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Structures of *cis*-Amminedichloro(cyclopentylamine)platinum 0.25-Hydrate and Potassium Trichloro(cyclopentylamine)platinate 0.5-Hydrate

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Abstract. *cis*-Pt(NH₃)(cyclopentylamine)Cl₂· $\frac{1}{4}$ H₂O, C₅H₁₄Cl₂N₂Pt·0.25H₂O, *M_r* = 372.7, tetragonal, *P4b2*, *a* = 11.456 (4), *c* = 16.172 (6) Å, *V* = 2122 (1) Å³, *Z* = 8, *D_x* = 2.333 Mg m⁻³, λ(Mo Kα) = 0.71069 Å, μ = 13.83 mm⁻¹, *F*(000) = 1380, *T* = 295 K, *R* = 0.040 for 725 unique observed reflections. Pt has *cis* square-planar coordination with bond distances Pt–Cl = 2.31 (1), 2.33 (1) Å and Pt–N = 2.02 (3) and 1.99 (2) Å. The C atoms in the cyclopentylamine ligand, especially C(4), show very high thermal motion. The structure is stabilized by hydrogen bonding involving the ammine ligand and the amino group with the chloro ligands. K[Pt(cyclopentylamine)Cl₃] $\cdot\frac{1}{2}$ H₂O, K⁺·C₅H₁₁Cl₃NPt⁻·0.5H₂O, *M_r* = 434.7, monoclinic, *C2/c*, *a* = 33.169 (21), *b* = 5.881 (4), *c* = 12.280 (8) Å, β = 102.45 (5)°, *Z* = 8, *V* = 2339 (3) Å³, *D_x* = 2.468 Mg m⁻³, λ(Mo Kα) = 0.71069 Å, μ = 13.13 mm⁻¹, *F*(000) = 1608, *T* = 295 K, *R* = 0.043 for 1399 unique observed reflections. Pt has square-planar coordination with Pt–Cl = 2.313 (4), 2.309 (4), 2.304 (4) Å, and Pt–N = 2.03 (1) Å. The carbon atoms of cyclopentylamine are disordered and two positions were refined for C(1). The compound crystallizes with a molecule of water located on a twofold axis. The water molecule is exceptionally close to the K⁺ ion [O⋯K = 2.756 (9) Å]. The compound is stabilized by hydrogen bonds between the amino group and one chloro ligand.

Introduction. Platinum(II) compounds of the type *cis*-PtL₂Cl₂ with *L* = cycloalkylamine are less toxic than cisplatin, *cis*-Pt(NH₃)₂Cl₂ (Braddock, Connors,

Jones, Khokhar, Melzack & Tobe, 1975). A considerable decrease in toxicity was also observed as ring size of the amine increases. The changes in toxicity were postulated to be structure-related (Braddock *et al.*, 1975). The low toxicity of the cyclic amine complexes was suggested to be caused by the great flexibility of the larger rings, allowing orientation of the rings so that they protect the axial positions above and below the platinum plane, thus preventing coordination to the S atoms in the kidney tubules (Lock, Speranzini & Zvagulis, 1980).

The crystal structures of *cis*-Pt(cyclobutylamine)₂Cl₂ (Lock & Zvagulis, 1981) and *cis*-Pt(cyclohexylamine)₂Cl₂ (Lock, Speranzini & Zvagulis, 1980) have confirmed that a few carbon atoms of the rings are in a position to block the two axial sites. However, the crystal structure of *cis*-Pt(cyclopropylamine)₂Cl₂ (Howard-Lock, Lock, Turner & Zvagulis, 1981) has shown that in the solid state the two cyclopropyl rings are on the same side of the platinum coordination plane, leaving the second axial site opened. But in solution there is probably free rotation about the Pt–N bonds and again the two axial positions could be partly blocked.

