parameters for all non-H atoms converged to R and wR of 0·049 and 0·067 with $w = 1/\sigma^2(F_o)$. H-atom coordinates, located from difference Fourier maps, included in structure-factor calculations, but not refined. 1866 reflections, $I > 3{\cdot}0\sigma_I$, used. $\Delta_{max}/\sigma = 0{\cdot}09$; max. and min. heights in final difference Fourier synthesis = 0·34 and −0·47 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations on a Gould SEL 32/27 computer using the *GX* package (Mallinson & Muir, 1985).

Discussion. Final positional and equivalent isotropic thermal parameters are given in Table 1.* Bond lengths, bond angles and pertinent intermolecular contacts are given in Table 2. Fig. 1 shows the atom-numbering scheme and Fig. 2 the contents of the unit cell along **a**.

The crystal contains two different types of glutarate residue (Fig. 1) – half-acid anions (R_1) and glutaric acid molecules (R_2). These can be differentiated by the location of the H atoms attached to O(1), O(4) and O(6) and by the dimensions of their CO_2^- groups (Table 2). The carbon skeleton of the glutaric acid molecule is substantially planar (r.m.s. deviation 0·014 Å) with the carboxyl groups inclined at 3·4 [C(6), O(6), O(7)] and 34·5° [C(10), O(1), O(8)] to the plane of the five carbon atoms. Atoms C(2), C(3), C(4), C(5) also approach coplanarity. The structure contains pairs of centrosymmetrically-related antiparallel helices formed from the end-to-end linking of R_1 residues into infinite chains by short asymmetric hydrogen bonds, O(4)···O(3) 2·506 (4) Å. These chains spiral around the twofold screw axes at $\frac{1}{4}y\frac{1}{2}$ and $\frac{3}{4}y\frac{1}{2}$. In this respect the structure is that of a type B_2 acid salt (Currie & Speakman, 1970). Cross-linking of helices which are related by the translation ($\bar{1}01$) occurs through hydrogen bonding [O(5)···O(6) 2·641 (4) and O(1)···O(2) 2·553 (3) Å] to the glutaric acid molecules which are arranged in ribbons along the edges of the unit cells (Fig. 2). By contrast $KH_3(CH_2)(CO_2)_2$ (Currie, 1972) has planar HY^- residues linked into rings interconnected by H_2Y molecules while $KH_3(CH_2)_2(CO_2)_2$ (Dunlop & Speakman, 1973) has succinic acid molecules 'festooned' along the B_2 chains. The pattern in $LiH_3(CH_2)(CO_2)_2$ (Soriano-Garcia & Parthasarathy, 1978) is again different, with chains of alternating HY^- and H_2Y residues. The K ion is coordinated by eight O atoms at distances less than 3·1 Å (Table 2). There are no further contacts less than 3·5 Å; O(4) and O(6) do not coordinate to the cation – both are of the O(H) type.

* 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 44590 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-Diamminebis(1-methylcytosine-N^3)platinum(II) Diperchlorate

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Abstract. $[Pt(NH_3)_2(C_5H_7N_3O)_2](ClO_4)_2$, M_r = 678·31, monoclinic, $P2_1/n$,* a = 14·569 (3), b = 10·388 (3), c = 6·753 (1) Å, β = 96·35 (1)°, V = 1015·8 (4) Å^3, Z = 2, D_m = 2·23 (2), D_x = 2·22 g cm^{-3}, graphite-monochromated Mo $K\alpha$ radiation, λ = 0·71069 Å, $F(000)$ = 652, μ = 76·0 cm^{-1}, T = 297 K, R = 0·0529 and wR = 0·0483 for 1794 unique reflections and 183 parameters. The Pt atom resides on an inversion centre and is coordinated to the N atom of each ammonia and the N(3) of each

* This non-standard cell may be transformed to the $P2_1/c$ cell, a = 6·753 (1), b = 10·388 (3), c = 15·365 (3) Å, β = 109·55 (1)° by the matrix (00 −1/010/101).

