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Bis[μ-3-(N'-methyl-4,4′-bipyridinium-1-yl)propionamidato]bis[*cis*-diammineplatinum(II)] hexaperchlorate dihydrate: a head-to-tail isomer

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In the title compound, $[Pt_2(\mu\text{-}C_{14}H_{16}N_3O)_2(NH_3)_4](ClO_4)_6\cdot 2H_2O$, the diplatinum(II) cation is found to be a head-to-tail isomer. The intradimer Pt—Pt distance [3.0304 (7) Å] is much shorter than the value of 3.0852 (13) Å reported for the analogous *cis*-diammineplatinum(II) dimer bridged by 2-(N'-methyl-4,4′-bipyridinium-1-yl)acetamidates [Sakai, Ikuta, Tsubomura, Kato, Yokoyama, Kajiwara & Ito (2003). *Acta Cryst.* E**59**, m780–m783], showing that the electron density at the metal centers is higher in the title system than in the previously reported system. The shortest interdimer Pt···Pt distance is 8.4585 (8) Å.

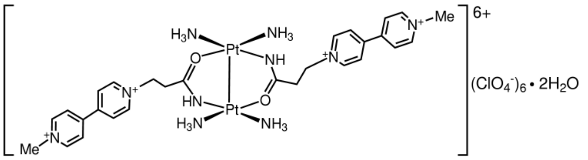
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.021 Å
Some non-H atoms missing
Disorder in solvent or counterion
R factor = 0.060
wR factor = 0.121
Data-to-parameter ratio = 18.5

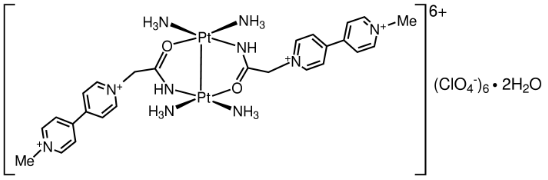
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

We previously reported that amidate-bridged *cis*-diammineplatinum(II) dimers, *viz.* $[Pt_2(NH_3)_4(\mu\text{-amidato})_2]^{2+}$ (amidate is acetamidate, α-pyrrolidinonate, α-pyridonate *etc.*), are generally active as H_2-evolving catalysts in a well known photosystem consisting of edta (usually, ethylenediaminetetraacetic acid disodium salt), $Ru(bpy)_3{}^{2+}$ (bpy is 2,2′-bipyridine), and methylviologen (usually, *N,N*′-dimethyl-4,4′-bipyridinium dichloride) (Sakai & Matsumoto, 1990; Sakai *et al.*, 1993). It should be noted here that two geometrical isomers, head-to-head (HH) and head-to-tail (HT), are possible for this class of doubly-bridged dimers, because of the asymmetric feature of amidate N—C—O units. In addition, edta, $Ru(bpy)_3{}^{2+}$ and methylviologen, respectively, serve as a sacrificial electron donor, a photosensitizer and an electron relay. One of our interests over many years has concentrated on the development of molecular devices which replace three major components, that is, $Ru(bpy)_3{}^{2+}$, methylviologen and the $Pt^{II}{}_2$ dimer. Such devices are considered as one-component systems. Considerable attention has also been paid to the studies on two-component systems in which the photochemical reduction of water into H_2 by edta is driven by $Ru(bpy)_3{}^{2+}$ and a hybrid compound made up of both the methylviologen and the H_2-evolving $Pt^{II}{}_2$ units. In this context, the title compound, HT-$[Pt_2(NH_3)_4(\mu\text{-}L1)_2](ClO_4)_6\cdot 2H_2O$ [(I); $L1$ is 3-(N'-methyl-4,4′-bipyridinium-1-yl)propionamidate], together with an anlogous dimer, HT-$[Pt_2(NH_3)_4(\mu\text{-}L2)_2](ClO_4)_6\cdot 2H_2O$ [(II); Sakai, Ikuta *et al.*, 2003; $L2$ is 2-(N'-methyl-4,4′-bipyridinium-1-yl)acetamidate], were first prepared in 1994 (Ikuta *et al.*, 1994), and were found to be somewhat effective. It has been confirmed that the H_2-evolving activity of (I), evaluated under the three-component system, is slightly higher than that of the acetamidate-bridged analog (Sakai *et al.*, 1993), and is *ca* 1.5 times higher than that of (II) (Sakai *et al.*, 1995). Importantly, preliminary experiments have shown that the photolysis of a solution containing edta, $Ru(bpy)_3{}^{2+}$, and either (I) or (II), in the absence of methylviologen, also results in evolution of H_2 in a relatively high quantum efficiency (Sakai *et al.*, 1995). Nevertheless, it took a long time to refine the reliable synthetic methods yielding (I), (II) and the related complexes (Sakai, Shiomi *et al.*, 2003; Sakai, Ikuta *et al.*, 2003). We now report the synthesis and crystal structure of (I).



