= phenyl, *p*-chlorophenyl, *p*-methoxyphenyl or cyclohexyl) which was recently prepared in our laboratory. A related series of 2-quinaldinate (quin) complexes, $[Rh(quin)(CO)\{P(X\text{-}C_6H_4)_3\}_2]$ (X = 4-CH_3O, 4-CH_3, 3-CH_3, 4-F and 4-Cl), crystallizing in two isomeric forms was recently reported (Heras, Cano, Lobo & Pinilla, 1989). This structure determination established the stereochemistry of the addition product between $[Rh(cupf)(CO)(PX_3)]$ and an additional phosphine molecule and will help us to elucide the mechanism of this reaction. Structural details are comparable to those of $[Rh(cupf)(CO)(PPh_3)]$ (Basson, Leipoldt, Roodt & Venter, 1986), $[Rh(cupf)(CO)(CH_3)(I)(PPh_3)]$ (Basson, Leipoldt, Roodt & Venter, 1987) and $[Rh(Cl)_2\{ONN(C_6H_4Me\text{-}p)O\}(H_2O)(PPh_3)].0{\cdot}5Me_2CO$ (Ahmed, Edwards, Jones, McCleverty, Rothin & Tate, 1988).

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cis-Chloro(1-methylcytosine-N^3)(N,N,N',N'-tetramethylethylenediamine)platinum(II) Perchlorate Hemihydrate, *cis*-$[PtCl(C_5H_7N_3O)(C_6H_{16}N_2)]ClO_4.0{\cdot}5H_2O$

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Abstract. $C_{11}H_{24}N_5O_{5{\cdot}5}Cl_2Pt$, $M_r = 580{\cdot}34$, monoclinic, $C2/c$, $a = 12{\cdot}329\,(3)$, $b = 11{\cdot}078\,(3)$, $c = 27{\cdot}417\,(7)$ Å, $\beta = 93{\cdot}26\,(2)°$, $V = 3739\,(2)$ Å^3, $Z = 8$, $D_x = 2{\cdot}062$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 7{\cdot}91$ mm^{-1}, $F(000) = 2248$, $T = 291\,(1)$ K, final $R = 0{\cdot}029$ for 3138 unique observed [$F \geq 3{\cdot}0\sigma(F)$] diffractometer data. The cation contains N,N,N',N'-tetramethylethylenediamine chelated to platinum, coordinated chloride and a 1-methylcytosine nucleobase bound through the N(3) position. The angles of the coordination sphere of platinum display some deviation from ideal square planarity which is caused by the bite of the ethylenediamine [N(11)—Pt(1)—N(12) 85·8 (2)°]. Pt—N and Pt—Cl distances as well as geometry of the methylcytosine ring [Pesch, Preut & Lippert (1989). *Inorg. Chim. Acta.* In the press; Orbell, Marzilli & Kistenmacher (1981). *J. Am. Chem. Soc.* **103**, 5126–5133; Britten, Lippert, Lock & Pilon (1982). *Inorg. Chem.* **21**, 1936–1941] and of the diamine ligand [Orbell, Taylor, Birch, Lawton, Vilkins & Keefe (1988). *Inorg. Chim. Acta*, **152**, 125–134] are normal. The puckering of the diamine ligands is such that C(11) and C(12) are on opposite sides of the platinum coordination plane. The 1-methylcytosine ring is almost at right angles to the plane [86·2 (1)°]. The only hydrogen bond of less than 3 Å involves H_2O protons and oxygen sites of the 1-methylcytosine in such a way that H_2O links pairs of cations.

Experimental. The title compound was prepared by reaction of $Pt(C_6H_{16}N_2)Cl_2$, obtained according to a modified Dhara method (Dhara, 1970) from K_2PtCl_4 and $C_6H_{16}N_2$ with $AgClO_4$ (1 equiv.) and 1-methylcytosine (1 equiv.) at pH 5–6, 80 h, 303 K. On slow evaporation, pale yellow crystals were isolated in 35% yield. According to ^1H NMR spectroscopy, the filtrate contained more of the title compound together with the bis(1-methylcytosine) complex and free ligand. Crystal size ~0·12 × 0·12 × 0·33 mm, D_m