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cytosine nucleobase. This arrangement maintains the square-planar geometry about the Pt atom. The 1-methylcytosine nucleobases are coplanar with each other and are roughly at right angles to the ligand square plane [74·9 (3)°]. The amine groups above and below the nucleobase planes form hydrogen bonds to the perchlorate anions in the crystal lattice.

Introduction. Although many complexes of the *cis*-diammineplatinum(II) moiety with nucleobases have been characterized by X-ray crystallography, there have been very few X-ray studies of the corresponding *trans* complexes (Lippert, Lock & Speranzini, 1981*a*; Beyerle-Pfnur, Brown, Faggiani, Lippert & Lock, 1985). Such studies are necessary to allow comparison of structural differences in the *cis* and *trans* complexes.

trans-[Pt$(NH_3)_2$(1-MeC)(9-MeG)]$(ClO_4)_2$ was prepared by a modification of the method of Lippert, Lock & Speranzini (1981*b*) for *trans*-[Pt$(NH_3)_2$(1-MeC)(9-MeA)]$(ClO_4)_2$.H_2O with use of 9-methylguanine instead of 9-methyladenine. Upon slow evaporation of the aqueous solution of the desired product (pH = 5·0), long colourless needles of the title compound, *trans*-[Pt$(NH_3)_2$(1-MeC)$_2$]$(ClO_4)_2$, precipitated from solution and were isolated and identified by the use of single-crystal X-ray crystallography.

Experimental. Density was determined by flotation in a $CHCl_3/CH_2I_2$ solvent mixture. Crystal 0·2 × 0·1 × 0·1 mm. Precession photographs confirmed the space group as $P2_1/n$. Unit-cell parameters refined by least-squares fit of positional angles on 15 strong independent reflections measured on a Nicolet *P*3 diffractometer for $18·7 \leq 2\theta \leq 25·7°$, with use of monochromated Mo $K\alpha$ radiation. Intensities of $h,k,\pm l$ measured by the θ(crystal)–2θ(counter) scan technique. Scan rate 4·88 to 29·30° min^{-1} in 2θ. The ratio of total background time to scan time is 1:1. Two standard reflections monitored every 48 scans showed that no correction for instrument instability or crystal decay was required. 2067 measured reflections gave 1794 unique reflections. $R_{int} = 0·0083$. Reflections with $3\sigma_I \geq I \geq -3\sigma_I$ were treated by the method of French & Wilson (1978). Lp and absorption corrections were made (absorption correction factors, A^*, 1·84–2·91). Structure solved by the heavy-atom method. Anisotropic least-squares refinement minimized $\sum w(|F_o|-|F_c|)^2$, $w = (\sigma_F^2 + 0·000642F_o^2)^{-1}$. All H atoms were located in difference maps and their positions and temperature factors refined. Final $R = 0·0529$ and $wR = 0·0483$. The correction for secondary extinction, x (Sheldrick, 1976), was applied but its value was not significant [–0·0001 (2)]. In the final refinement cycle $(\Delta/\sigma)_{max} = 0·004$, $(\Delta/\sigma)_{avg} = 0·001$, $S = 1·188$. Final difference maps revealed no significant regions of electron density, with max. 0·357, min. –0·231 e $Å^{-3}$. Scattering factors for non-hydrogen atoms from *International Tables for X-ray Crystallography* (Cromer & Waber, 1974). Corrections for anomalous dispersion were made for Pt and Cl (Cromer & Ibers, 1974). Calculations employed *SHELX*76 (Sheldrick, 1976), *SNOOPI* (Davies, 1983) and *XTAL* (Stewart & Hall, 1983).*

