

Discussion. The atomic nomenclature and the configuration of the molecule are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1. Final atomic coordinates for non-H atoms are given in Table 1;* selected interatomic distances, angles and short intramolecular contacts are reported in Tables 2 and 3.

The U–O bond lengths within the UO_2 unit compare with the mean value of 1.764 Å for fivefold equatorial coordination (*Gmelin's Handbook of Inorganic Chemistry*, 1983). The equatorial U–O(THF) bond distances at 2.45 (2)–2.47 (1) Å are in the range observed for uranyl and uranium complexes. In $[\text{UO}_2\text{Cl}(\text{C}_6\text{H}_5\text{CONOHC}_6\text{H}_5)] \cdot 2\text{C}_4\text{H}_8\text{O}$ (Smith & Raymond, 1979) the corresponding distances were 2.42 (3) and 2.47 (3) Å; in $[\text{UO}_2(\text{NO}_3)_2] \cdot 2\text{C}_4\text{H}_8\text{O}$ (Reynolds, Zalkin & Templeton, 1977) 2.42 (2) Å; in $[\text{U}(\text{BH}_4)_4] \cdot 2\text{C}_4\text{H}_8\text{O}$ (Rietz, Edelstein, Ruben, Templeton & Zalkin, 1978) 2.47 (1) Å; and in $[\text{U}(\text{C}_9\text{H}_7)]\text{Br}_3 \cdot 2\text{C}_4\text{H}_8\text{O}$ (Rebizant, Spirlet & Goffart, 1985) 2.466 (8) Å.

The lengthening of the U–Br bond distances [2.845 (3), 2.856 (3) Å] in comparison with the values observed in $[\text{NH}_4]_2[\text{UO}_2\text{Br}_4] \cdot 2\text{H}_2\text{O}$ (Van den Bossche, Spirlet, Rebizant & Goffart, 1987), 2.812 (1) and 2.815 (1) Å, and in $[\text{U}(\text{C}_9\text{H}_7)]\text{Br}_3 \cdot 2\text{C}_4\text{H}_8\text{O}$ (Rebizant *et al.*, 1985), 2.759 (1), 2.775 (2) and 2.791 (1) Å, is a

*Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43870 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

result of steric interference with the three equatorial THF rings. Table 3 shows evidence of interligand compressions which have resulted in distortions of the molecule from the ideal geometry of a precisely planar equatorial set of ligands. Indeed the coordinating ligand atoms are substantially displaced out of the mean equatorial plane (by up to 0.3 Å). A significant bending of 15.6 and 15.8° respectively of the two neighbouring THF ligands towards Br is also observed. As expected the outermost C atoms of the THF molecules exhibit large thermal motions, principally perpendicular to the rings, and some C–C bond lengths are therefore foreshortened. The stacking in the crystal is dictated by van der Waals interactions.

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Structure of Dichlorobis(2,2-dimethoxy-2-phenylethylamine)palladium(II)

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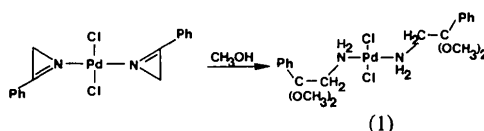
Abstract. $[\text{PdCl}_2(\text{C}_{10}\text{H}_{15}\text{NO}_2)_2]$, $M_r = 539.4$, triclinic, $P\bar{1}$, $a = 10.391$ (2), $b = 10.457$ (1), $c = 5.7833$ (4) Å, $\alpha = 105.875$ (7), $\beta = 103.875$ (8), $\gamma = 87.21$ (1)° (standardized parameters are: $a = 5.7833$, $b = 10.391$, $c = 10.457$ Å, $\alpha = 87.21$, $\beta = 74.125$, $\gamma = 76.125^\circ$), $U = 586.7$ Å³, $Z = 1$, $D_x = 1.53$ Mg m^{−3}, Mo $K\alpha$, λ

$= 0.71069$ Å, $\mu = 1.03$ mm^{−1}, $F(000) = 276$, $T = 291$ K, final $R = 0.025$ for 4187 unique observed reflections. The Pd atom exhibits a square-planar coordination geometry with the amine groups occupying *trans* positions. Bond lengths and angles fall within normal ranges.

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Introduction. Hassner, Bunnell & Haltiwanger (1978) reported that variously substituted 2*H*-azirines react with dichlorobis(benzonitrile)palladium(II) in benzene to give stable metal-coordinated 2*H*-azirine complexes. Bis(3-phenyl-2*H*-azirine)palladium dichloride undergoes a slow opening of the C–N single bond in benzene to form bis(η^2 -3-chloro-1-azaallyl)palladium dichloride (Faria dos Santos Filho & Schuchardt, 1984). In methanol, however, the C=N double bond of the palladium complex opens and the title compound (1) is formed (Faria dos Santos Filho, 1982). This study was undertaken to determine the structure of (1).