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not determined, $\omega/2\theta$ scan, scan speed 3·00–14·65° min^{-1} in θ, scan width (1·2 + dispersion)°; Nicolet *R*3*m*/*V* diffractometer, graphite-monochromated Mo $K\alpha$; lattice parameters from least-squares fit with 28 reflections up to $2\theta = 29{\cdot}74°$; ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity; six standard reflections (600; 080; 0,0,10; $\bar{6}00$; $0\bar{8}0$; $0,0,\overline{10}$) recorded every 2·5 h, only random deviations over 85·75 h of X-ray exposure; 7458 reflections measured $3{\cdot}0° \le 2\theta \le 50{\cdot}0°$, $-15 \le h \le 15$, $-13 \le k \le 1$, $-33 \le l \le 33$; after averaging ($R_{\rm int} = 0{\cdot}023$): 3305 unique reflections, 3138 with $F \ge 3{\cdot}0\sigma(F)$; Lorentz–polarization correction and absorption correction *via* ψ scans, max./min. transmission 1·00/0·95; systematic absences (hkl) $h + k = 2n + 1$, ($h0l$) $l = 2n + 1$ conform to space groups *C*2/*c* and *Cc*; structure solution *via* direct methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C—H, N—H 0·96 Å); refinement on *F* with 3138 reflections and 223 refined parameters; $w = 1{\cdot}0/[\sigma^2(F) + (0{\cdot}0037F^2)]$ which led to featureless analysis of variance in terms of $\sin\theta$ and F_o; $S = 1{\cdot}44$, $R = 0{\cdot}029$, $wR = 0{\cdot}030$ ($R = 0{\cdot}031$, $wR = 0{\cdot}038$ for all 3305 data), $(\Delta/\sigma)_{\rm max} = 0{\cdot}11$; largest peak in final ΔF map ± 0·8 (3) e Å^{-3}; atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974); programs: *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *SCHAKAL* (Keller, 1986), *PCK*83 (Williams, 1984), *PLATON* (Spek, 1982), *MISSYM* (Le Page, 1987). The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit cell in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1.* Bond lengths, bond angles, torsion angles and least-squares planes are given in Table 2.

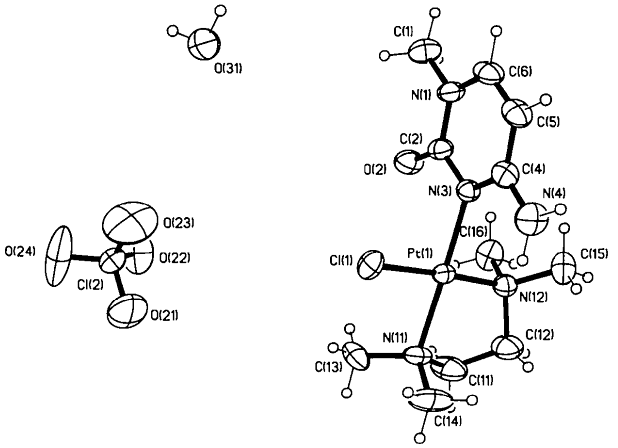

Fig. 1. General view (*SHELXTL-Plus* graphic) showing the atom-numbering scheme.

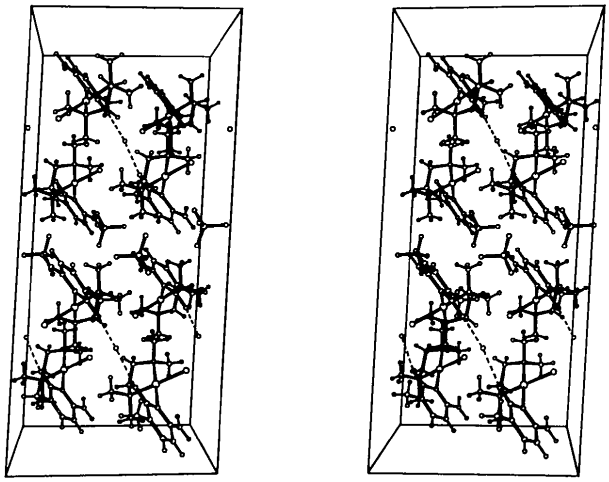

Fig. 2. Stereoscopic view (*SCHAKAL* graphic) of the unit cell (*a* horizontal, *c* vertical).

