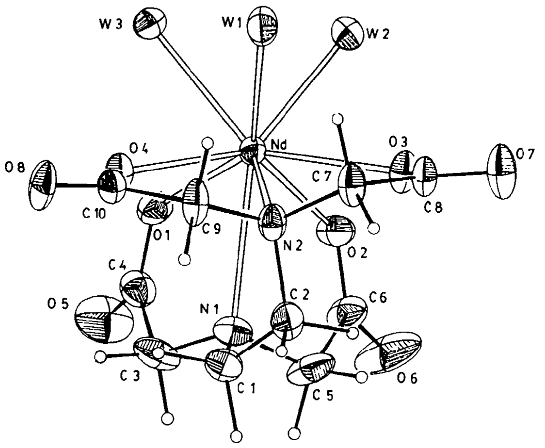

Fig. 1. *ORTEP* drawing of the complex anion with the atom-numbering scheme.

Crystal water oxygen atoms *W*4 and *W*8 were not found to have disordered positions as in the Sm salt solved by neutron diffraction (Engel *et al.*, 1984) nor does *W*8 have such a high temperature factor as in the other structures. Instead, disordered positions of *W*7 were found (*W*7*A* and *W*7*B*). The same hydrogen-bonding network as in the neutron diffraction study of the Sm complex was found for the Nd salt, except for the bond involving *W*7*B*. The differences in the positions of the crystal water molecules are influenced by the larger K^+ ion, which is surrounded by seven O atoms at distances ranging from 2·57 (3) or 2·66 (3) to 2·895 (4) Å. Bond distances and angles in the edta moiety are normal.

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References

Djordjević, C. & Vuletić, N. (1980). *Inorg. Chem.* **19**, 3049–3053.

Engel, D. W., Takusagawa, F. & Koetzle, T. F. (1984). *Acta Cryst.* C**40**, 1687–1693.

Hoard, J. L., Lee, B. & Lind, M. D. (1965). *J. Am. Chem. Soc.* **87**, 1612–1613.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Lee, B. (1967). *Diss. Abstr. B*, **28**, 84.

Nassimbeni, L. R., Wright, M. R. W., van Niekerk, J. C. & McCallum, P. A. (1979). *Acta Cryst.* B**35**, 1341–1345.

Rogers, D. (1981). *Acta Cryst.* A**37**, 734–741.

Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Templeton, L. K., Templeton, D. H. & Zalkin, A. (1985). *Acta Cryst.* C**41**, 355–358.

Templeton, L. K., Templeton, D. H., Zalkin, A. & Ruben, H. W. (1982). *Acta Cryst.* B**38**, 2155–2159.

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Structure of *trans*-Dichloro-*cis*-dichlorobis(1-propylimidazole)platinum(IV)

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Abstract. $[PtCl_4(C_6H_{10}N_2)_2]$, $M_r = 557{\cdot}2$, monoclinic, $P2_1/a$, $a = 10{\cdot}015\ (4)$, $b = 16{\cdot}134\ (6)$, $c = 12{\cdot}175\ (5)$ Å, $\beta = 108{\cdot}12\ (5)^\circ$, $V = 1869{\cdot}7\ (9)$ Å^3, $Z = 4$, $D_m = 2{\cdot}00\ (5)$, $D_x = 1{\cdot}98$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 8{\cdot}46$ mm^{-1}, $F(000) = 1064$, $T = 294\ (2)$ K, $R = 0{\cdot}038$ for 2663 unique observed reflections. The complex consists of monomeric $PtCl_4$-(1-propylimidazole)$_2$ units. The coordination geometry is octahedral. The two 1-propylimidazole ligands are *cis*-coordinated to Pt, the Pt—N distances are 2·061 (9) and 2·045 (8) Å. The Pt—Cl distances ranging from 2·310 (3) to 2·322 (3) Å are normal for complexes of Pt^{IV}.

Introduction. In contrast to neutral Pt^{II} complexes, the Pt^{IV} analogues are rather poorly investigated though their biological and therapeutical properties seem to be equally interesting. Therefore we now present the crystal structure of tetrachlorobis(1-propylimidazole)-platinum(IV) which we obtained by a modification of the method of Braddock, Connors, Jones, Khokhar, Melzack & Tobe (1975).