We have synthesized several platinum(II) compounds containing one NH₃ and one cycloalkylamine ligand, *cis*-Pt(*L*)(NH₃)Cl₂ (Rochon & Kong, 1986), in order to compare the antitumor activity and toxicity of these compounds with those of cisplatin and *cis*-PtL₂Cl₂. The three compounds *cis*-Pt(*L*)(NH₃)Cl₂ with *L* = cyclopropylamine, cyclobutylamine and cyclopentylamine were found to be much more toxic than the

corresponding *cis*-PtL₂Cl₂ compounds, but there seems to be a slight decrease in toxicity as ring size of L increases. The toxicity of these compounds is similar to that of cisplatin (Rochon & Melanson, 1986).

We have recently reported the crystal structure of *cis*-Pt(NH₃)(cyclobutylamine)Cl₂ (Rochon & Melanson, 1986). In the solid state, the cycloalkylamine blocks only one axial site, although in solution, because of the free rotation around the Pt–N bond, both axial sites should be partly protected, but less than in the bis(cycloalkylamine) complexes. We have therefore suggested that other factors like hydrogen bonding of the ammine ligand should be taken into consideration as a possible explanation for the differences in toxicity observed for the different types of Pt compounds. We have now determined the crystal structure of *cis*-Pt(NH₃)(cyclopentylamine)Cl₂. For comparison, the structure of the monoamine Pt^{II} complex K[Pt(cyclopentylamine)Cl₃], whose synthesis has recently been reported (Rochon, Melanson & Doyon, 1987), has also been examined. No X-ray diffraction work has been performed so far on cyclopentylamine metal complexes.

Experimental. The two compounds *cis*-Pt(NH₃)(cpa)Cl₂· $\frac{1}{4}$ H₂O (I) and K[Pt(cpa)Cl₃]. $\frac{1}{2}$ H₂O (II) (cpa = cyclopentylamine) were synthesized as already reported (Rochon & Kong, 1986; Rochon, Melanson & Doyon, 1987). Compound (II) was recrystallized in acetone. The synthetic procedure for compound (I) usually produced a powder, but on one occasion pale-yellow crystals were obtained. Most of the crystals were either twinned or too small for crystallographic methods. Only one crystal was found adequate and its structure was determined. Recrystallization of the powder in different solvents or combination of solvents was not successful.

The experimental details of the two crystal structure determinations are shown in Table 1. Space groups determined by precession photographs; Syntex P1; graphite-monochromatized Mo K α radiation; cell parameters from refined angles of 15 centered reflections; $\sigma(I)$ calculated as in Authier-Martin & Beauchamp (1977) for (I) and Melanson & Rochon (1975) for (II); absorption correction based on equations of crystal faces; data corrected for Lorentz and polarization effects; atomic scattering factors of Cromer & Mann (1968) for Pt, Cl, K, O, N, C and of Stewart, Davidson & Simpson (1965) for H; anomalous-dispersion terms of Pt, Cl and K from Cromer (1965).

Patterson map for (II) showed position of Pt; other non-hydrogen atoms located by structure factors and Fourier-map calculations. The C atoms in the cpa ligands were very disordered. Two positions (occupancy factor = 0.5) were refined for C(1). A residual peak in K[Pt(cpa)Cl₃] located on the twofold axis was assigned to a molecule of water. For (I), space groups

Table 1. *Experimental details of the two crystal structures*

	<i>cis</i> -Pt(NH ₃)(cpa)Cl ₂ · $\frac{1}{4}$ H ₂ O	K[Pt(cpa)Cl ₃]. $\frac{1}{2}$ H ₂ O
Max. 2 θ (°)	60	50
Quadrants	<i>h, k, l</i> (<i>k</i> \geq <i>h</i>)	<i>h, k, \pml</i>
Scan technique	2 θ/θ	2 θ/θ
Scan speed (° min ⁻¹)	1.0–24.0	1.0–24.0
Standard reflections, variation (%)	400; 040; 006 <1.7	070; 400; 313 <1.5
No. of independent reflections	1707	2066
Observed reflections	725 (<i>I</i> _{net} > 3 σ)	1399 (<i>I</i> _{net} > 2.5 σ)
<i>h, k, l</i>	0 \rightarrow 11, <i>h</i> \rightarrow 16, 0 \rightarrow 22	0 \rightarrow 39, 0 \rightarrow 7, –14 \rightarrow 14
μ (mm ⁻¹)	13.82	13.13
Crystal faces and dimensions (mm)	(001)–(00 $\bar{1}$) (0.38) (110)–($\bar{1}$ 10) (0.29) (1 $\bar{1}$ 0)–($\bar{1}$ 10) (0.13)	(100)–($\bar{1}$ 00) (0.026) (001)–(00 $\bar{1}$) (0.066) (010)–(0 $\bar{1}$ 0) (0.594)
Transmission factors	0.060–0.176	0.421–0.719
$\Delta\rho_{\max}$ (e Å ⁻³)	1.3 (close to Pt)	1.45 (close to Pt)
<i>R</i>	0.040	0.043
<i>wR</i>	0.041	0.038
Weighting scheme	1/ σ^2 (<i>F</i>)	1/ σ^2 (<i>F</i>)
Standard deviation (unit weight)	1.21	1.58