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* (Å^2 × 10^4)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Pt(1)	0·22550 (1)	0·93841 (2)	0·34266 (1)	258
Cl(1)	0·3740 (1)	0·8584 (1)	0·30447 (5)	391
Cl(2)	0·0855 (1)	0·7093 (1)	0·03304 (5)	412
O(2)	0·1021 (3)	0·7005 (4)	0·3303 (1)	455
O(21)	0·1269 (6)	0·8261 (6)	0·0395 (3)	1246
O(22)	−0·0283 (4)	0·7158 (6)	0·0361 (2)	851
O(23)	0·1296 (7)	0·6385 (7)	0·0705 (3)	1435
O(24)	0·1085 (6)	0·6602 (8)	−0·0114 (2)	1371
O(31)	0·5	0·3430 (6)	0·25	542
N(1)	0·1722 (4)	0·5830 (4)	0·3915 (2)	381
N(3)	0·2267 (3)	0·7865 (4)	0·3847 (1)	293
N(4)	0·3570 (4)	0·8657 (5)	0·4389 (2)	492
N(11)	0·2211 (4)	1·0893 (4)	0·2983 (2)	385
N(12)	0·0976 (3)	1·0181 (4)	0·3770 (2)	346
C(1)	0·1013 (6)	0·4842 (6)	0·3738 (3)	598
C(2)	0·1634 (4)	0·6912 (5)	0·3668 (2)	334
C(4)	0·2957 (4)	0·7725 (5)	0·4244 (2)	360
C(5)	0·3017 (5)	0·6617 (5)	0·4492 (2)	439
C(6)	0·2389 (5)	0·5699 (5)	0·4320 (2)	429
C(11)	0·1162 (5)	1·1557 (6)	0·3083 (3)	598
C(12)	0·0988 (6)	1·1496 (5)	0·3619 (3)	569
C(13)	0·2152 (6)	1·0552 (6)	0·2455 (2)	543
C(14)	0·3158 (5)	1·1675 (6)	0·3089 (3)	674
C(15)	0·1099 (5)	1·0163 (6)	0·4313 (2)	500
C(16)	−0·0064 (5)	0·9595 (6)	0·3618 (2)	499

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52635 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å), *bond angles* (°), *torsion angles* (°), *least-squares planes, dihedral angle* (°) *and hydrogen bond* (Å)

Pt(1)—Cl(1)	2·335 (1)	N(3)—C(2)	1·386 (6)
Pt(1)—N(3)	2·039 (4)	N(3)—C(4)	1·350 (6)
Pt(1)—N(11)	2·066 (4)	N(4)—C(4)	1·327 (7)
Pt(1)—N(12)	2·078 (4)	N(11)—C(11)	1·526 (8)
Cl(2)—O(21)	1·398 (7)	N(11)—C(13)	1·494 (7)
Cl(2)—O(22)	1·413 (5)	N(11)—C(14)	1·470 (8)
Cl(2)—O(23)	1·379 (8)	N(12)—C(12)	1·514 (8)
Cl(2)—O(24)	1·378 (7)	N(12)—C(15)	1·488 (7)
O(2)—C(2)	1·223 (6)	N(12)—C(16)	1·476 (7)
N(1)—C(1)	1·466 (8)	C(4)—C(5)	1·403 (8)
N(1)—C(2)	1·378 (7)	C(5)—C(6)	1·346 (8)
N(1)—C(6)	1·352 (7)	C(11)—C(12)	1·50 (1)