Discussion. The cation and anion are shown in Fig. 1. Table 1 lists the final atomic parameters for non-hydrogen atoms and the corresponding bond lengths and angles are given in Table 2. The Pt atom lies on an inversion centre and so the ligand square plane comprises a *trans* arrangement of the ammonia N atoms and also of the N(3) atoms of the 1-methylcytosine ligands. The cytosine bases are coplanar. The dihedral angle between the pyrimidine rings and the ligand square plane is 74·9 (3)°, which is very close to the value reported for the cation of the corresponding nitrate salt (78·2°; Lippert *et al.*, 1981*a*). Also, the *anti* conformation of the ligands and all the important structural parameters in the cation do not differ significantly from those of the aforementioned nitrate salt (Lippert *et al.*, 1981*a*), except for the C(5)—C(6) distance which is significantly shorter in the perchlorate salt [1·34 (1) *vs* 1·41 (1) Å]. The shorter C(5)—C(6) distance we report does agree, however, with values reported for other platinum–cytosine cations (Beyerle-Pfnur *et al.*, 1985; Faggiani, Lock & Lippert, 1985; Faggiani, Lippert & Lock, 1982; Lippert *et al.*, 1981*b*; Lippert, Thewalt, Schollhorn, Goodgame & Rollins, 1984; Lock, Speranzini & Powell, 1976;

* Lists of structure factors, anisotropic temperature factors, H-atom positions, and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44565 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

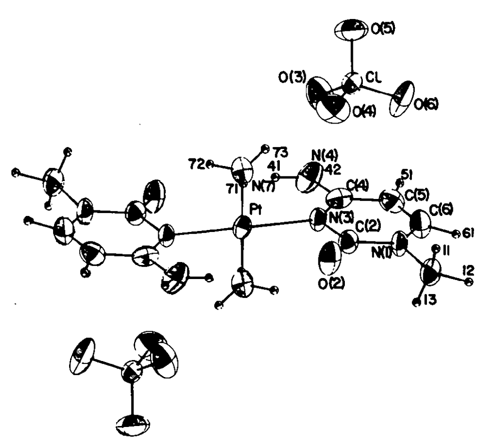

Fig. 1. The cation and anions, showing the atom numbering. For clarity, H atoms are numbered with affixes only, in smaller print, and in this diagram are given isotropic temperature factors of $U = 0·01$ $Å^2$.

Table 1. *Atomic positional parameters* ($\times 10^4$) *and equivalent isotropic temperature factors* ($Å^2 \times 10^3$)

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33} + 2U_{13}\cos\beta).$$

	x	y	z	U_{eq}
Pt	5000	5000	5000	30·2 (3)
N(1)	6538 (4)	2259 (6)	2182 (9)	35 (3)
C(2)	6044 (5)	3327 (8)	2575 (11)	35 (4)
N(3)	5555 (4)	3306 (5)	4232 (8)	29 (3)
C(4)	5545 (5)	2235 (8)	5337 (11)	39 (4)
C(5)	6063 (6)	1145 (8)	4889 (13)	44 (4)
C(6)	6552 (6)	1196 (8)	3329 (13)	45 (4)
N(7)	3945 (5)	4803 (8)	2736 (11)	40 (4)
C(1)	7135 (7)	2344 (10)	554 (14)	47 (5)
O(2)	6019 (4)	4281 (6)	1494 (8)	50 (3)
N(4)	5068 (6)	2226 (10)	6908 (11)	52 (5)
Cl	3521 (1)	1478 (2)	846 (3)	37 (1)
O(3)	3558 (5)	1861 (7)	2885 (9)	67 (4)
O(4)	3621 (5)	2596 (7)	−339 (10)	67 (4)
O(5)	2650 (4)	897 (7)	261 (11)	66 (4)
O(6)	4227 (5)	591 (9)	605 (14)	87 (5)

The platinum atom lies on an inversion centre and its coordinates were fixed at ($\tfrac{1}{2}, \tfrac{1}{2}, \tfrac{1}{2}$).

