The asymmetric unit of the title complex, trans-
[PdCl₂(NH₂Bu)₂], 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 deriv-
atives 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)dichloropalladium
compounds to determine the structural consequences of
steric bulk.

Complex (I) exists as two independent square-planar mol-
ecules 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(η⁵-C₅H₅)(NH₂Bu)Cl] and [Pd(η⁵-C₅H₅)(NH₂Bu)Cl] and
was spectroscopically identical to the material synthesized according
to the literature method (Nakayama et al., 1984).
Crystal data

\[ [\text{PdCl}_2(\text{C}_4\text{H}_11\text{N})_2] \]

\[ M_r = 323.58 \]

Triclinic, \( P \)

\[ a = 6.2557 (10) \AA \]

\[ b = 10.6500 (11) \AA \]

\[ c = 20.472 (2) \AA \]

\[ \alpha = 94.641 (8) \]

\[ \beta = 90.978 (13) \]

\[ \gamma = 93.824 (11) \]

\[ V = 1351.7 (3) \AA^3 \]

\[ Z = 4 \]

\[ D_r = 1.590 \text{ Mg m}^{-3} \]

Mo \( K\alpha \) radiation

Cell parameters from 34 reflections

\[ \theta = 5.1-12.5^\circ \]

\[ \mu = 1.73 \text{ mm}^{-1} \]

\[ T = 223 (2) \text{ K} \]

Block, orange

\[ 0.6 \times 0.3 \times 0.3 \text{ mm} \]

Data collection

Siemens P4 diffractometer

Profile fitting of \( \psi \) scan

<table>
<thead>
<tr>
<th>Data collection</th>
<th>( \psi ) scan</th>
</tr>
</thead>
<tbody>
<tr>
<td>(XSCANS; Siemens, 1996),</td>
<td>T_{max} = 0.537, T_{min} = 0.594</td>
</tr>
<tr>
<td>7873 measured reflections</td>
<td>6177 independent reflections</td>
</tr>
<tr>
<td>5913 reflections with ( I &gt; 2\sigma(I) )</td>
<td>( R_{int} = 0.019 )</td>
</tr>
</tbody>
</table>

Refinement

Refinement on \( F^2 \)

\[ R(F^2) = 0.038 \]

\[ wR(F^2) = 0.098 \]

\[ S = 1.23 \]

6177 reflections

271 parameters

H atoms treated by a mixture of independent and constrained refinement

Refinement on \( F^2 \)

\[ R(F^2) = 0.038 \]

\[ wR(F^2) = 0.098 \]

\[ S = 1.23 \]

6177 reflections

271 parameters

H atoms treated by a mixture of independent and constrained refinement

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 \( U_{iso}(H) = 1.5U_{eq}(C) \), 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 C12 and the deepest hole 1.47 Å from atom C11.

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).

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References