Experimental. 0·48 g (0·001 mol) of *cis*-dichlorobis-(1-propylimidazole)platinum(II) was suspended in 20 ml of 2*M* HCl and 4 ml of 30% H_2O_2. The mixture was boiled until a yellow solution was obtained, then it

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was extracted with CH_2Cl_2 to remove by-products and the water solution was evaporated. The crude substance was crystallized from dimethylformamide (m.p. 496 K). Yellow crystals 0·14 × 0·14 × 0·20 mm. D_m by flotation in CCl_4/1,2-dibromoethane, monoclinic from Weissenberg photographs; Syntex $P2_1$ computer-controlled four-circle diffractometer; scintillation counter, graphite monochromator; cell parameters by least squares from setting angles of 15 reflections with $15 < 2\theta < 26°$ measured on diffractometer; 2876 independent reflections; $2\theta_{\max} = 55{\cdot}0°$; variable θ–2θ scans, scan rate 2·0–29·3° $\min^{-1}$ depending on intensity, two standards measured every 50 reflections, variation in their intensities ±4%; corrections for Lorentz and polarization effects, but not for extinction; calculations performed on a NOVA 1200 computer with programs supplied by Syntex (1976), scattering factors for neutral atoms, corrected for anomalous dispersion (for Pt and Cl), from *International Tables for X-ray Crystallography* (1974); for structure solution and refinement 2663 amplitudes with $F \geq 7\sigma(F)$ retained, $h\,0 \rightarrow 11$, $k\,0 \rightarrow 19$, $l\,-14 \rightarrow 13$, Patterson synthesis and difference-Fourier methods, full-matrix least-squares refinement minimizing $\sum w(|F_o| - |F_c|)^2$; $w = 1/\sigma^2(F)$; H atoms included in geometrically calculated positions (with $B = 4{\cdot}5$ Å^2), non-H atoms with anisotropic thermal parameters; an absorption correction following the *DIFABS* procedure (Walker & Stuart, 1983) was applied to isotropically refined data; max. and min. correction factors 1·235 and 0·745, respectively; max. Δ/σ in final cycle 0·1; max. and min. electron density in final difference map 0·20 and −0·28 e Å^{-3}; $R = 0{\cdot}038$, $wR = 0{\cdot}050$, $S = 3{\cdot}558$.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{1}{3}(B_{11} + B_{22} + B_{33}).$$

	x	y	z	B_{eq}
Pt	0·23031 (4)	0·01945 (3)	0·26588 (3)	2·03 (2)
Cl(1)	0·2041 (3)	0·0989 (2)	0·1026 (2)	3·7 (2)
Cl(2)	0·4659 (3)	0·0009 (2)	0·2847 (3)	3·8 (3)
Cl(3)	0·1718 (3)	−0·0990 (2)	0·1547 (2)	3·3 (2)
Cl(4)	0·2564 (3)	−0·0590 (2)	0·4311 (2)	3·2 (2)
N(11)	0·0208 (8)	0·0379 (5)	0·2471 (7)	2·4 (7)
N(12)	−0·2018 (9)	0·0520 (6)	0·1701 (7)	2·9 (7)
N(21)	0·2779 (9)	0·1238 (5)	0·3659 (7)	2·7 (7)
N(22)	0·3737 (10)	0·2040 (6)	0·5106 (8)	3·6 (8)
C(11)	−0·0768 (11)	0·0423 (7)	0·1510 (9)	2·8 (9)
C(12)	−0·0356 (11)	0·0433 (7)	0·3367 (8)	2·9 (9)
C(13)	−0·1765 (12)	0·0537 (8)	0·2851 (9)	3·5 (9)
C(14)	−0·3380 (11)	0·0663 (8)	0·0808 (9)	3·5 (10)
C(15)	−0·3678 (15)	0·1561 (11)	0·0651 (13)	6·1 (15)
C(16)	−0·5010 (16)	0·1730 (11)	−0·0291 (15)	7·3 (18)
C(21)	0·3732 (11)	0·1277 (8)	0·4692 (9)	3·0 (9)
C(22)	0·2179 (15)	0·1985 (8)	0·3420 (11)	4·7 (12)
C(23)	0·2785 (16)	0·2500 (7)	0·4310 (11)	5·0 (13)
C(24)	0·4598 (15)	0·2313 (8)	0·6269 (11)	4·9 (13)
C(25)	0·5631 (4)	0·2952 (10)	0·6255 (14)	5·9 (15)
C(26)	0·6508 (15)	0·3215 (10)	0·7444 (14)	5·9 (15)

