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N-(Carbamoylmethyl)pyridinium Perchlorate and N-(Carbamoylmethyl)-N'-methyl-4,4'-bipyridinium Diperchlorate Hydrate

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## Abstract

The title compounds, $C_7H_9N_2O^+.ClO_4^-$, (1), and $C_{13}H_{15}N_3O^{2+}.2ClO_4^-.H_2O$, (2), have been synthesized and their crystal structures determined. The C—N(carbamoyl) bond in each compound has some double-bond character [bond lengths 1.319 (5) for (1) and 1.325 (7) Å for (2)], indicating that the sp^2 hybridization is delocalized over the O—C—N(carbamoyl) unit. The carbamoylmethyl plane is canted with respect to the attached pyridinium plane at an angle of 63.5 (2) for

(1) and 79.1 (2)° for (2). Two major conformers have been found in the molecular mechanics calculations performed on the N-carbamoylmethylpyridinium cation.

Comment

We have recently shown that the amidate-bridged *cis*-diammineplatinum(II) dimers, $[Pt_2^{II}(NH_3)_4(\mu\text{-amidato-}N,O)_2]^{2+}$ (amidato is acetamidato, *etc.*), are generally efficient as H_2-producing catalysts in a well known photosystem consisting of edta, tris(2,2'-bipyridine)-ruthenium(II) and methyl viologen (N,N'-dimethyl-4,4'-bipyridinium salt) (Sakai, Kizaki, Tsubomura & Matsumoto, 1993). In this system, the methyl viologen cation serves as an electron acceptor which oxidatively quenches the excited state of the ruthenium complex. The resulting radical cation reduces water to molecular hydrogen in the presence of a suitable catalyst such as colloidal platinum or the platinum(II) dimers mentioned above. From the standpoint of so-called 'supramolecular chemistry', it is worth developing systems in which more than two components of the above essential chemical species are combined into a single bifunctional molecule in an appropriate spatial arrangement. With the aim of obtaining new platinum dimers covalently attached to pyridinium moieties, with quenching properties similar to those of methyl viologen, the title amides, (1) and (2), have been prepared and characterized. Other important similar examples may be the N-carboxyalkyl-N'-methyl-4,4'-bipyridinium compounds reported by Willner *et al.* (1994).

(1)

(2)
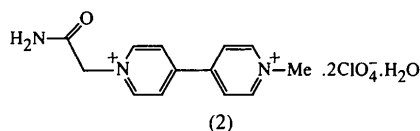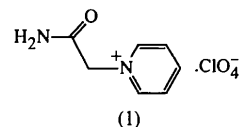
The crystal structures of (1) and (2) are shown in Figs. 1 and 2, respectively. The carbamoylmethyl plane is canted with respect to the attached pyridinium plane at an angle of 63.5 (2) for (1) and 79.1 (2)° for (2). Both the carbamoylmethyl and pyridinium moieties are close to being planar with the four- and six-atom r.m.s. deviations less than 0.006 Å. It is notable that the orientation of the carbamoyl unit in (1) is very similar to that in (2), suggesting that this conformation is the thermodynamically favourable one. Perhaps this is due to the electrostatic attraction between the positively charged pyridinium N atom and the negatively charged carbamoyl O atom, however, it must be noted that this conformation also minimizes steric interactions between the O(1) atom and the *ortho*-H atoms [H1 and H5 in (1)],

and H5 and H8 in (2)], which would be severe had the carbamoyl moiety been coplanar with the pyridinium ring. The two pyridinium planes in (2) are slightly twisted towards one another at an angle of 6.1 (2)°, which can be explained in terms of the steric repulsions among the H atoms riding on the 3-, 3'-, 5- and 5'-positions of the 4,4'-bipyridinium moiety.
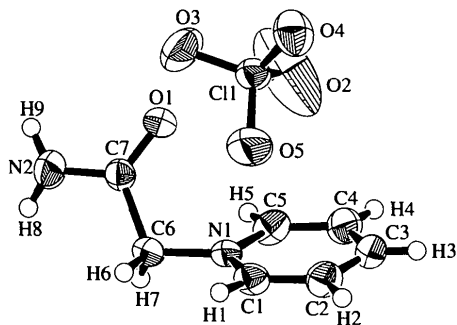