(I)



(II)

A dinuclear $\mathrm{Pt^{II}}_2$ cation, six perchlorate anions and apparently 1.5 molecules of water are found in the asymmetric unit of (I). As previously reported for several analogous dimers doubly bridged by chain amidates, the binding directions of two bridging amidates could be rationally determined (see *Experimental*). As a result, the complex has been shown to be a head-to-tail isomer in the crystal structure, even though the complex is likely to give a mixture of both the HH and the HT isomer in solution, as previously observed for the related amidate-bridged $\mathrm{Pt^{II}}_2$ dimers (see, for example, Sakai, Tanaka *et al.*, 1998).

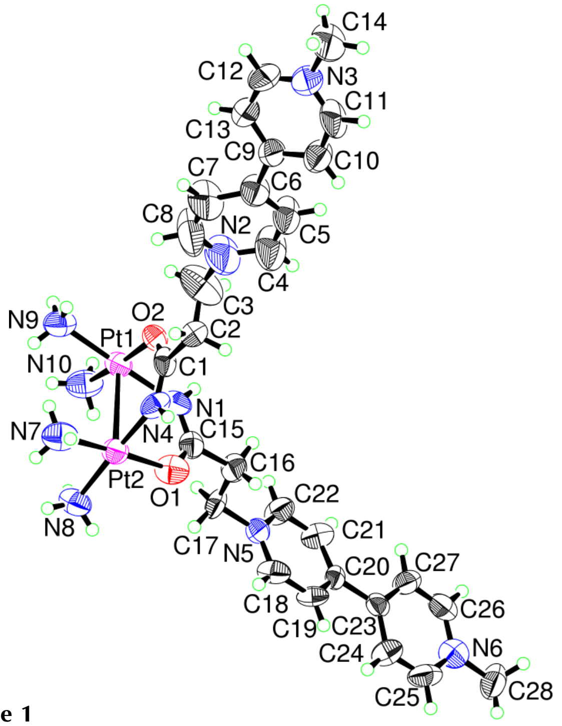


Figure 1
The structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. Perchlorate ions and water molecules have been omitted for clarity.

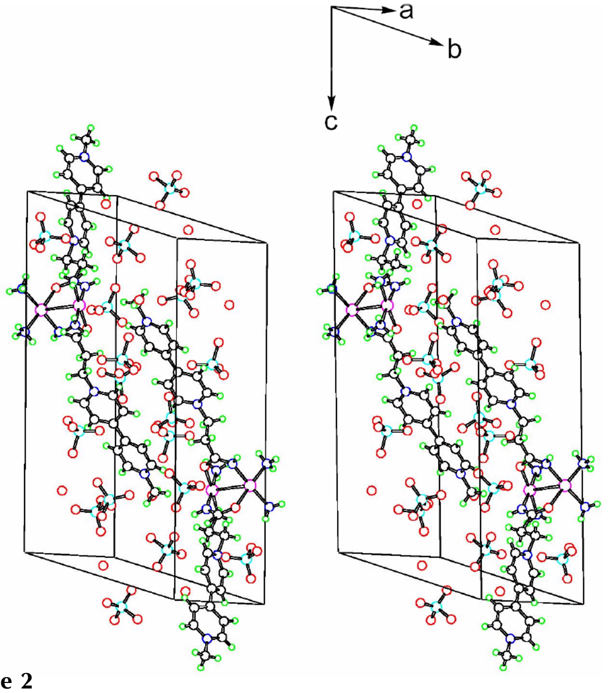


Figure 2
A stereoview for the crystal packing of (I), with atoms represented by spheres of arbitrary sizes. The minor disorder components of the perchlorate ions have been omitted for clarity.