P4bm, *P4/mbm* and *P $\bar{4}$ b2* were consistent with *4/mmm* Laue symmetry and systematic absences (0*kl*, *k* = 2*n*; *h*0*l*, *h* = 2*n*) revealed by precession photographs and confirmed by the full data set. The structure could not be solved in the first two space groups, where the Pt atoms would occupy special positions [8(*k*) in *P4/mbm*; two sets of 4(*c*) in *P4bm*]. In *P $\bar{4}$ b2*, the 8 Pt atoms (general position) were readily located from Patterson map. The Cl, C and N positions were obtained from ΔF map phased on Pt. Thermal motion was very high for the cyclopentyl ring, especially for C(4). All attempts to identify and refine resolved sites failed. Residual electron density of 2.5 e Å⁻³ at (0.45, 0.95, 0) [equipoint 4(*h*)] in the ΔF map raised problems. It could only be assigned to the O atom of a lattice water molecule, but no direct confirmation from elemental analysis or density measurement was possible since only one crystal was available. Furthermore, half occupancy had to be postulated, because full occupancy would require two symmetry-equivalent oxygens to be only 1.9 Å apart. This model refined normally. Nevertheless, the size and shape of the thermal ellipsoid suggest that water does not lie exactly on the twofold axis. In various attempts to displace it, refinement invariably carried it back to the twofold axis. Thus, the refined oxygen position probably represents the average location of half-oxygens disordered on two sites off the axis. Refinement for both structures using full-matrix least-squares calculations minimizing $\sum w(F_o - F_c)^2$; isotropic secondary-extinction correction (Coppens & Hamilton, 1970); H atoms fixed at calculated positions (C–H = 0.95 Å, N–H = 0.95 Å) with isotropic *B* = 9.0 (I) and 6.0 Å² (II). Refinement of crystal (I) with all (*x, y, z*) coordinates changed into (–*x, –y, –z*) converged to higher *R* factors (0.046 and 0.049) indicating that the coordinates of Table 2 correspond to the absolute structure. Calculations on a Cyber 835 with

programs of Authier-Martin & Beauchamp (1977) for (I) and on a Cyber 830 with programs of Melanson & Rochon (1975) for (II).*

Discussion. The refined atomic parameters are listed in Table 2. A labeled diagram of the two structures is shown in Fig. 1. The coordination around the Pt atom is square planar and the angles around the Pt atom are close to the expected values of 90 and 180°. The weighted best coordination plane was calculated through the five atoms. The deviations are for (I): Pt, 0.000 (1); Cl(1), 0.001 (10); Cl(2), 0.006 (14); N(1), 0.02 (3); N(2), -0.03 (3) Å; and for (II): Pt, 0.0004 (6); Cl(1), -0.002 (4); Cl(2), -0.007 (4); Cl(3), -0.003 (5); N, -0.09 (1) Å. The bond distances and angles are reported in Table 3. Pt(NH₃)(cpa)Cl₂ is the *cis* isomer as expected from previous studies (Rochon & Kong, 1986).