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Pt–Cl(1)	2·311 (3)	Pt–Cl(2)	2·318 (3)
Pt–Cl(3)	2·310 (3)	Pt–Cl(4)	2·322 (3)
Pt–N(1)	2·061 (9)	Pt–N(2)	2·045 (8)
N(11)–C(11)	1·272 (13)	N(21)–C(21)	1·322 (13)
N(12)–C(11)	1·352 (15)	N(22)–C(21)	1·329 (15)
N(11)–C(12)	1·377 (13)	N(21)–C(22)	1·337 (13)
N(12)–C(13)	1·344 (13)	N(22)–C(23)	1·350 (17)
C(12)–C(13)	1·365 (17)	C(22)–C(23)	1·350 (18)
N(12)–C(14)	1·474 (14)	N(22)–C(24)	1·478 (16)
C(14)–C(15)	1·479 (21)	C(24)–C(25)	1·465 (21)
C(15)–C(16)	1·489 (23)	C(25)–C(26)	1·502 (23)
Cl(1)–Pt–Cl(2)	89·8 (1)	Cl(2)–Pt–N(21)	90·3 (2)
Cl(1)–Pt–Cl(3)	90·8 (1)	Cl(3)–Pt–Cl(4)	89·8 (1)
Cl(1)–Pt–Cl(4)	179·3 (1)	Cl(3)–Pt–N(11)	89·5 (2)
Cl(1)–Pt–N(11)	89·2 (2)	Cl(3)–Pt–N(21)	178·5 (2)
Cl(1)–Pt–N(21)	89·9 (2)	Cl(4)–Pt–N(11)	90·6 (2)
Cl(2)–Pt–Cl(3)	90·9 (1)	Cl(4)–Pt–N(21)	89·4 (2)
Cl(2)–Pt–Cl(4)	90·4 (1)	N(11)–Pt–N(21)	89·3 (3)
Cl(2)–Pt–N(11)	178·9 (2)		
Pt–N(11)–C(11)	125·0 (7)	Pt–N(21)–C(21)	124·8 (7)
Pt–N(11)–C(12)	125·1 (7)	Pt–N(21)–C(22)	127·3 (8)
C(11)–N(11)–C(12)	109·8 (9)	C(21)–N(21)–C(22)	107·8 (10)
N(11)–C(12)–C(13)	105·3 (9)	N(21)–C(22)–C(23)	108·7 (12)
C(12)–C(13)–N(12)	108·0 (10)	C(22)–C(23)–N(22)	106·1 (12)
C(13)–N(12)–C(11)	107·3 (9)	C(23)–N(22)–C(21)	108·5 (10)
N(12)–C(11)–N(11)	109·6 (9)	N(22)–C(21)–N(21)	108·7 (10)
C(11)–N(12)–C(14)	125·9 (9)	C(21)–N(22)–C(24)	124·9 (10)
C(13)–N(12)–C(14)	126·6 (10)	C(23)–N(22)–C(24)	126·5 (11)
N(12)–C(14)–C(15)	110·6 (10)	N(22)–C(24)–C(25)	113·8 (12)
C(14)–C(15)–C(16)	112·0 (13)	C(24)–C(25)–C(26)	113·0 (13)

Discussion. The final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* A perspective drawing of the $[PtCl_4(C_6H_{10}N_2)_2]$ complex is presented in Fig. 1. Table 2 shows interatomic distances and angles.

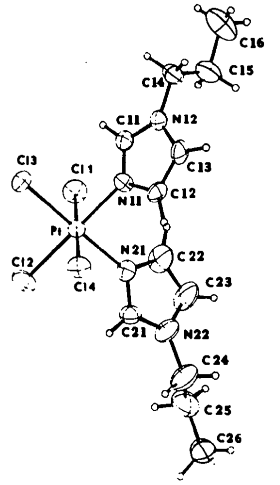

Fig. 1. A view of the title complex, showing the atom numbering.