The intradimer Pt—Pt distance in (I) [3.0304 (7) Å] is much shorter than that reported for (II) [3.0852 (13) Å] (Sakai, Ikuta *et al.*, 2003). The considerably long $\mathrm{Pt^{II}}$—$\mathrm{Pt^{II}}$ distance in (II) was previously interpreted in terms of the fact that the positively charged bipyridinium moieties in (II) serve as electron-withdrawing groups to diminish the electron density at the $\mathrm{Pt^{II}}$ centers, leading to the weakening of the 'mutual dative bonds' within the diplatinum cation [two $\mathrm{Pt^{II}}$ centers in close proximity are considered to form a dative $\mathrm{Pt}(5d_{z^2}) \rightarrow \mathrm{Pt}(6p$ or $6s)$ bond with one another; Connick *et al.*, 1997]. The shorter Pt—Pt distance in (I) is consistent with the fact that the bipyridinium moieties in (I) serve as poorer electron-withdrawing groups, because the distance between the bipyridinium unit and the $\mathrm{Pt^{II}}_2$ core in (I) is longer than that in (II). The N(bipyridinium)—C(bridging amidate) distances in (I) [N2—C1 = 3.831 (18) Å and N5—C15 = 3.781 (17) Å] are *ca* 1.4 Å longer than those reported for (II) [2.41 (3) and 2.46 (3) Å; Sakai, Ikuta *et al.* 2003], where the N(bipyridinium) atom denotes the N atom of a viologen unit which is closer to the diplatinum entity.

The dihedral angle between the two Pt coordination planes within the dimeric unit (τ) and the average torsional twist of them about the Pt—Pt axis (ω) are estimated as τ = 37.8 (3)° and ω = 3.8 (9)° (see also Table 1), where ω = 0° denotes that the two Pt coordination planes stack in an eclipsed fashion. The corresponding values were reported as τ = 40.8 (5)° and ω = 5 (1)° for (II) (Sakai, Ikuta *et al.*, 2003). Atoms Pt1 and Pt2

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are shifted out of their coordination planes by 0.073 (5) and 0.065 (5) Å, respectively, in such a manner that they have an attractive interaction with one another.

The pyridinium plane directly attached to the propionamidate unit is inclined with respect to the bridging O—C—N unit by 89.8 (7)° for N2/C4–C8 and 6.8 (24)° for N5/C18–C22. The twist angles of the two pyridinium planes within the individual bipyridinium moieties are 32.0 (7)° for the bipyridinium involving N2 and N3, and 16.2 (6)° for that involving N5 and N6. As previously discussed for (II) (Sakai, Ikuta *et al.*, 2003), these values are both effectively larger than that observed for the uncoordinated ligand *L*2H (a protonated form of *L*2) [6.1 (2)°; Sakai *et al.*, 1997], implying that part of the electron density at the $\mathrm{Pt^{II}}_2$ core is shifted towards the π*(bipyridinium) orbitals, giving rise to the lower aromaticity at the central C—C bonds of bipyridinium units. It should be also noted that the relatively large twist of 32.0 (7)° observed for the bipyridinium unit involving N2 and N3 is, in part, due to the strain enhanced by the intermolecular electrostatic interactions between the anions and the cations.

Fig. 2 shows a crystal packing view of (I). The shortest intermolecular Pt···Pt distance is observed as Pt1···Pt2(x, $y - 1$, z) = 8.4585 (8) Å (Table 1), confirming the lack of any intermolecular Pt—Pt interaction in the crystal structure. The crystal packing is stabilized both by the electrostatic interactions between the complex cations and perchlorate anions and by hydrogen bonds formed between the ammines and the O atoms of perchlorates as well as those between the water molecules and the O atoms of perchlorates (Tables 2 and 3).