The Pt-Cl bond lengths are normal [from 2.304 (4) to 2.327 (10) Å] and agree well with the published values found in aminochloroplatinum(II) compounds (Rochon & Melanson, 1986; Rochon, Melanson & Doyon, 1987; Lock & Zvagulis, 1981). The C atoms in the organic ligand are disordered as shown by the high thermal factors, resulting in low precision of the distances and angles. In K[Pt(cpa)Cl₃].½H₂O two positions were refined for C(1) leading to two possible conformations (50% each) both of which are of the envelope type: C(1) and C(1') deviate by -0.50 (5) and 0.32 (5) Å, respectively, from the plane formed by the other four ring atoms. The disorder could not be resolved in *cis*-Pt(NH₃)(cpa)Cl₂.½H₂O where C(4) is particularly agitated which explains the artificially short distances around C(4). Very few crystal structures have been reported on monosubstituted cyclopentyl derivatives and they were generally found to contain disordered cyclopentyl rings (Winter, Hecht & Bradaczek, 1981; Mallikarjunan, Chacko & Zand, 1972; Sarma, Ramirez, Narayanan, McKeever, Okazaki & Marecek, 1978).

The structure of *cis*-Pt(NH₃)(cpa)Cl₂.½H₂O consists of layers of molecules parallel to the *ab* plane which is shown in Fig. 2. Hydrogen bonding plays an important role in stabilizing the crystal. The two amine protons are hydrogen bonded to Cl(1) and Cl(2) with distances N(1)⋯Cl = 3.48 (3) and 3.37 (3) Å and acceptable angles (Table 4). At least two of the ammine protons are also involved in the hydrogen-bonding system, with N(2)⋯Cl distances of 3.28 (3) and 3.46 (3) Å. All the hydrogen bonds are located on the same side of the layers defining a hydrophilic face, and the hydrophobic

Table 2. Positional parameters ($\times 10^4$ for Pt, Cl, K and $\times 10^3$ for N, O, C) with their e.s.d.'s and temperature factors ($\times 10^3$)

$$U_{eq} = \frac{1}{3} \sum_{i=1}^3 \sum_{j=1}^3 U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq} (Å ²)
<i>cis</i> -Pt(NH ₃)(cpa)Cl ₂ .½H ₂ O				
Pt	1731 (1)	6769 (1)	1697 (1)	53
Cl(1)	1352 (9)	8492 (8)	982 (7)	70
Cl(2)	-32 (8)	6868 (9)	2387 (10)	81
O	441 (6)	-59 (6)	0	82
N(1)	205 (2)	527 (2)	231 (2)	52
N(2)	328 (2)	664 (3)	114 (2)	60
C(1)	292 (4)	532 (4)	303 (2)	119
C(2)	234 (7)	589 (7)	374 (4)	279
C(3)	231 (6)	491 (10)	431 (3)	332
C(4)	334 (17)	452 (20)	416 (7)	1332
C(5)	309 (5)	425 (7)	346 (4)	224
K[Pt(cpa)Cl ₃].½H ₂ O				
Pt	915.7 (2)	2187.4 (9)	1925.2 (5)	38
K	385 (1)	1529 (6)	4144 (3)	51
Cl(1)	502 (1)	-1015 (6)	1781 (3)	47
Cl(2)	540 (1)	3456 (6)	227 (3)	53
Cl(3)	1294 (1)	971 (8)	3621 (3)	64
N	126 (1)	507 (2)	210 (1)	53
O	0	447 (2)	250	49
C(1)	172 (1)	489 (10)	192 (4)	77
C(1')	153 (1)	538 (9)	141 (5)	75
C(2)	189 (1)	729 (4)	187 (2)	99
C(3)	220 (1)	697 (5)	122 (2)	126
C(4)	210 (1)	485 (5)	56 (2)	105
C(5)	174 (1)	380 (4)	88 (2)	121