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* Lists of structure factors, anisotropic thermal parameters, least-squares-planes data and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44402 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The complex consists of monomeric $PtCl_4(C_6H_{10}N_2)_2$ units. The Pt atom is octahedrally surrounded by four Cl atoms, and by the N atoms from the two 1-propylimidazole ligands in a *cis* geometry. All four Pt—Cl bonds are equal [ranging from 2·310 (3) to 2·322 (3) Å] and are practically identical to those observed in other six-coordinate Pt^{IV} complexes: tetramethylethylenediammonium hexachloroplatinate(IV) [2·304–2·316 (5) Å] (Bisi-Castellani, Manotti-Lanfredi, Tiripicchio, Maresca & Natile, 1984); 2·318 (2) Å in 9-methylguaninium hexachloroplatinate(IV) dihydrate (Terzis & Mentzafos, 1983) and 2·314 (1) Å in K_2PtCl_6 (Ohba & Saito, 1984). The Pt—N(11) and Pt—N(21) bond distances of 2·061 (9) and 2·045 (8) Å are in good agreement with those found for other compounds of Pt^{IV} with nitrogen-donor ligands (Fanizzi, Natile, Maresca, Manotti-Lanfredi & Tiripicchio, 1984).

The structural parameters of the two independent imidazole rings are very similar and are well within the range observed in other metal complexes and derivatives (Navarro-Ranninger, Martinez-Carrera & García-Blanco, 1983; Brouant, Barbe, Marsura & Luu-Duc, 1985). Both imidazole rings are planar; the propyl group is twisted as indicated by the torsion angles C(13)—N(12)—C(14)—C(15) = 80 (1) and C(23)—N(22)—C(24)—C(25) = 67 (2)°. The dihedral angle between the planes of the two imidazole rings is 111 (2)°, while the dihedral angles between the N(11) and N(21) ligand-ring planes and the coordination plane around Pt^{IV} are 124 (1) and 51 (1)° respectively.

There are no abnormally short distances. The crystal structure consists of discrete molecules of $PtCl_4$(1-propylimidazole)$_2$ with adjacent molecules held by van der Waals forces.

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References

Bisi-Castellani, C., Manotti-Lanfredi, A. M., Tiripicchio, A., Maresca, L. & Natile, G. (1984). *Inorg. Chim. Acta*, **90**, 155–159.

Braddock, P. D., Connors, T. A., Jones, M., Khokhar, A. R., Melzack, D. H. & Tobe, M. L. (1975). *Chem.-Biol. Interact.* **11**, 145–161.

Brouant, P., Barbe, J., Marsura, A. & Luu-duc, C. (1985). *Acta Cryst.* C**41**, 1369–1372.

Fanizzi, F. P., Natile, G., Maresca, L., Manotti-Lanfredi, A. M. & Tiripicchio, A. (1984). *J. Chem. Soc. Dalton Trans.* pp. 1467–1470.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Navarro-Ranninger, M. C., Martinez-Carrera, S. & García-Blanco, S. (1983). *Acta Cryst.* C**39**, 186–188.

Ohba, S. & Saito, Y. (1984). *Acta Cryst.* C**40**, 1639–1641.

Syntex (1976). *XTL/XTLE Structure Determination System*. Syntex Analytical Instruments, Cupertino, California, USA.

Terzis, A. & Mentzafos, D. (1983). *Inorg. Chem.* **22**, 1140–1143.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1988). C**44**, 439–443

A Neutron Diffraction Study of Bis(cyclopentadienyl)(methyl)(methylene)tantalum(V) at 15 K*

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Abstract. $[Ta(C_5H_5)_2(CH_2)(CH_3)]$, $M_r = 340{\cdot}206$, monoclinic, $P2_1/c$, $a = 6{\cdot}492$ (1), $b = 11{\cdot}539$ (2), $c = 15{\cdot}174$ (2) Å, $\beta = 117{\cdot}33$ (2)°, $V = 1009{\cdot}8$ (3) Å^3, $Z = 4$, $D_x = 2{\cdot}238$ Mg m^{-3}, neutrons, $\lambda = 1{\cdot}1617$ (2) Å, $\mu = 0{\cdot}2473$ mm^{-1}, $F(000) = 122{\cdot}68$ fm. $T = 15(0{\cdot}5)$ K. The structure has been refined based on 2446 reflections to yield an unweighted R value based on F^2 of 0·043. The molecule has approximate C_s point symmetry. All C—H bond distances have been determined with e.s.d.'s ≤ 0·003 Å. The mean C—H distance [1·091 (4) Å] in the methyl group is slightly longer than that in the methylene group [1·081 (5) Å], while the

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