Experimental

N-Methyl-4,4′-bipyridinium iodide was prepared according to the literature method of van Emon *et al.* (1986). *N*-(2-Carbamoylethyl)-*N*′-methyl-4,4′-bipyridinium diperchlorate was prepared in the same manner as previously described for the synthesis of *N*-(carbamoylmethyl)-*N*′-methyl-4,4′-bipyridinium diperchlorate dihydrate (Sakai *et al.*, 1997). A solution of 3-bromopropionamide (10 mmol, 1.52 g) and *N*-methyl-4,4′-bipyridinium iodide (10 mmol, 2.98 g) in dry methanol (25 ml; dried over molecular sieve 4 A) was refluxed for 48 h. After the solution was cooled to room temperature, the resulting orange powder was collected by filtration and air-dried. The product was then converted into the chloride salt using anion exchange resin (Amberlite IRA-400 in the Cl form). The chloride salt was further treated with two equivalents of silver perchlorate in water in the dark at 333 K for 1 h, followed by removal of AgCl by filtration. The filtrate was evaporated at 313–323 K almost to dryness. (Caution: perchlorate salts are potentially explosive and must be handled with care, even though we never experienced any difficulty in handling this material.) To the oily solution was added an excess of ethanol to give the product as a colorless powder which was recrystallized from a water–ethanol mixture (1:7 *v*/*v*) (yield 72%). Analysis calculated for $\mathrm{C_{14}H_{17}Cl_2N_3O_9}$: C 38.03, H 3.87, N 9.50%; found: C 38.09, H 3.83, N 9.34%. ^{1}H NMR ($\mathrm{D_2O}$, 296 K): δ 3.17 (*t*, 2H, *J* = 6.3 Hz), 4.51 (*s*, 3H), 5.02 (*t*, 2H, *J* = 6.3 Hz), 8.53 (*d*, 2H, *J* = 7.1 Hz), 8.56 (*d*, 2H, *J* = 6.8 Hz), 9.07 (*d*, 2H, *J* = 6.6 Hz), 9.17 (*d*, 2H, *J* = 7.1 Hz).

Compound (I) was prepared as follows: to an aqueous solution of *cis*-$\mathrm{[Pt(NH_3)_2(OH_2)_2](ClO_4)_2}$ (0.2 mmol Pt/2 ml $\mathrm{H_2O}$), prepared as previously described (Sakai, Takeshita *et al.*, 1998), was added *N*-(2-carbamoylethyl)-*N*′-methyl-4,4′-bipyridinium diperchlorate (0.32 mmol). The solution was heated at 343 K for 4 h followed by addition of a solution of $\mathrm{NaClO_4}$ (6.0 mmol) in water (0.35 ml). After the solution was filtered for the removal of insoluble materials, the filtrate was left to stand at room temperature for a few days. The yellow needles deposited were collected by filtration and air-dried (yield 15%). Analysis calculated for $\mathrm{C_{28}H_{48}Cl_6N_{10}O_{28}Pt_2}$: C 21.34, H 3.07, N 8.89%; found: C 21.47, H 2.60, N 8.55%. ^{1}H NMR ($\mathrm{D_2O}$, 296 K): δ 3.10–3.16 (*m*, 2H), 4.50 (*s*, 3H), 8.51–8.57 (*m*, 4H), 9.05–9.16 (*m*, 4H), where the multiplet (2H) derived from the methylene unit attached to the bipyridinium unit overlapped the solvent signal (4.8–4.9 p.p.m.). Although only 1.5 molecules of water could be located in the formula unit, we tentatively assumed that this compound is a dihydrate product, and this is reflected in the *Crystal data* as presented. The crystals of (I) are stable in air at room temperature. A diffraction-quality single crystal was mounted on a glass fibre with epoxy resin.