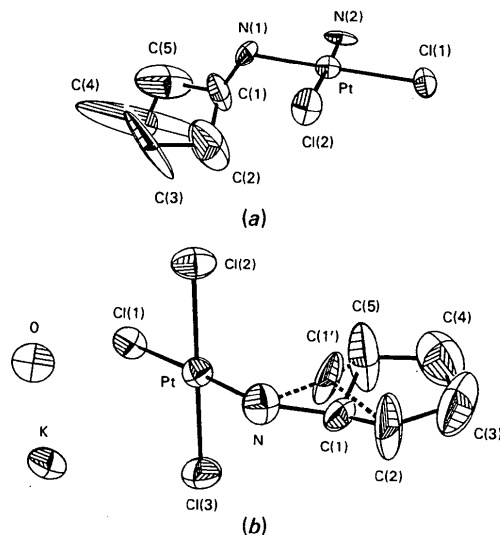


Fig. 1. Labeled diagrams of (a) *cis*-Pt(NH₃)(cpa)Cl₂.½H₂O and (b) K[Pt(cpa)Cl₃].½H₂O.

cyclopentylamine ligands are located on the other side of the layer. The O(1)⋯Cl(1) distance [3.99 (6) Å] from the average position for the water oxygen to two Cl(1) atoms is beyond the accepted range for hydrogen bonding (Stout & Jensen, 1968). However, as mentioned earlier, water is certainly disordered over several sites at some distance from the twofold axis, so that it is actually closer to one of the Cl(1) atoms and possibly

* Lists of anisotropic thermal factors, H coordinates, weighted least-squares planes and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51666 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. Bond distances (Å) and angles (°)

<i>cis</i> -Pt(NH ₃)(cpa)Cl ₂ · $\frac{1}{2}$ H ₂ O			
Pt—Cl(1)	2.327 (10)	C(1)—C(2)	1.48 (8)
Pt—Cl(2)	2.310 (12)	C(2)—C(3)	1.45 (12)
Pt—N(1)	2.02 (3)	C(3)—C(4)	1.28 (22)
Pt—N(2)	1.99 (2)	C(4)—C(5)	1.21 (14)
N(1)—C(1)	1.53 (5)	C(1)—C(5)	1.42 (9)
Cl(1)—Pt—Cl(2)	92.1 (4)	N(1)—C(1)—C(2)	108 (4)
Cl(1)—Pt—N(1)	179.2 (8)	N(1)—C(1)—C(5)	115 (4)
Cl(1)—Pt—N(2)	90.2 (8)	C(2)—C(1)—C(5)	94 (5)
Cl(2)—Pt—N(1)	87.8 (9)	C(1)—C(2)—C(3)	99 (6)
Cl(2)—Pt—N(2)	177.5 (9)	C(2)—C(3)—C(4)	97 (10)
N(1)—Pt—N(2)	89.9 (12)	C(3)—C(4)—C(5)	93 (13)
Pt—N(1)—C(1)	117 (2)	C(4)—C(5)—C(1)	106 (10)
K Pt(cpa)Cl ₃ · $\frac{1}{2}$ H ₂ O			
Pt—Cl(1)	2.315 (4)	C(1)—C(2)	1.52 (7)
Pt—Cl(2)	2.311 (4)	C(1')—C(2)	1.64 (6)
Pt—Cl(3)	2.302 (4)	C(2)—C(3)	1.45 (3)
Pt—N	2.040 (13)	C(3)—C(4)	1.49 (4)
N—C(1)	1.59 (5)	C(4)—C(5)	1.50 (4)
N—C(1')	1.37 (5)	C(1)—C(5)	1.43 (6)
		C(1')—C(5)	1.39 (6)
Cl(1)—Pt—Cl(2)	89.9 (1)	N—C(1)—C(5)	112 (4)
Cl(1)—Pt—Cl(3)	90.9 (1)	N—C(1')—C(5)	130 (4)
Cl(1)—Pt—N	177.1 (4)	C(1)—C(2)—C(3)	102 (3)
Cl(2)—Pt—Cl(3)	179.2 (2)	C(1')—C(2)—C(3)	106 (3)
Cl(2)—Pt—N	90.8 (4)	C(2)—C(3)—C(4)	108 (2)
Cl(3)—Pt—N	88.4 (4)	C(3)—C(4)—C(5)	107 (2)
Pt—N—C(1)	118 (2)	C(4)—C(5)—C(1)	103 (3)
Pt—N—C(1')	118 (2)	C(4)—C(5)—C(1')	110 (3)
N—C(1)—C(2)	108 (2)	C(2)—C(1)—C(5)	108 (4)
N—C(1')—C(2)	113 (4)	C(2)—C(1')—C(5)	103 (3)