Experimental. Compound (1) was prepared by refluxing 2 mmol of bis(3-phenyl-2*H*-azirine)palladium dichloride in 20 ml of methanol for 3 h. After evaporation of the methanol, (1) was recrystallized from chloroform/petroleum ether and obtained in 95% yield (m.p. 454–455 K). Crystals of (1) were grown as yellow prisms from chloroform at room temperature. The crystal chosen for data collection was 0.07 × 0.10 × 0.05 mm. The unit-cell parameters were obtained by a least-squares fit to the θ values of 75 automatically centred reflections ($9.3 \leq \theta \leq 27.4^\circ$). Intensity data were measured within the range $2.0 \leq \theta \leq 32.9^\circ$ on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ X-radiation by a θ – 2θ scan technique in 48 steps, where the time spent measuring the background was half that taken to measure the peak. The intensities of three standard reflections, remeasured every 100 reflections, showed no significant variation during data collection. Data were corrected for Lorentz and polarization effects, but not for absorption. Of a total of 4366 ($\pm h, \pm k, \pm l$) measured intensities ($-15 \leq h \leq 15$, $-15 \leq k \leq 15$, $0 \leq l \leq 8$), 4187 satisfied the criterion $I \geq 2.0\sigma(I)$, and only these were used in the solution and refinement of the structure. Computer programs used in this investigation include modified versions of *DATAP* data reduction program (Coppens, Leiserowitz & Rabinovich, 1965), *TRACER* (Jacobson & Lawton, 1965) for cell reduction, *SHELX* (Sheldrick, 1974) for crystal structure solution and refinement, a modified version of the *ORFLS* least-squares program for blocked-diagonal refinement (Busing, Martin & Levy, 1962), *DAESD* program for distances and angles (Davis & Harris, 1970), *XANADU* molecular geometry program (Roberts & Sheldrick, 1976), and *ORTEP* thermal-ellipsoid plotting program (Johnson, 1976).

The structure was solved by the heavy-atom method. Refinement was by blocked least-squares methods,

where the function minimized was $\sum w(\Delta F)^2$ with $w = 1/\sigma(F)^2$ and $\Delta F = |F_o| - |F_c|$. The x , y and z coordinates for the Pd atom were fixed at 0.0 during refinement. Refinement of the non-H-atom positional and isotropic thermal parameters resulted in $R = 0.089$ ($wR = 0.123$). The positions of the H atoms were located on a difference Fourier map and included in the refinement with isotropic thermal parameters. Refinement converged at $R = 0.025$ ($wR = 0.038$) for 193 variables and 4187 reflections, and the value of S was 1.23, indicating a slight underestimation of the error of

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (isotropic thermal parameters for H)

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} (Å ²)
Pd	0.0000	0.0000	0.0000	0.032
Cl	0.1616 (1)	0.0523 (1)	−0.1715 (1)	0.049
O(1)	−0.1535 (1)	0.3011 (1)	−0.4732 (2)	0.043
O(2)	−0.1934 (1)	0.4579 (1)	−0.1225 (3)	0.049
N	−0.1345 (1)	0.1015 (1)	−0.2017 (3)	0.038
C(1)	−0.1328 (2)	0.2461 (2)	−0.0820 (3)	0.038
C(2)	−0.2082 (2)	0.3231 (2)	−0.2644 (3)	0.036
C(3)	−0.3531 (2)	0.2773 (2)	−0.3578 (3)	0.039
C(4)	−0.4363 (2)	0.3116 (3)	−0.1945 (4)	0.060
C(5)	−0.5680 (3)	0.2680 (3)	−0.2710 (6)	0.077
C(6)	−0.6173 (2)	0.1890 (3)	−0.5098 (5)	0.065
C(7)	−0.5354 (2)	0.1546 (3)	−0.6709 (5)	0.060
C(8)	−0.4035 (2)	0.1984 (2)	−0.5962 (4)	0.048
C(9)	−0.0150 (2)	0.3357 (3)	−0.4188 (5)	0.055
C(10)	−0.2545 (3)	0.5513 (2)	−0.2557 (6)	0.063
H(N1)	−0.214 (3)	0.074 (3)	−0.226 (5)	0.065
H(N2)	−0.121 (2)	0.086 (2)	−0.354 (4)	0.036
H(1A)	−0.033 (3)	0.273 (3)	0.001 (5)	0.059
H(1B)	−0.171 (2)	0.260 (2)	0.066 (4)	0.027
H(4)	−0.405 (3)	0.355 (3)	−0.017 (6)	0.073
H(5)	−0.614 (3)	0.263 (3)	−0.136 (6)	0.094
H(6)	−0.713 (3)	0.148 (3)	−0.588 (5)	0.059
H(7)	−0.564 (3)	0.103 (3)	−0.847 (6)	0.076
H(8)	−0.342 (2)	0.173 (2)	−0.717 (4)	0.038
H(9A)	−0.001 (3)	0.418 (3)	−0.305 (5)	0.066
H(9B)	−0.007 (9)	0.333 (9)	−0.522 (9)	0.169
H(9C)	0.034 (7)	0.273 (7)	−0.538 (9)	0.126
H(10A)	−0.234 (3)	0.520 (3)	−0.398 (7)	0.081
H(10B)	−0.356 (3)	0.542 (3)	−0.304 (5)	0.065
H(10C)	−0.229 (2)	0.626 (3)	−0.169 (5)	0.049