Crystal data

$\mathrm{[Pt_2(C_{14}H_{16}N_3O)_2(NH_3)_4]}$-$\mathrm{(ClO_4)_6 \cdot 2H_2O}$
$M_r = 1575.64$
Triclinic, $P\overline{1}$
$a = 10.0927$ (5) Å
$b = 11.0827$ (5) Å
$c = 24.4798$ (10) Å
$\alpha = 78.116$ (2)°
$\beta = 79.305$ (2)°
$\gamma = 74.210$ (2)°
$V = 2553.9$ (2) Å^3
$Z = 2$
$D_x = 2.049$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2521 reflections
$\theta = 1.9$–27.5°
$\mu = 5.88$ mm^{-1}
$T = 296$ (2) K
Needle, yellow
0.12 × 0.04 × 0.03 mm

Data collection

Bruker SMART APEX CCD-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.666$, $T_{\max} = 0.831$
24 579 measured reflections
11 715 independent reflections
3519 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.122$
$\theta_{\max} = 27.5°$
$h = -12 \rightarrow 13$
$k = -13 \rightarrow 14$
$l = -25 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.121$
$S = 0.79$
11715 reflections
633 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0288P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.82$ e Å^{-3}
$\Delta\rho_{\min} = -0.82$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Pt1—O2	2.041 (8)	Pt2—N4	1.985 (9)
Pt1—N1	2.007 (11)	Pt2—N7	2.009 (10)
Pt1—N9	2.038 (10)	Pt2—N8	2.057 (8)
Pt1—N10	2.021 (9)	Pt1—Pt2	3.0304 (7)
Pt2—O1	1.997 (9)	Pt1—Pt2$^{\rm i}$	8.4585 (8)
N1—Pt1—N10	88.3 (4)	N4—Pt2—O1	92.1 (4)
N10—Pt1—N9	90.2 (4)	N4—Pt2—N7	87.6 (4)
N1—Pt1—O2	92.5 (4)	O1—Pt2—N8	87.5 (4)
N9—Pt1—O2	88.8 (4)	N7—Pt2—N8	92.6 (4)
N1—Pt1—N9	177.4 (4)	O1—Pt2—N7	175.9 (4)
N10—Pt1—O2	173.9 (3)	N4—Pt2—N8	176.7 (4)
O2—Pt1—Pt2—N4	−3.8 (4)	N9—Pt1—Pt2—N7	−5.0 (4)
N1—Pt1—Pt2—O1	−3.0 (4)	N10—Pt1—Pt2—N8	−3.3 (4)

Symmetry code: (i) x, $y - 1$, z.

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Table 2
Contact distances (Å).

O8*B*···O28	3.17 (3)	O27···O3$^{\rm ii}$	3.10 (6)
O22*A*···O27	2.74 (4)	O28···O7*B*$^{\rm iii}$	3.07 (3)
O22*B*···O27	3.17 (5)	O28···O7*A*$^{\rm iii}$	3.19 (5)

Symmetry codes: (ii) $1 - x, -y, 1 - z$; (iii) $1 - x, 1 - y, -z$.

Table 3
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H7*C*···O13*B*$^{\rm iv}$	0.89	2.64	2.94 (3)	101
N8—H8*C*···O26$^{\rm v}$	0.89	2.28	3.159 (15)	170
N8—H8*A*···O21*A*$^{\rm vi}$	0.89	2.06	2.92 (2)	161
N8—H8*B*···O13*A*$^{\rm iv}$	0.89	2.48	3.003 (18)	118
N8—H8*B*···O16*B*$^{\rm iv}$	0.89	2.20	3.01 (2)	150
N8—H8*B*···O18*A*$^{\rm iv}$	0.89	2.34	3.155 (15)	152
N9—H9*A*···O22*A*$^{\rm ii}$	0.89	2.29	3.155 (18)	164
N9—H9*C*···O13*B*$^{\rm iv}$	0.89	2.42	3.22 (3)	150
N9—H9*C*···O14*A*$^{\rm iv}$	0.89	2.49	3.148 (18)	132
N10—H10*A*···O18*B*$^{\rm ii}$	0.89	2.49	3.00 (3)	117
N10—H10*A*···O20*A*$^{\rm ii}$	0.89	2.31	2.857 (18)	120
N10—H10*A*···O20*B*$^{\rm ii}$	0.89	2.30	3.04 (3)	141
N10—H10*C*···O18*A*$^{\rm iv}$	0.89	2.36	3.155 (15)	149
N10—H10*C*···O18*B*$^{\rm iv}$	0.89	2.28	3.00 (3)	137
N4—H4*A*···O9*A*	0.86	2.20	2.96 (3)	147
N7—H7*B*···O10*B*	0.89	2.22	3.09 (2)	165
N7—H7*C*···O25$^{\rm v}$	0.89	2.59	3.151 (14)	122
N7—H7*C*···O26$^{\rm v}$	0.89	2.15	3.042 (14)	177

Symmetry codes: (ii) $1 - x, -y, 1 - z$; (iv) $x - 1, y, z$; (v) $-x, 1 - y, 1 - z$; (vi) $1 - x, 1 - y, 1 - z$.