Fig. 1. The structure of (1) showing the atom-labeling scheme. Displacement ellipsoids are shown at the 50% probability level.
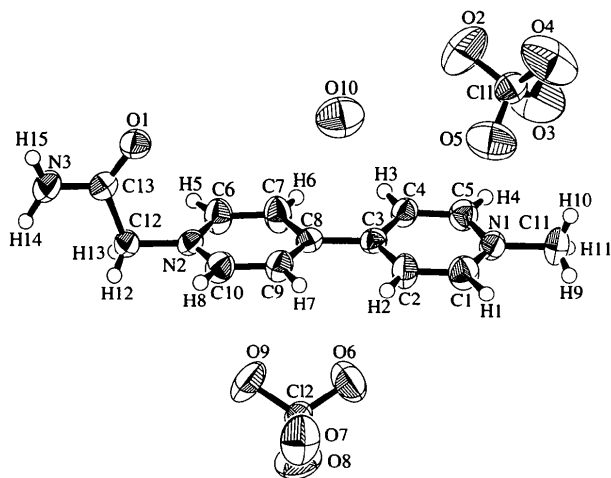

Fig. 2. The structure of (2) showing the atom-labeling scheme. Displacement ellipsoids are shown at the 50% probability level.

The conformation of the cation can be expressed by two variables α and β corresponding to the torsion angles C(1)—N(1)—C(6)—C(7) and N(1)—C(6)—C(7)—O(1), respectively. Geometry optimizations using the HyperMM+ program (Hypercube Inc, 1995) were performed starting with various possible conformations and only two energy-minimized structures were found. One has a symmetric structure which is essentially the same as that observed in the crystal of (1) (energy = 11.55 kcal mol^{-1}, α = 90 and β = 0°). The other has a relatively asymmetric structure but has a slightly lower energy (energy = 11.10 kcal mol^{-1}, α = 77 and β = −117°). At α = 90°, there are distinct minima at β =

0, 120, 240 and 360°. Conformations with α smaller than 77° have been found to possess a relatively high energy, which is clearly due to the steric repulsions between the carbamoyl unit and the H atoms on the 2- and 6-positions of the pyridyl ring mentioned above. The crystallographically-observed conformation can thus be regarded as one of the two possible energy-minimized structures, although it is slightly distorted. Very similar energy-minimized structures have also been found for the dication of (2) (21.17 kcal mol^{-1}, α = 90 and β = 0°; 20.71 kcal mol^{-1}, α = 77 and β = −118°; the twist angle between the two pyridyl planes is less than 0.5° in both cases).

The amidate-bridged cis-diammineplatinum(II) dimers of (1) and (2) can be prepared from reactions of (1) and (2) with cis-$[Pt(NH_3)_2(OH_2)_2]^{2+}$ in aqueous media. The complex of (2) has been found to show an efficient catalytic function in the photosystem mentioned above. The details will be reported elsewhere.