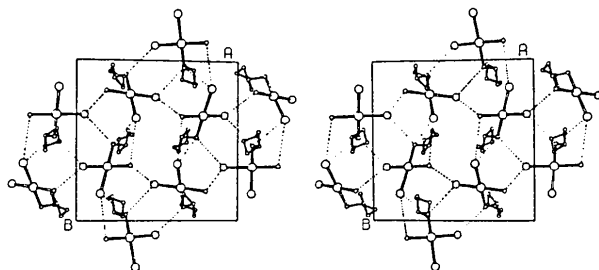


Fig. 2. Stereoscopic view of the packing of the molecules in the crystal *cis*-Pt(NH₃)(cpa)Cl₂· $\frac{1}{2}$ H₂O in the *ab* plane (the water molecules have been omitted). Dashed lines correspond to H bonds.

close enough for some weak O—H...Cl(1) interaction to take place.

The packing of the molecules in the crystal K|Pt(cpa)Cl₃· $\frac{1}{2}$ H₂O is shown in Fig. 3. The O atom of the water molecule is located on a twofold axis, exceptionally close to two K⁺ ions with distances O...K⁺ = 2.756 (9) Å and forms hydrogen bonds with two Cl(1) ligands with distances O...Cl(1) = 3.35 (1) Å. The K⁺ ion is surrounded by six Cl atoms at distances 3.192 (4) to 3.360 (5) Å, suggesting that packing energy around the K⁺ ion is an important stabilizing factor in the crystal. The amino group is hydrogen bonded to one chloro ligand. The distance N...Cl(1) = 3.38 (1) Å. There are no short N...O

Table 4. Distances (Å) and angles (°) between atoms involved in hydrogen bonds

<i>cis</i> -Pt(NH ₃)(cpa)Cl ₂ · $\frac{1}{2}$ H ₂ O			
N(1)...Cl(1) ⁱ	3.48 (3)	C(1)—N(1)...Cl(1)	99 (2)
		Pt—N(1)...Cl(1)	107 (1)
N(1)...Cl(2) ⁱⁱ	3.37 (3)	C(1)—N(1)...Cl(2)	116 (2)
		Pt—N(1)...Cl(2)	121 (1)
		Cl(1')...N(1)...Cl(2) ⁱⁱⁱ	88 (1)
N(2)...Cl(1) ⁱⁱⁱ	3.46 (3)	Pt—N(2)...Cl(1)	121 (1)
N(2)...Cl(2) ^{iv}	3.28 (3)	Pt—N(2)...Cl(2)	102 (1)
		Cl(1) ⁱⁱⁱ ...N(2)...Cl(2) ^{iv}	128 (1)
K Pt(cpa)Cl ₃ · $\frac{1}{2}$ H ₂ O			
N...Cl(1) ^v	3.38 (1)	Pt—N...Cl(1)	99.3 (5)
		C(1)—N...Cl(1)	138 (2)
		C(1')—N...Cl(1)	114 (2)
O...Cl(1) ^v	3.35 (1)		
O...Cl(1) ^{vi}	3.35 (1)	Cl(1) ^v ...O...Cl(1) ^{vi}	75.3 (2)

Symmetry code: (i) $\frac{1}{2}-x, y+\frac{1}{2}, z$; (ii) $-x, 1-y, z$; (iii) $y-\frac{1}{2}, x+\frac{1}{2}, -z$; (iv) $x+\frac{1}{2}, \frac{3}{2}-y, z$; (v) $x, y+1, z$; (vi) $-x, y+1, \frac{1}{2}-z$.