As recently reported for several different dimers doubly bridged by chain amidate ligands (Sakai, Kurashima *et al.*, 2003; Sakai, Ikuta *et al.*, 2003; Sakai, Shiomi *et al.*, 2003), the binding directions of O and NH for the amidate ligands were rationally determined by the results of least-squares calculations performed for two possible arrangements for each ligand as follows. A wrong combination gave an asymmetric feature with regard to the equivalent displacement parameters of the assumed O and N atoms, while an appropriate combination gave a moderate balance in these values. For O1 and N1, $U_{\rm eq}$(O1) = 0.072 (3) Å^2 and $U_{\rm eq}$(N1) = 0.060 (3) Å^2 were judged to be correct, while the reverse selection gave values of $U_{\rm eq}$(N instead of O1) = 0.047 (3) Å^2 and $U_{\rm eq}$(O instead of N1) = 0.091 (3) Å^2. For O2 and N4, $U_{\rm eq}$(O2) = 0.056 (3) Å^2 and $U_{\rm eq}$(N4) = 0.058 (3) Å^2 were judged to be correct, while the reverse selection gave values of $U_{\rm eq}$(N instead of O2) = 0.034 (3) Å^2 and $U_{\rm eq}$(O instead of N4) = 0.089 (3) Å^2.

Four of the six ClO_4^- anions show orientational disorder. Around each Cl atom there are two sets of possible positions as follows: O7*A*/O8*A*/O9*A*/O10*A* and O7*B*/O8*B*/O9*B*/O10*B* around Cl2; O11*A*/O12*A*/O13*A*/O14*A* and O11*B*/O12*B*/O13*B*/O14*B* around Cl3; O15*A*/O16*A*/O17*A*/O18*A* and O15*B*/O16*B*/O17*B*/O18*B* around Cl4; and O19*A*/O20*A*/O21*A*/O22*A* and O19*B*/O20*B*/O21*B*/O22*B* around Cl5. It was assumed that the disordered O atoms around each Cl atom have the same isotropic displacement parameter. Furthermore, Cl—O distances were restrained at 1.43 (3) Å and the six O···O distances within each perchlorate anion were restrained as equal. The occupancy factors of sites *A* and *B* (sof$_A$ and sof$_B$) around each Cl converged to the following values: sof$_A$(Cl2) = 0.33 (2) and sof$_B$(Cl2) = 0.67 (2); sof$_A$(Cl3) = 0.685 (9) and sof$_B$(Cl3) = 0.315 (9); sof$_A$(Cl4) = 0.702 (8) and sof$_B$(Cl4) = 0.298 (8); and sof$_A$(Cl5) = 0.64 (2) and sof$_B$(Cl5) = 0.36 (2).

All H atoms, except those of water molecules, were placed at idealized positions [C—H(methyl) = 0.96 Å, C—H(aromatic) = 0.93 Å and N—H(ammine) = 0.89 Å] and included in the refinement in the riding-model approximation, with $U_{\rm iso}$(methyl H) = $1.5U_{\rm eq}$(bonded C), $U_{\rm iso}$(aromatic H) = $1.2U_{\rm eq}$(bonded C) and $U_{\rm iso}$(ammine H) = $1.5U_{\rm eq}$(bonded N). The highest peak was located 0.39 Å from atom O11*A*, while the deepest hole was located 0.45 Å from Pt1. Water H atoms were not located.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *KENX* (Sakai, 2002); software used to prepare material for publication: *SHELXL*97, *TEXSAN* (Molecular Structure Corporation, 2001), *KENX* and *ORTEP*II (Johnson, 1976).

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