Experimental

A solution of 2-bromoacetamide (10 mmol, 1.38 g) and pyridine (10 mmol, 0.80 g) in dry ethanol (25 ml) was stirred at room temperature for 24 h. This resulted in deposition of a white powder of N-carbamoylmethylpyridinium bromide which was collected by filtration and air dried (yield: 75%). Analysis calculated for $C_7H_9BrN_2O$: C 38.73, H 4.18, N 12.91, Br 36.81%; found: C 38.83, H 4.21, N 12.98, Br 36.81%. The bromide salt and one equivalent of $AgClO_4$ were dissolved in a minimum amount of water. The mixture was stirred at 333 K for 1 h followed by filtration to remove AgBr. The filtrate was evaporated at 313–323 K almost to dryness. The residue was redissolved in a minimum amount of hot methanol (323 K) followed by filtration while hot to remove insoluble materials. The filtrate was allowed to stand in air at room temperature and its volume was gradually concentrated to afford colorless needles of (1) (yield: 53%). Analysis calculated for $C_7H_9ClN_2O_5$: C 35.53, H 3.83, N 11.84%; found: C 35.33, H 3.59, N 11.90%. ^{1}H NMR (D_2O, 296 K; Jeol JNM-GX270): δ 5.52 (s, 2H), 8.13 (t, 2H, J = 7.2 Hz), 8.65 (t, 1H, J = 8.0 Hz), 8.81 p.p.m. (d, 2H, J = 5.4 Hz).

A solution of bromomethane (23 mmol, 2.18 g) in dry ethanol (10 ml) was added to a solution of 4,4'-bipyridine (69 mmol, 10.8 g) in dry ethanol (50 ml). The solution was stirred at room temperature for 24 h to give a pale yellow precipitate of N-methyl-4,4'-bipyridinium bromide hemihydrate. After adding a large excess of ether, the powder deposited was collected by filtration and was dissolved in a minimum amount of ethanol at 333 K followed by filtration to remove insoluble materials. A large excess of ether was again added to the filtrate to reprecipitate the product (yield: 70–80%). Analysis calculated for $C_{11}H_{12}BrN_2O_{0.5}$: C 50.79, H 4.65, N 10.77, Br 30.72%; found: C 50.95, H 4.65, N 10.75, Br 31.81%. ^{1}H NMR (D_2O, 296 K): δ 4.43 (s, 3H), 7.90 (d, 2H, J = 6.5 Hz), 8.38 (d, 2H, J = 6.5 Hz), 8.77 (d, 2H, J = 6.5 Hz), 8.89 p.p.m. (d, 2H, J = 6.5 Hz).

A solution of 2-bromoacetamide (10 mmol, 1.38 g) and N-methyl-4,4'-bipyridinium bromide hemihydrate (10 mmol, 2.60 g) in dry methanol (25 ml) was stirred at room tem-

perature for 24 h, resulting in deposition of a pale yellow powder of *N*-carbamoylmethyl-*N'*-methyl-4,4'-bipyridinium dibromide hydrate, which was collected by filtration and air dried (yield: 73%). Analysis calculated for $C_{13}H_{17}Br_2N_3O_2$: C 38.35, H 4.21, N 10.32, Br 9.25%; found: C 38.47, H 4.27, N 10.48, Br 38.60%. The bromide salt and two equivalents of $AgClO_4$ were dissolved in a minimum amount of water. The mixture was stirred at 333 K for 1 h followed by filtration to remove the AgBr precipitated. The filtrate was evaporated at 313–323 K almost to dryness. The residue was redissolved in a minimum amount of water/ethanol (1:7) at 323 K followed by filtration while hot to remove insoluble materials. The filtrate was allowed to stand in air at room temperature and its volume was gradually concentrated to afford colorless needles of the ligand in its hemihydrate form (yield: 40%). Analysis calculated for $C_{13}H_{15}Cl_2N_3O_9.0.5H_2O$: C 35.73, H 3.69, N 9.61%; found: C 35.94, H 3.41, N 9.70%. The monohydrate salt of (2) was obtained by recrystallizing the compound from hot water. Analysis calculated for $C_{13}H_{15}Cl_2N_3O_9.H_2O$: C 34.99, H 3.84, N 9.42%; found: C 35.26, H 3.58, N 9.48%. ^1H NMR (D_2O, 296 K): δ 4.51 (*s*, 3H), 5.64 (*s*, 2H), 8.55 (*d*, 2H, *J* = 6.6 Hz), 8.60 (*d*, 2H, *J* = 6.8 Hz), 9.07 p.p.m. (pseudo-*d*, 4H, *J* = 5.9 Hz). Crystals of (1) and (2) used in the X-ray measurements were obtained by recrystallizing them from hot water.

