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6,6′-Dimethyl-2,2′-[oxalylbis(azanediyl)]-dipyridinium dichloride acetonitrile solvate

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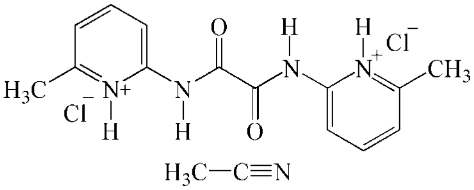
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.094; data-to-parameter ratio = 13.7.

In the crystal structure of the title compound, $C_{14}H_{16}N_4O_2^{2+}\cdot 2Cl^-\cdot CH_3CN$, weak intermolecular N—H⋯Cl hydrogen bonds are found between the H atoms bound to the pyridine and amine N atoms and the chloride anions. The asymmetric unit consits of one half cationic molecule which is located on a centre of inversion, one chloride anion in a general position and one half acetonitrile molecule which is located on a twofold axis. Because of symmetry, the C—H hydrogens of the acetonitrile solvent molecule are disordered over two orientations.

Related literature

For Ag(I) complexes incorporating *N*,*N*′-bis(2-pyridyl)oxamide ligands which show one- and two-dimensional structures, see: Hsu & Chen (2004); Hu *et al.* (2004). For the synthesis of the starting reactant, see: Cheng *et al.* (2009).



Experimental

Crystal data

$C_{14}H_{16}N_4O_2^{2+}\cdot 2Cl^-\cdot C_2H_3N$
M_r = 384.26
Monoclinic, $P2/c$
a = 10.6740 (19) Å
b = 8.7637 (5) Å
c = 10.370 (3) Å
β = 109.83 (2)°
V = 912.5 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 295 K
0.6 × 0.2 × 0.1 mm

Data collection

Bruker P4 diffractometer
Absorption correction: ψ scan (*XSCANS*; Siemens, 1995)
T_{min} = 0.823, T_{max} = 0.922
2165 measured reflections
1619 independent reflections
1308 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
3 standard reflections every 97 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.094
S = 1.07
1619 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1A⋯Cl^{i}	0.86	2.14	2.9772 (16)	165
N2—H2A⋯Cl^{i}	0.86	2.43	3.2057 (17)	150

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

Data collection: *XSCANS* (Siemens, 1995); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2195).

References

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1995). *XSCANS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supplementary materials

Acta Cryst. (2010). E**66**, o2386   [**doi:10.1107/S1600536810033519**]

6,6'-Dimethyl-2,2'-[oxalylbis(azanediyl)]dipyridinium dichloride acetonitrile solvate

H.-L. Hu, P.-C. Cheng, C.-J. Wu and J.-D. Chen

Comment

Several Ag(I) complexes containg *N*,*N'*-bis(2-pyridyl)oxamide ligands have been prepared, which show one-dimensional and two-dimensional structures (Hsu, *et al.*, 2004; Hu, *et al.*, 2004). To investigate the steric effect of the alkyl groups on the structural type of such complexes, we have synthesized *N*,*N'*-bis(6-methyl-2-pyridyl)oxamide (Cheng, *et al.*, 2009) and reacted with metal salts. Within this project the crystal structure of the title compound was determined.

In the crystal structure the cationic molecules are almost planar and the O atoms are trans-oriented (Fig. 1). The *N*,*N'*-Bis(6-methyl-2-pyridinium)oxamide cations and the chloride anions are connected by weak intermolecular N—H···Cl hydrogen bonding (Tab. 1).

Experimental

N,*N'*-bis(6-methyl-2-pyridyl)oxamide (0.30 g, 1.1 mmol) (Cheng, *et al.*, 2009) and $CuCl_2$ (0.15 g, 1.1 mmol) were placed in a flask containing 10 ml CH_2Cl_2, which was refluxed for 8 h. The precipitate was then filtered and dried in vacuum. Coloress plate crystals of the title compound suitable for X-ray crystallography were obtained by slow evaporization of the solvent from a solution of the precipitate in CH_3CN.

Refinement

All the hydrogen atoms were placed into idealized positions (methyl H atoms allowed to rotate but not to tip) and constrained by the riding atom approximation with *C*—H = 0.93 — 0.96 Å, N—H = 0.86 Å and U_{iso}(H) = 1.2 U_{eq}(C, N) (1.5 for methyl H atoms).

Figures

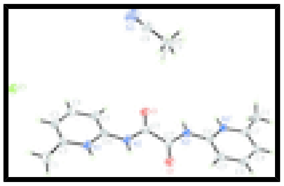


Fig. 1. Crystal structure of the title compound with labeling and displacement ellipsoids drawn at the 30% probability level. Symmetry code: i = $-x + 1, -y + 2, -z + 1$. Disordering is shown as full and open bonds.