Table 2. *Selected interatomic distances* (Å) *and bond angles* (°)

Pt–N(3)	2·028 (6)	N(1)–C(1)	1·48 (1)
C(2)–O(2)	1·23 (1)	Cl–O(5)	1·422 (7)
C(4)–N(4)	1·33 (1)	N(1)–C(2)	1·36 (1)
C(6)–N(1)	1·35 (1)	N(3)–C(4)	1·34 (1)
Cl–O(4)	1·427 (8)	C(5)–C(6)	1·34 (1)
Pt–N(7)	2·055 (8)	Cl–O(3)	1·428 (7)
C(2)–N(3)	1·39 (1)	Cl–O(6)	1·405 (9)
C(4)–C(5)	1·41 (1)		

N(3)–Pt–N(7)	90·6 (3)	O(2)–C(2)–N(3)	120·4 (8)
N(3)–C(4)–N(4)	119·2 (9)	O(3)–Cl–O(5)	108·9 (5)
C(4)–C(5)–C(6)	118·8 (8)	O(4)–Cl–O(6)	110·3 (5)
C(6)–N(1)–C(2)	121·9 (7)	Pt–N(3)–C(4)	123·3 (6)
N(1)–C(2)–N(3)	118·2 (7)	N(4)–C(4)–C(5)	120·2 (9)
O(3)–Cl–O(4)	108·6 (4)	C(6)–N(1)–C(1)	120·3 (8)
O(4)–Cl–O(5)	109·5 (4)	N(1)–C(2)–O(2)	121·5 (8)
Pt–N(3)–C(2)	116·5 (5)	C(2)–N(3)–C(4)	119·9 (7)
N(3)–C(4)–C(5)	120·5 (8)	O(3)–Cl–O(6)	110·2 (5)
C(5)–C(6)–N(1)	120·7 (9)	O(5)–Cl–O(6)	109·4 (5)
C(1)–N(1)–C(2)	117·6 (7)		

Possible hydrogen-bond distances (Å) and angles (°)

A–H···*B*	*A*···*B* (Å)	*A*–H (Å)	H···*B* (Å)	*A*–H···*B* (°)
N(7)–H(71)···O(2^{i})	3·02 (1)	0·9 (1)	2·1 (1)	160 (10)
N(7)–H(73)···O(3)	3·11 (1)	0·86 (9)	2·27 (9)	168 (8)
N(7)–H(72)···O(5^{ii})	3·04 (1)	0·90 (9)	2·3 (1)	137 (8)
N(4)–H(41)···O(4)	2·99 (1)	0·90 (6)	2·50 (7)	114 (5)

Atoms are related to those given in Table 1 as follows: (i) $1-x, 1-y, -z$; (ii) $\tfrac{1}{2}-x, \tfrac{1}{2}+y, \tfrac{1}{2}-z$.

Schollhorn, Thewalt, Raudaschl-Sieber & Lippert, 1986).

The packing of the title compound (Fig. 2) is quite different from that in the nitrate salt. In the latter, π–π interactions between stacks of nitrate anions and pyrimidine rings, reinforced by $NH_3\cdots NO_3^-$ hydrogen bonds, meant that intercation ring–ring dihedral angles were small. No such constraints are imposed by the tetrahedral perchlorate ions and the dihedral angles between rings in molecules related by the 2_1 axis are 40·7 (3)°. The cations and anions form hydrogen-bonded layers at $x = 0, \tfrac{1}{2}$. Within the layers perchlorate ions are hydrogen-bonded to one cation through N(7)···O(3) and N(7)···O(5) and to its *b*-translated neighbour through N(4)···O(4). In the **c** direction translationally equivalent cations are hydrogen bonded through N(7)···O(2). In the **a** direction interactions between layers are primarily van der Waals. The only oxygen atom of the perchlorate group not involved in hydrogen bonding, O(6), has a considerably larger temperature factor than the other oxygen atoms.

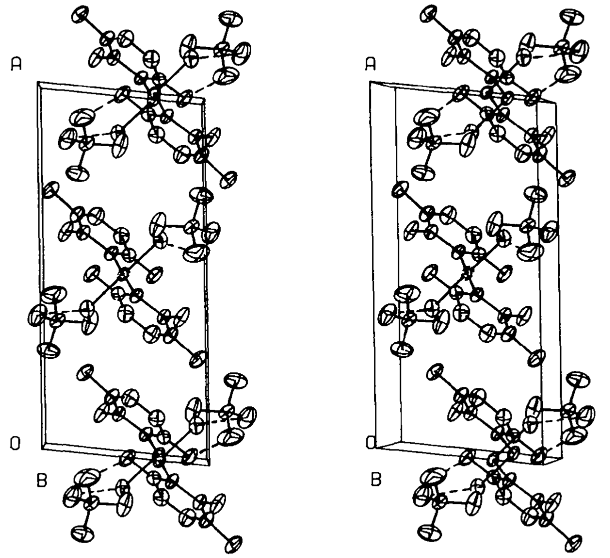

Fig. 2. The packing of the title compound within the unit cell. **a** and **c*** are parallel to the side and bottom of the page, respectively, and the view is down **b**. Hydrogen bonds are indicated by broken lines.