Table 2. Bond angles (°) and torsion angles (°) e.s.d.'s in parentheses

Cl–Pd–N	89.5 (1)	Cl*–Pd–N	90.5 (1)
Pd–N–C(1)	113.1 (1)	N–C(1)–C(2)	111.4 (1)
C(1)–C(2)–C(3)	110.4 (1)	C(1)–C(2)–O(1)	111.3 (1)
C(1)–C(2)–O(2)	103.8 (1)	O(1)–C(2)–C(3)	107.4 (1)
O(2)–C(2)–C(3)	112.4 (1)	O(1)–C(2)–O(2)	111.7 (1)
C(2)–O(1)–C(9)	115.0 (1)	C(2)–O(2)–C(10)	114.4 (2)
C(2)–C(3)–C(4)	118.6 (2)	C(2)–C(3)–C(8)	122.3 (2)
C(4)–C(3)–C(8)	119.0 (2)		
Cl–Pd–N–C(1)	−84.2 (1)	Pd–N–C(1)–C(2)	165.8 (1)
N–C(1)–C(2)–O(1)	−58.2 (1)	N–C(1)–C(2)–O(2)	−178.5 (1)
N–C(1)–C(2)–C(3)	60.9 (1)	C(1)–C(2)–C(3)–C(4)	70.8 (1)
C(1)–C(2)–C(3)–C(8)	−106.6 (1)	C(1)–C(2)–O(1)–C(9)	−57.5 (1)
C(1)–C(2)–O(2)–C(10)	179.0 (1)		

* Refers to the symmetry operation: $-x, -y, -z$.

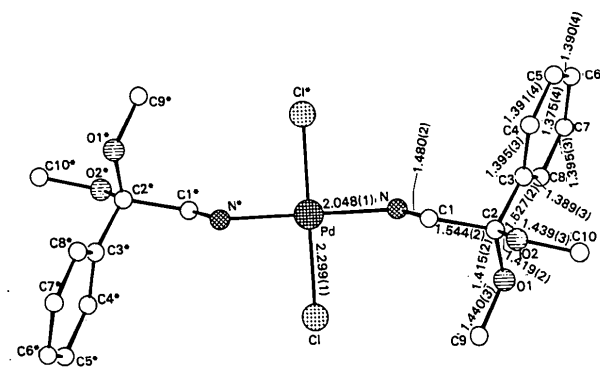


Fig. 1. The molecular structure of (1).

an observation of unit weight. In the final refinement cycle the maximum shift to e.s.d. ratio was 0.6. A correction for the effects of anomalous dispersion for Pd ($f' -1.177, f'' 1.007$) and Cl ($f' 0.132, f'' 0.159$) was included in the structure-factor calculations. Atomic scattering curves were taken from *International Tables for X-ray Crystallography* (1974). In the final difference Fourier synthesis, peaks of $\pm 1.5 \text{ e } \text{\AA}^{-3}$ were observed in the vicinity of the Pd atom with much lower values elsewhere.*

Discussion. The results of the X-ray analysis are summarized in Tables 1 and 2, which give the final atomic coordinates and bond angles, and Fig. 1, which shows the molecular structure of (1) and the bond distances. The molecule crystallizes with one molecule

* Lists of structure amplitudes and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43873 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

in the centrosymmetric space group $P\bar{1}$. The metal atom lies on an exact centre of symmetry and has square-planar coordination geometry with the two amine groups occupying *trans* positions. The Pd–N bond length is comparable with those found in other complexes containing an amine bonded to a Pd atom [2.018 (8) Å, Drew, Riedl & Rodgers (1972); 2.054 (9), 2.055 (9), 1.985 (8) Å, Ferguson & Parvez, (1979)]. Bond distances and angles in the remainder of the molecule are as expected.

There are no intermolecular contacts between non-H atoms less than 3.2 Å.

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$\Delta^{1(2)}$ -Dehydro-2-methylsparteinium Diperchlorate Monohydrate

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Abstract. $\text{C}_{16}\text{H}_{26}\text{N}_2^{2+} \cdot 2\text{ClO}_4^- \cdot \text{H}_2\text{O}$, $M_r = 463.3$, orthorhombic, $P2_12_12_1$, $a = 13.185$ (1), $b = 17.650$ (2), $c = 9.029$ (1) Å, $V = 2101.2$ (5) Å³, $Z = 4$, $D_m = 1.46$ (1), $D_x = 1.47$ g cm⁻³, $\lambda(\text{Cu K}\alpha) = 1.54178$ Å,

$\mu(\text{Cu } K\alpha) = 31.0 \text{ cm}^{-1}$, $F(000) = 976$, $T = 298 \text{ K}$, $R = 0.072$ for 1592 unique observed reflections. The quinolizidinium and quinolizidine moieties have quasi-*trans* and *trans* configurations, respectively.