N(11)—Pt(1)—N(12)	85·8 (2)	Pt(1)—N(11)—C(11)	106·2 (4)
N(3)—Pt(1)—N(12)	94·4 (2)	C(13)—N(11)—C(14)	109·6 (5)
N(3)—Pt(1)—N(11)	178·1 (2)	C(11)—N(11)—C(14)	110·6 (5)
Cl(1)—Pt(1)—N(12)	177·0 (1)	C(11)—N(11)—C(13)	107·5 (5)
Cl(1)—Pt(1)—N(11)	92·2 (1)	Pt(1)—N(12)—C(16)	110·8 (4)
Cl(1)—Pt(1)—N(3)	87·7 (1)	Pt(1)—N(12)—C(15)	114·2 (3)
O(23)—Cl(2)—O(24)	109·9 (5)	Pt(1)—N(12)—C(12)	105·4 (4)
O(22)—Cl(2)—O(24)	109·2 (4)	C(15)—N(12)—C(16)	108·2 (4)
O(22)—Cl(2)—O(23)	109·6 (4)	C(12)—N(12)—C(16)	111·6 (5)
O(21)—Cl(2)—O(24)	112·7 (4)	C(12)—N(12)—C(15)	106·4 (5)
O(21)—Cl(2)—O(23)	107·8 (5)	N(1)—C(2)—N(3)	117·5 (5)
O(21)—Cl(2)—O(22)	107·6 (4)	O(2)—C(2)—N(3)	122·3 (5)
C(2)—N(1)—C(6)	121·6 (4)	O(2)—C(2)—N(1)	120·2 (5)
C(1)—N(1)—C(6)	121·0 (5)	N(3)—C(4)—N(4)	118·3 (5)
C(1)—N(1)—C(2)	117·2 (5)	N(4)—C(4)—C(5)	121·4 (5)
Pt(1)—N(3)—C(4)	122·3 (3)	N(3)—C(4)—C(5)	120·3 (5)
Pt(1)—N(3)—C(2)	116·3 (3)	C(4)—C(5)—C(6)	118·6 (5)
C(2)—N(3)—C(4)	120·9 (4)	N(1)—C(6)—C(5)	121·1 (5)
Pt(1)—N(11)—C(14)	111·5 (4)	N(11)—C(11)—C(12)	108·9 (5)
Pt(1)—N(11)—C(13)	111·4 (3)	N(12)—C(12)—C(11)	108·3 (5)
H(311)—O(31)—H(311^{i})	114·9		

N(11)—Pt(1)—N(12)—C(12)	−17·1 (4)	N(12)—Pt(1)—N(11)—C(11)	−11·6 (4)
Cl(1)—Pt(1)—N(3)—C(2)	−90·7 (3)	Pt(1)—N(11)—C(11)—C(12)	39·2 (6)
Cl(1)—Pt(1)—N(3)—C(4)	80·9 (4)	Pt(1)—N(12)—C(12)—C(11)	43·6 (5)
N(12)—Pt(1)—N(3)—C(4)	−97·0 (4)	N(11)—C(11)—C(12)—N(12)	−56·5 (6)
N(12)—Pt(1)—N(3)—C(2)	91·5 (4)		

No.	Plane through atoms	Equation of the plane (x along a; y in the plane ab; z along c^*)	χ^2
1	N(1),C(2),N(3),C(4),C(5),C(6)	$0{\cdot}765x - 0{\cdot}286y - 0{\cdot}578z = -6{\cdot}89$ Å	14·7
2	Pt(1),Cl(1),N(3),N(11),N(12)	$-0{\cdot}5879x - 0{\cdot}456y - 0{\cdot}6684z = -12{\cdot}33$ Å	416·1

Dihedral angle 1,2 86·2 (1)

Donor···Acceptor	
O(31)···O(2^{ii})	2·934 (5)

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$, (ii) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$.

Related literature. The bulkiness and hydrogen-bond capacity of amine ligands (A) in complexes of the type *cis*-A_2PtX_2 have been discussed with regard to the antitumor efficacy of the Pt drug (Cleare & Hoeschele, 1973), DNA binding properties (Sherman & Lippard, 1987; Hambley, 1988) and rotational freedom about the Pt—X axis when X = nucleobase (Cramer & Dahlstrom, 1979; Marcelis, Erkelens & Reedijk, 1984; Reily & Marzilli, 1986). We have prepared the title compound as well as related bis(nucleobase) complexes containing the N,N,N',N'-tetramethylethylenediamine ligand in order to study the effect of steric bulk and hindered nucleobase rotation on the rate of ligand substitution by excess cyanide (Frommer & Lippert, 1989). This question relates to the as yet unanswered question of incomplete removal of antitumor Pt drugs from DNA by cyanide.

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