6,6'-Dimethyl-2,2'-[oxalylbis(azanediyl)]dipyridinium dichloride acetonitrile solvate

Crystal data

$C_{14}H_{16}N_4O_2^{2+}\cdot 2Cl^{-}\cdot C_2H_3N$	$F(000) = 400$
$M_r = 384.26$	$D_x = 1.399$ Mg m^{-3}

Monoclinic, $P2/c$

Hall symbol: -P 2yc

$a = 10.6740\ (19)$ Å

$b = 8.7637\ (5)$ Å

$c = 10.370\ (3)$ Å

$\beta = 109.83\ (2)°$

$V = 912.5\ (3)$ Å^3

$Z = 2$

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 28 reflections

$\theta = 4.7–12.5°$

$\mu = 0.38$ mm^{-1}

$T = 295$ K

Plate, colourless

0.6 × 0.2 × 0.1 mm

Data collection

Bruker P4 diffractometer

Radiation source: fine-focus sealed tube

graphite

ω scans

Absorption correction: ψ scan (*XSCANS*; Siemens, 1995)

$T_{\min} = 0.823$, $T_{\max} = 0.922$

2165 measured reflections

1619 independent reflections

1308 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\max} = 25.0°$, $\theta_{\min} = 2.0°$

$h = -12 \rightarrow 12$

$k = -10 \rightarrow 1$

$l = -12 \rightarrow 1$

3 standard reflections every 97 reflections

intensity decay: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.094$

$S = 1.07$

1619 reflections

118 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.2279P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.20$ e Å^{-3}

$\Delta\rho_{\min} = -0.17$ e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0090 (15)

Special details

Experimental. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl	0.23722 (6)	0.11398 (6)	0.09664 (5)	0.0569 (2)	
O	0.55710 (15)	0.81506 (16)	0.54656 (14)	0.0542 (4)	
N1	0.27012 (15)	0.78055 (18)	0.15297 (15)	0.0400 (4)	
H1A	0.2513	0.8715	0.1212	0.048*	
N2	0.39872 (16)	0.90108 (18)	0.34987 (15)	0.0424 (4)	
H2A	0.3653	0.9837	0.3073	0.051*	
N3	1.0000	0.6381 (4)	0.2500	0.0953 (12)	
C1	0.1214 (2)	0.7019 (3)	−0.0703 (2)	0.0596 (6)	
H1B	0.0933	0.6103	−0.1230	0.089*	
H1C	0.1660	0.7672	−0.1153	0.089*	
H1D	0.0450	0.7538	−0.0626	0.089*	
C2	0.2141 (2)	0.6624 (2)	0.0687 (2)	0.0443 (5)	
C3	0.2463 (2)	0.5175 (2)	0.1183 (2)	0.0521 (5)	
H3A	0.2118	0.4336	0.0627	0.062*	
C4	0.3305 (2)	0.4971 (3)	0.2514 (2)	0.0567 (6)	
H4A	0.3514	0.3985	0.2851	0.068*	
C5	0.3847 (2)	0.6201 (2)	0.3363 (2)	0.0522 (5)	
H5A	0.4406	0.6054	0.4262	0.063*	
C6	0.35325 (19)	0.7650 (2)	0.28310 (18)	0.0395 (4)	
C7	0.4915 (2)	0.9157 (2)	0.47672 (19)	0.0420 (5)	
C8	1.0000	0.7663 (4)	0.2500	0.0583 (8)	
C9	1.0000	0.9293 (4)	0.2500	0.0666 (9)	
H9B	1.0894	0.9658	0.2704	0.100*	0.50
H9A	0.9650	0.9658	0.3182	0.100*	0.50
H9C	0.9456	0.9658	0.1614	0.100*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0769 (4)	0.0307 (3)	0.0530 (3)	0.0030 (2)	0.0089 (3)	0.0052 (2)
O	0.0635 (9)	0.0404 (9)	0.0450 (7)	0.0124 (7)	0.0005 (7)	0.0022 (6)
N1	0.0498 (9)	0.0268 (8)	0.0401 (8)	0.0023 (7)	0.0112 (7)	0.0022 (6)
N2	0.0546 (10)	0.0297 (9)	0.0373 (8)	0.0057 (7)	0.0082 (7)	0.0012 (6)
N3	0.107 (3)	0.052 (2)	0.136 (3)	0.000	0.054 (3)	0.000
C1	0.0696 (15)	0.0438 (13)	0.0510 (12)	−0.0032 (11)	0.0017 (11)	−0.0076 (10)
C2	0.0499 (11)	0.0326 (10)	0.0494 (11)	−0.0034 (9)	0.0155 (9)	−0.0053 (8)
C3	0.0604 (14)	0.0300 (11)	0.0625 (13)	−0.0045 (9)	0.0166 (11)	−0.0053 (10)

C4	0.0689 (14)	0.0289 (10)	0.0680 (14)	0.0042 (10)	0.0178 (12)	0.0110 (10)
C5	0.0639 (13)	0.0361 (11)	0.0491 (11)	0.0044 (10)	0.0095 (10)	0.0071 (9)
C6	0.0465 (10)	0.0334 (10)	0.0382 (9)	0.0016 (8)	0.0140 (8)	0.0010 (8)
C7	0.0472 (11)	0.0398 (11)	0.0364 (10)	0.0059 (9)	0.0109 (8)	−0.0008 (8)
C8	0.0587 (19)	0.053 (2)	0.066 (2)	0.000	0.0248 (16)	0.000
C9	0.080 (2)	0.0492 (19)	0.068 (2)	0.000	0.0212 (18)	0.000

