</a>
<b>Published Date</b>	2005

USIR is a digital collection of the research output of the University of Salford. Where copyright permits, full text material held in the repository is made freely available online and can be read, downloaded and copied for non-commercial private study or research purposes. Please check the manuscript for any further copyright restrictions.

For more information, including our policy and submission procedure, please contact the Repository Team at: [usir@salford.ac.uk](mailto:usir@salford.ac.uk).

**Neil M. Boag\* and  
 Sarah Clapham**

Chemistry and Nanotechnology, Institute for  
 Materials Research, Cockcroft Building,  
 University of Salford, Salford, M5 4WT, England

Correspondence e-mail:  
 N.M.Boag@salford.ac.uk

**Key indicators**

Single-crystal X-ray study  
 T = 223 K  
 Mean  $\sigma(\text{C}-\text{C}) = 0.008 \text{ \AA}$   
 R factor = 0.038  
 wR factor = 0.098  
 Data-to-parameter ratio = 22.8

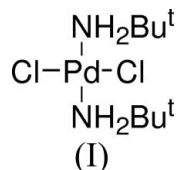
For details of how these key indicators were  
 automatically derived from the article, see  
<http://journals.iucr.org/e>.

***trans*-Bis(*tert*-butylamine)dichloropalladium(II)**

The asymmetric unit of the title complex, *trans*-[PdCl<sub>2</sub>(NH<sub>2</sub><sup>t</sup>Bu)<sub>2</sub>], consists of two independent square-planar molecules, linked together in a hydrogen-bonding network, with the resultant alignment of the *tert*-butyl groups defining a two-dimensional layered structure approximately parallel to (001).

**Comment**

We have noted that the chemistry of *tert*-butylamine derivatives of palladium frequently differs from other primary amine complexes due to the steric bulk of the *tert*-butyl group. The availability of crystals of the title complex, (I), allowed comparison with other bis(primary amine)dichloro complexes of palladium to determine the structural consequences of steric bulk.



Complex (I) exists as two independent square-planar molecules in the asymmetric unit. The orientation of the *tert*-butylamine groups is such that both molecules are pseudo-centrosymmetric. Analysis of the 14 previously reported bis(primary amine)dichloropalladium(II) structures (Fletcher *et al.*, 1996) gives averages of 2.300 (8) Å and 2.047 (9) Å for the Pd–Cl and the Pd–N bonds, respectively, with a mean deviation of the N–Pd–Cl angles of *ca* 1.4° from the ideal 90°. The Pd–Cl and Pd–N bond lengths in (I) range from 2.3015 (11) to 2.3072 (12) and 2.046 (4) to 2.058 (4) Å, respectively; this indicates that, in this complex, the bulky *tert*-butyl group has no obvious structural consequence, although the average N–Pd–Cl angle in complex (I) does show a significantly smaller deviation from the 90° required by ideal square-planar geometry [0.46° (molecule 1), 0.37° (molecule 2)]. The molecules are linked together in a hydrogen-bonding network, resulting in the formation of a two-dimensional layered structure, externally defined by the *tert*-butyl groups and approximately parallel to (001).

**Experimental**

Complex (I) crystallized from a dichloromethane/hexane solution of *trans*-[Pd( $\eta^1$ -C<sub>5</sub>H<sub>5</sub>)(NH<sub>2</sub><sup>t</sup>Bu)<sub>2</sub>Cl] and [Pd( $\eta^5$ -C<sub>5</sub>H<sub>5</sub>)(NH<sub>2</sub><sup>t</sup>Bu)Cl] and was spectroscopically identical to the material synthesized according to the literature method (Nakayama *et al.*, 1984).

Received 23 August 2005  
 Accepted 20 September 2005  
 Online 30 September 2005

## Crystal data

[PdCl<sub>2</sub>(C<sub>4</sub>H<sub>11</sub>N)<sub>2</sub>]  
*M<sub>r</sub>* = 323.58  
 Triclinic, *P* $\bar{1}$   
*a* = 6.2357 (10) Å  
*b* = 10.6500 (11) Å  
*c* = 20.472 (2) Å  
 $\alpha$  = 94.641 (8)°  
 $\beta$  = 90.978 (13)°  
 $\gamma$  = 93.824 (11)°  
*V* = 1351.7 (3) Å<sup>3</sup>

*Z* = 4  
*D<sub>x</sub>* = 1.590 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 34 reflections  
 $\theta$  = 5.1–12.5°  
 $\mu$  = 1.73 mm<sup>-1</sup>  
*T* = 223 (2) K  
 Block, orange  
 0.6 × 0.3 × 0.3 mm

## Data collection

Siemens P4 diffractometer  
 Profile fitting of  $\omega/2\theta$  scans  
 Absorption correction:  $\psi$  scan  
 (XSCANS; Siemens, 1996).  
*T<sub>min</sub>* = 0.537, *T<sub>max</sub>* = 0.594  
 7873 measured reflections  
 6177 independent reflections  
 5913 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.019  
 $\theta_{\max}$  = 27.5°  
*h* = -8 → 1  
*k* = -13 → 13  
*l* = -26 → 26  
 3 standard reflections  
 every 97 reflections  
 intensity decay: 4%

## Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.038  
*wR*(*F*<sup>2</sup>) = 0.098  
*S* = 1.23  
 6177 reflections  
 271 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0118P)^2 + 6.285P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 1.01 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -1.24 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Pd1—N12	2.046 (4)	Pd2—N21	2.057 (4)
Pd1—N11	2.050 (4)	Pd2—N22	2.058 (4)
Pd1—Cl11	2.3015 (11)	Pd2—Cl22	2.3051 (12)
Pd1—Cl12	2.3030 (11)	Pd2—Cl21	2.3072 (12)
N12—Pd1—N11	179.27 (16)	N21—Pd2—N22	179.06 (16)
N12—Pd1—Cl11	90.17 (12)	N21—Pd2—Cl22	89.93 (12)
N11—Pd1—Cl11	89.35 (12)	N22—Pd2—Cl22	90.71 (12)
N12—Pd1—Cl12	89.74 (12)	N21—Pd2—Cl21	90.04 (12)
N11—Pd1—Cl12	90.74 (12)	N22—Pd2—Cl21	89.32 (12)
Cl11—Pd1—Cl12	179.34 (5)	Cl22—Pd2—Cl21	179.26 (6)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N11—H112...Cl11 <sup>i</sup>	0.81 (6)	2.62 (6)	3.408 (4)	163 (5)
N12—H121...Cl21 <sup>ii</sup>	0.82 (6)	2.75 (6)	3.416 (4)	140 (5)
N12—H122...Cl12 <sup>iii</sup>	0.84 (6)	2.60 (6)	3.423 (4)	165 (5)
N21—H211...Cl11	0.79 (6)	2.59 (6)	3.327 (4)	157 (5)
N21—H212...Cl22 <sup>iii</sup>	0.84 (6)	2.76 (6)	3.502 (4)	148 (5)
N22—H221...Cl21 <sup>i</sup>	0.80 (6)	2.71 (6)	3.481 (4)	164 (5)
N22—H222...Cl12 <sup>iv</sup>	0.92 (6)	2.52 (6)	3.347 (4)	149 (4)

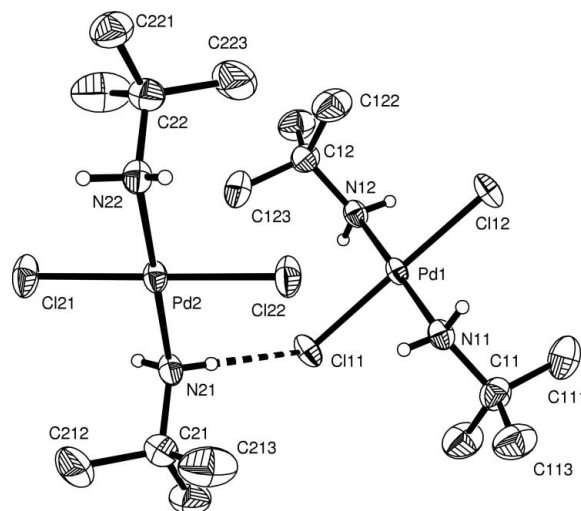
Symmetry codes: (i) *x* + 1, *y*, *z*; (ii) *x*, *y* - 1, *z*; (iii) *x* - 1, *y*, *z*; (iv) *x*, *y* + 1, *z*.

Figure 1

A view of the two independent molecules in (I). Displacement ellipsoids are drawn at the 50% probability level. *tert*-Butyl H atoms have been omitted. The dashed line indicates a hydrogen bond.

Methyl-H atoms were placed in calculated positions and subsequently constrained to an ideal geometry, with C—H distances of 0.97 Å and *U<sub>iso</sub>*(H) = 1.5*U<sub>eq</sub>*(C), with each group allowed to rotate freely about its C—C bond. The positions of the amine H atoms were identified from a difference Fourier map and allowed to refine freely with fixed isotropic displacement parameters; N—H = 0.79 (6)–0.92 (6) Å. The highest peak is located 1.21 Å from atom Cl21 and the deepest hole 1.47 Å from atom Cl12.

Data collection: XSCANS (Siemens, 1996); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SHELXTL-Plus (Siemens, 1995); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL-Plus; software used to prepare material for publication: SHELXL97 (Sheldrick, 1997).

We thank the EPSRC for a studentship and the SCI for a Messel Scholarship (SC). We acknowledge the use of the EPSRC's Chemical Database Service at Daresbury.

## References

- Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.  
 Nakayama, K., Komorita, T. & Shimura, Y. (1984). *Bull. Chem. Soc. Jpn.* **57**, 1336–1347.  
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.  
 Siemens (1995). SHELXTL-Plus. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Siemens (1996). XSCANS. Version 2.20. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.