Compound (1)

Crystal data

$C_7H_9N_2O^+.ClO_4^-$
$M_r = 236.61$
Monoclinic
$P2_1/c$
$a = 5.352\,(2)$ Å
$b = 15.186\,(2)$ Å
$c = 12.013\,(1)$ Å
$\beta = 95.87\,(2)°$
$V = 971.2\,(4)$ Å^3
$Z = 4$
$D_x = 1.618$ Mg m^{-3}
D_m not measured

Cu *K*α radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 33.46–34.87°$
$\mu = 3.60$ mm^{-1}
$T = 296$ K
Prism
$0.50 \times 0.38 \times 0.38$ mm
Colorless

Data collection

Rigaku AFC-5*S* diffractometer
$\theta/2\theta$ scans
Absorption correction:
empirical *via* ψ scans
(North, Phillips &
Mathews, 1968)
$T_{min} = 0.19$, $T_{max} = 0.25$
1680 measured reflections
1502 independent reflections

1255 reflections with
$I > 3\sigma(I)$
$R_{int} = 0.0196$
$\theta_{max} = 60°$
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 17$
$l = -13 \rightarrow 13$
3 standard reflections
every 150 reflections
intensity decay: 3.17%

Refinement

Refinement on *F*
$R = 0.0559$
$wR = 0.0766$
$S = 2.90$
1255 reflections
173 parameters
All H atoms refined

$\Delta\rho_{max} = 0.59$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}
Extinction correction:
Zachariasen (1968) type
2, Gaussian isotropic
Extinction coefficient:
$8.8\,(5) \times 10^2$

$w = 1/[\sigma^2(F) + 0.0016F^2]$
$(\Delta/\sigma)_{max} = 0.0010$

Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °) for (1)*

Cl(1)—O(2)	1.377 (4)	N(1)—C(6)	1.467 (5)
Cl(1)—O(3)	1.404 (4)	N(2)—C(7)	1.319 (5)
Cl(1)—O(4)	1.421 (3)	C(1)—C(2)	1.363 (6)
Cl(1)—O(5)	1.407 (4)	C(2)—C(3)	1.368 (6)
O(1)—C(7)	1.208 (4)	C(3)—C(4)	1.372 (6)
N(1)—C(1)	1.343 (5)	C(4)—C(5)	1.363 (6)
N(1)—C(5)	1.345 (5)	C(6)—C(7)	1.521 (5)
O(2)—Cl(1)—O(3)	111.5 (4)	N(1)—C(1)—C(2)	120.3 (4)
O(2)—Cl(1)—O(4)	109.0 (3)	C(1)—C(2)—C(3)	119.6 (4)
O(2)—Cl(1)—O(5)	109.6 (4)	C(2)—C(3)—C(4)	119.7 (4)
O(3)—Cl(1)—O(4)	111.4 (2)	C(3)—C(4)—C(5)	119.2 (4)
O(3)—Cl(1)—O(5)	106.6 (3)	N(1)—C(5)—C(4)	120.5 (4)
O(4)—Cl(1)—O(5)	108.6 (3)	N(1)—C(6)—C(7)	111.7 (3)
C(1)—N(1)—C(5)	120.6 (4)	O(1)—C(7)—N(2)	123.9 (4)
C(1)—N(1)—C(6)	120.7 (3)	O(1)—C(7)—C(6)	121.6 (3)
C(5)—N(1)—C(6)	118.7 (3)	N(2)—C(7)—C(6)	114.5 (4)

Compound (2)