Geometric parameters (Å, °)

O—C7	1.204 (2)	C2—C3	1.370 (3)
N1—C6	1.347 (2)	C3—C4	1.380 (3)
N1—C2	1.356 (2)	C3—H3A	0.9300
N1—H1A	0.8600	C4—C5	1.388 (3)
N2—C7	1.358 (2)	C4—H4A	0.9300
N2—C6	1.382 (2)	C5—C6	1.379 (3)
N2—H2A	0.8600	C5—H5A	0.9300
N3—C8	1.123 (5)	C7—C7^{i}	1.545 (4)
C1—C2	1.486 (3)	C8—C9	1.429 (5)
C1—H1B	0.9600	C9—H9B	0.9600
C1—H1C	0.9600	C9—H9A	0.9600
C1—H1D	0.9600	C9—H9C	0.9600
C6—N1—C2	124.41 (17)	C3—C4—H4A	119.2
C6—N1—H1A	117.8	C5—C4—H4A	119.2
C2—N1—H1A	117.8	C6—C5—C4	117.98 (19)
C7—N2—C6	125.72 (16)	C6—C5—H5A	121.0
C7—N2—H2A	117.1	C4—C5—H5A	121.0
C6—N2—H2A	117.1	N1—C6—C5	118.82 (18)
C2—C1—H1B	109.5	N1—C6—N2	114.49 (16)
C2—C1—H1C	109.5	C5—C6—N2	126.69 (17)
H1B—C1—H1C	109.5	O—C7—N2	126.70 (18)
C2—C1—H1D	109.5	O—C7—C7^{i}	122.0 (2)
H1B—C1—H1D	109.5	N2—C7—C7^{i}	111.3 (2)
H1C—C1—H1D	109.5	N3—C8—C9	180.000 (2)
N1—C2—C3	117.77 (18)	C8—C9—H9B	109.5
N1—C2—C1	116.77 (18)	C8—C9—H9A	109.5
C3—C2—C1	125.46 (19)	H9B—C9—H9A	109.5
C2—C3—C4	119.4 (2)	C8—C9—H9C	109.5
C2—C3—H3A	120.3	H9B—C9—H9C	109.5
C4—C3—H3A	120.3	H9A—C9—H9C	109.5
C3—C4—C5	121.6 (2)		

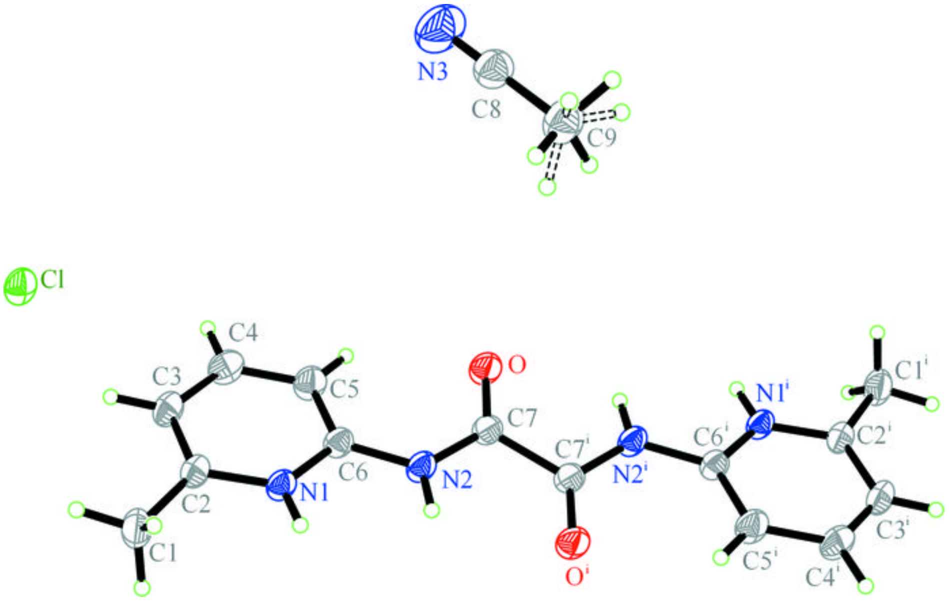
Symmetry codes: (i) $-x+1, -y+2, -z+1$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···Cl^{ii}	0.86	2.14	2.9772 (16)	165.
N2—H2A···Cl^{ii}	0.86	2.43	3.2057 (17)	150.

Symmetry codes: (ii) $x, y+1, z$.

Fig. 1


N3
C8
C9
Cl
C4
C3
C5
O
C7
C7ⁱ
N2ⁱ
C6ⁱ
N1ⁱ
C1ⁱ
C2ⁱ
C2
N1
C6
N2
C1
Oⁱ
C5ⁱ
C4ⁱ
C3ⁱ