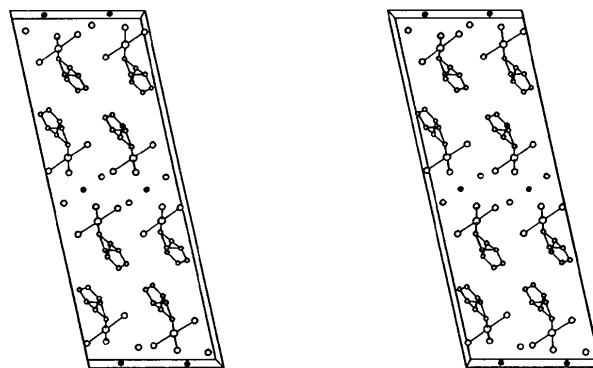


Fig. 3. Packing in the crystal K|Pt(cpa)Cl₃· $\frac{1}{2}$ H₂O (*c* axis horizontal, down *b* axis); the dark circles correspond to the water O atoms.

contacts. Fig. 3 shows clearly that the hydrophilic parts of the molecules are located around $a = 0$ and 0.5 where the K⁺ ions, the water molecules and the chloro ligands are found, while the hydrophobic cyclopentyl rings are orientated towards each other around $a = 0.25$ and 0.75 .

The large difference in toxicity between *cis*-Pt(cpa)₂Cl₂ (480 mg kg⁻¹) (Braddock *et al.*, 1975) and *cis*-Pt(NH₃)(cpa)Cl₂ (15–20 mg kg⁻¹) (Rochon & Melanson, 1986) cannot be explained solely by the fact that the cpa ligands protect the axial positions above and below the Pt plane, thus preventing coordination to the S atoms in the kidney tubules. In the solid state, the cycloalkyl ring in *cis*-Pt(NH₃)(cpa)Cl₂ blocks only one side of the plane but in solution free rotation around the Pt—N bond can be assumed and the two axial positions should be partly protected. Therefore other factors like hydrogen bonding around the NH₃ ligand should be considered. Extensive hydrogen bonding of the ammine

ligand in *cis*-Pt(NH₃)(cyclobutylamine)Cl₂ (toxicity ~ 10 mg kg⁻¹) has also been observed (Rochon & Melanson, 1986). The crystal structure of *cis*-Pt-(cpa)₂Cl₂ could provide further information, but so far it has not been possible to obtain the compound in a suitable crystalline form.

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Structure of a Rh^I Complex of a Bis(2,3-quinolino)-annulated *cis,syn,cis*-Tricyclo[6.3.0.0^{3,7}]undecane*

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Abstract. [Rh(cod)(bqtu)]BF₄ (2), C₃₃H₃₂N₂Rh.BF₄, *M_r* = 646.35, orthorhombic, *Pcmn*, *a* = 8.793 (2), *b* = 14.957 (2), *c* = 20.665 (3) Å, *V* = 2717.7 (6) Å³, *Z* = 4, *D_x* = 1.580 g cm⁻³, λ(Mo Kα) = 0.71073 Å, μ = 6.59 cm⁻¹, *F*(000) = 1320, *T* = 294 K, *R* = 0.0546 for 1829 reflections. The structure contains mononuclear ions with square-planar geometry about the Rh^I atom. The Rh^I atom is coordinated to the two double bonds of the 1,5-cyclooctadiene (cod) ligand and to two N atoms from the new bqtu ligand, (1). The two

midpoints of the double bonds and the two N atoms form the square-planar geometry. The axial sites are shielded by H atoms from the two ligands. The heptacyclic bqtu ligand has *cis*-fusion to the central five-membered ring leading to a folded conformation resulting in a cleft between π-electron systems with a *syn*-orientation of N atoms. The angle between these two systems is 87.0 (8)°.

Introduction. Our research groups are interested in fused-ring systems which exhibit significant strain energies, strong intramolecular interactions, enhanced or unusual reactivities or unusual geometric shapes (Marchand, 1988; Watson, 1982; Vögtle, Müller & Watson, 1984). Recently, the heptacyclic ligand (1), a bis(2,3-quinolino) derivative of *cis,syn,cis*-tricyclo-

* IUPAC name of ligand bqtu: 5b,5c,12a,13,13a,14-hexahydro-12*H*-cyclopenta[1'',2''':4,5;4'',3''':4',5']dicyclopenta[1,2-*b*:1',2'-*b'*]diquinoline.

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