Crystal data

$C_{13}H_{15}N_3O^{2+}.2ClO_4^-.H_2O$
$M_r = 446.20$
Monoclinic
$P2_1/c$
$a = 11.528\,(3)$ Å
$b = 8.643\,(3)$ Å
$c = 19.141\,(2)$ Å
$\beta = 106.56\,(1)°$
$V = 1828.0\,(8)$ Å^3
$Z = 4$
$D_x = 1.621$ Mg m^{-3}
D_m not measured

Cu *K*α radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 32.98–35.00°$
$\mu = 3.768$ mm^{-1}
$T = 296$ K
Prism
$0.33 \times 0.33 \times 0.25$ mm
Colorless

Data collection

Rigaku AFC-5*S* diffractometer
$\theta/2\theta$ scans
Absorption correction:
empirical *via* ψ scans
(North, Phillips &
Mathews, 1968)
$T_{min} = 0.29$, $T_{max} = 0.39$
3095 measured reflections
2933 independent reflections

2016 reflections with
$I > 3\sigma(I)$
$R_{int} = 0.0539$
$\theta_{max} = 60°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 9$
$l = -21 \rightarrow 20$
3 standard reflections
every 150 reflections
intensity decay: 2.01%

Refinement

Refinement on *F*
$R = 0.0569$
$wR = 0.0624$
$S = 2.89$
2016 reflections
314 parameters
All H atoms refined
$w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{max} = 0.0040$

$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}
Extinction correction:
Zachariasen (1968) type
2, Gaussian isotropic
Extinction coefficient: 10 (3)
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 2. *Selected geometric parameters (Å, °) for (2)*

Cl(1)—O(2)	1.391 (5)	N(2)—C(10)	1.333 (7)
Cl(1)—O(3)	1.383 (5)	N(2)—C(12)	1.472 (6)
Cl(1)—O(4)	1.374 (5)	N(3)—C(13)	1.325 (7)

Cl(1)—O(5)	1.401 (4)	C(1)—C(2)	1.366 (7)
Cl(2)—O(6)	1.416 (4)	C(2)—C(3)	1.386 (7)
Cl(2)—O(7)	1.404 (4)	C(3)—C(4)	1.379 (7)
Cl(2)—O(8)	1.405 (4)	C(3)—C(8)	1.488 (7)
Cl(2)—O(9)	1.413 (4)	C(4)—C(5)	1.365 (7)
O(1)—C(13)	1.223 (6)	C(6)—C(7)	1.377 (7)
N(1)—C(1)	1.335 (7)	C(7)—C(8)	1.374 (7)
N(1)—C(5)	1.340 (7)	C(8)—C(9)	1.377 (7)
N(1)—C(11)	1.489 (7)	C(9)—C(10)	1.376 (7)
N(2)—C(6)	1.334 (7)	C(12)—C(13)	1.520 (7)
O(2)—Cl(1)—O(3)	105.3 (4)	N(1)—C(1)—C(2)	121.3 (6)
O(2)—Cl(1)—O(4)	109.4 (4)	C(1)—C(2)—C(3)	120.1 (5)
O(2)—Cl(1)—O(5)	109.9 (4)	C(2)—C(3)—C(4)	117.2 (5)
O(3)—Cl(1)—O(4)	110.4 (4)	C(2)—C(3)—C(8)	121.1 (4)
O(3)—Cl(1)—O(5)	110.7 (3)	C(4)—C(3)—C(8)	121.7 (4)
O(4)—Cl(1)—O(5)	111.0 (3)	C(3)—C(4)—C(5)	121.0 (5)
O(6)—Cl(2)—O(7)	110.0 (3)	N(1)—C(5)—C(4)	120.5 (6)
O(6)—Cl(2)—O(8)	108.0 (3)	N(2)—C(6)—C(7)	120.2 (6)
O(6)—Cl(2)—O(9)	110.5 (3)	C(6)—C(7)—C(8)	120.8 (6)
O(7)—Cl(2)—O(8)	109.2 (3)	C(3)—C(8)—C(7)	121.6 (5)
O(7)—Cl(2)—O(9)	110.9 (3)	C(3)—C(8)—C(9)	120.8 (4)
O(8)—Cl(2)—O(9)	108.2 (3)	C(7)—C(8)—C(9)	117.6 (5)
C(1)—N(1)—C(5)	120.1 (5)	C(8)—C(9)—C(10)	119.9 (6)
C(1)—N(1)—C(11)	120.5 (5)	N(2)—C(10)—C(9)	121.1 (5)
C(5)—N(1)—C(11)	119.3 (5)	N(2)—C(12)—C(13)	110.1 (4)
C(6)—N(2)—C(10)	120.3 (5)	O(1)—C(13)—N(3)	125.6 (5)
C(6)—N(2)—C(12)	119.6 (5)	O(1)—C(13)—C(12)	121.5 (5)
C(10)—N(2)—C(12)	120.1 (5)	N(3)—C(13)—C(12)	112.8 (5)