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Structure of a New Polymorphic Form of Tris(cyclopentadienyl)lanthanum(III)

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Abstract. $[La(C_5H_5)_3]$, $M_r = 334 \cdot 20$, monoclinic, $P2_1/c$, $a = 15 \cdot 237$ (2), $b = 9 \cdot 790$ (2), $c = 16 \cdot 721$ (4) Å, $\beta = 93 \cdot 93$ (3)°, $V = 2489$ (2) Å^3, $Z = 8$, $D_x = 1 \cdot 784$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71073 Å, $\mu = 3 \cdot 4171$ mm^{-1}, $F(000) = 1296$, $T = 295$ (1) K, $R = 0 \cdot 028$ for 2129 observed reflections. Each La atom is η^5 bonded to three cyclopentadienide rings and η^1 bonded to a ring of an adjacent $(C_5H_5)_3La$ molecule. This sharing of one C atom between the molecular units produces zigzag polymeric chains along the *b* axis of the unit cell.

Introduction. Molecular and crystal structures of a number of tris(cyclopentadienyl) complexes of the lanthanoids, $(C_5H_5)_3Ln$, have been reported in the literature. They show various polymeric arrangements of $(C_5H_5)_3Ln$ units. The first (rather inaccurate) structure of $(C_5H_5)_3Sm$ (Wong, Lee & Lee, 1969) revealed a complex polymeric chain structure in which each metal ion forms contacts with four disordered rings. $(C_5H_5)_3Pr$ (Hinrichs, Melzer, Rehwoldt, Jahn & Fischer, 1983) also exhibits an infinite chain arrangement but each Pr ion is η^5 coordinated to three close-lying C_5H_5 ligands and η^2 coordinated to a fourth bridging C_5H_5 ligand. The La derivative (Eggers, Kopf & Fischer, 1986) was found to be isotypic with $(C_5H_5)_3Pr$. The structures of $(C_5H_5)_3Er$ and its isotype $(C_5H_5)_3Tm$ (Eggers, Hinrichs, Kopf, Jahn & Fischer, 1986) in contrast shows molecules containing just three η^5 C_5H_5 ligands bonded to the metal ion.

On the other hand, $(C_5H_5)_3Lu$, which is isotypic with $(C_5H_5)_3Sc$ (Eggers, Schultze, Kopf & Fischer, 1986), shows an infinite chain arrangement in which each Lu atom is bounded in a pentahapto fashion to two rings and in a monohapto fashion to two other bridging rings.

A novel experimental technique of preparation allowed us to obtain single crystals of a polymorphic variety of $(C_5H_5)_3La$. Its characterization by X-ray diffraction is reported here.

Experimental. Tris(cyclopentadienyl)lanthanum was prepared under argon atmosphere, by reaction of (cyclopentadienyl)potassium with lanthanum trichloride in tetrahydrofuran. The THF adduct was removed under controlled conditions [10^{-4} torr ($\sim 10^{-2}$ Pa), 363 K]. Colourless prismatic single crystals obtained by extraction with *n*-pentane followed by recrystallization. Crystals sealed in thin-walled glass capillaries under argon. The specimen selected for X-ray analysis was 0·25 × 0·25 × 0·30 mm.

Intensity data recorded on an Enraf–Nonius CAD-4 X-ray diffractometer, graphite-monochromated Mo $K\bar{\alpha}$ radiation. Cell parameters refined by least squares from angle data of 25 reflections. Space group unambiguously determined from systematic absences. 3632 unique reflections measured (θ–2θ scan mode) in range

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