Data collection and cell refinement used *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994a). The structures were solved by the direct-methods program *SIR*88 (Burla *et al.*, 1989). Atoms were also located using *DIRDIF* (Parthasarathi, Beurskens & Slot, 1983) and difference Fourier techniques. All non-H atoms were refined anisotropically by full-matrix least squares. The H atoms of the solvate water molecule in (2) were not located. All the remaining H atoms were refined isotropically. The largest extinction corrections were for the 040 reflection in (1) of 0.530 and for $10\bar{4}$ in (2) of 0.952. Best-plane calculations were performed using *BP*70 (Ito, 1982). All other calculations were performed using *TEXSAN* (Molecular Structure Corporation, 1994b). Molecular mechanics calculations were performed using the *HyperMM+* program in *HyperChem* (Hypercube Inc., 1995), in which default parameters were used and atomic charges were taken into consideration to account for the electrostatic contribution.

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Hydrated Structures of *N*-Methylated Cholamide Derivatives

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Abstract

The crystal structures of *N*-methylcholamide quarter-hydrate (*N*-methyl-3α,7α,12α-trihydroxy-5β-cholan-24-amide 0.25-hydrate, $C_{25}H_{43}NO_4.0.25H_2O$), recrystallized from acetone by diffusion of water, *N*-methylcholamide sesquihydrate (*N*-methyl-3α,7α,12α-trihydroxy-5β-cholan-24-amide 1.5-hydrate, $C_{25}H_{43}NO_4.1.5H_2O$), recrystallized from a 10:1 ethyl acetate–absolute ethanol solution by slow evaporation, and *N*,*N*-dimethylcholamide hemihydrate (*N*,*N*-dimethyl-3α,7α,12α-trihydroxy-5β-cholan-24-amide 0.5-hydrate, $C_{26}H_{45}NO_4.0.5H_2O$), recrystallized from a 1:1 absolute methanol–ethyl acetate solution by slow evaporation, have been determined. These structures are the first reported crystal structures of both *N*-methylcholamide and *N*,*N*-dimethylcholamide. Each structure contains water molecules that sit on special positions. The *N*-methylcholamide sesquihydrate and *N*,*N*-dimethylcholamide hemihydrate forms are isostructural.

Comment

Steroids often display the ability to crystallize into multiple solvated forms (Byrn, 1982). One such example is the bile acid cholic acid (3α,7α,12α-trihydroxy-5β-cholan-24-oic acid) and its various derivatives, *e.g.* methyl cholate (3α,7α,12α-trihydroxy-5β-cholan-24-oic acid methyl ester), sodium cholate (sodium 3α,7α,12α-trihydroxy-5β-cholan-24-oate) and cholamide [3α,7α,12α-trihydroxy-5β-cholan-24-amide, (I)]. Four different solvates of cholamide are known (Sada, Kondo, Miyata & Miki, 1994; Sada, Kondo, Miyata,