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3,3′-Carbonyldipyridinium bis(perchlorate)

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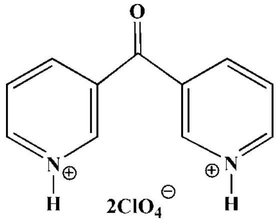
Key indicators: single-crystal X-ray study; *T* = 296 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.028; *wR* factor = 0.073; data-to-parameter ratio = 11.6.

In the title molecular salt, $C_{11}H_{10}N_2O^{2+}\cdot 2ClO_4^-$, the complete cation is generated by crystallographic twofold symmetry. The dihedral angle between the pyridyl rings is 67.07 (7)°. The crystal structure features N—H···Cl hydrogen bonds, forming sheets in the *ab* plane.

Related literature

For the dipyridyl ketone dication, see: Crook & McElvain (1930); Favaro *et al.* (1990). For metal complexes of di-3-pyridyl ketone, see: Chen & Mak (2005); Chen *et al.* (2009).



Experimental

Crystal data

$C_{11}H_{10}N_2O^{2+}\cdot 2ClO_4^-$
M_r = 385.11
Orthorhombic, $P2_12_12$
a = 8.5315 (3) Å
b = 15.1772 (6) Å
c = 5.6107 (2) Å
V = 726.50 (5) Å^3
Z = 2
Mo *K*α radiation
μ = 0.50 mm^{-1}
T = 296 K
0.40 × 0.30 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.835, T_{max} = 0.905
6343 measured reflections
1285 independent reflections
1235 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.073
S = 1.10
1285 reflections
111 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 503 Friedel pairs
Flack parameter: 0.10 (10)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H7···O2	0.86	2.22	2.907 (3)	136
N1^i—H7^i···O4	0.86	2.34	2.967 (2)	130

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2* and *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5923).

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supplementary materials

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3,3′-Carbonyldipyridinium bis(perchlorate)

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Comment

Di-3-pyridyl ketone is an extraordinary ligand within the family of basic building blocks for construction of metal-organic complexes with intriguing architectures (Chen & Mak, 2005; Chen *et al.*, 2009). However, the crystal structure of salts with the dipyridyl ketone dication is rarely reported until now. Several related literatures discussed the relationship between the acid-base properties of the dipyridyl ketone isomers and the positions of the nitrogen atoms on the rings, which were investigated by spectrophotometric measurements (Crook & McElvain, 1930; Favaro *et al.*, 1990). In the present context, we report the structure of diprotonated di-3-pyridyl ketone perchlorate salt (Fig. 1). The two pyridyl rings exhibit a dihedral angle of 67.07 (7)°. The crystal structure is stabilized by N—H···(perchlorate) hydrogen bonds forming sheets in the ab plane.

Experimental

Di-3-pyridyl ketone was prepared following the literature procedure of Chen & Mak (2005). Copper(II) perchlorate (37 mg, 0.1 mmol) was heated with di-3-pyridyl ketone (18 mg, 0.1 mmol) in acetonitrile (5 ml) at 373 K for 24 h. After cooling to room temperature, the precipitate which had formed was filtrated off. Crystals of the title salt was deposited by slow evaporation of the filtrate, which can be viewed as the product of the perchloric acid from the copper(II) perchlorate and di-3-pyridyl ketone (yield 11.5 mg, 30% based on di-3-pyridyl ketone).

Refinement

H atoms were placed in idealized positions and allowed to ride on their parent atoms, with C—H = 0.93 Å and N—H = 0.86 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,N).

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2* and *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); 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* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

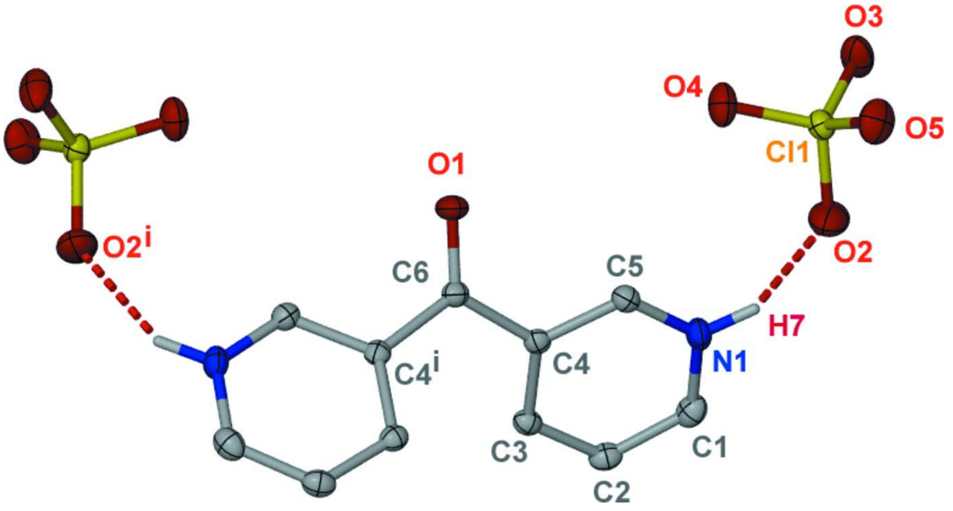

Figure 1

The atom-numbering scheme of the title salt; some H atoms have been omitted for clarity. Displacement ellipsoids are shown at the 30% probability level. [Symmetry code: (i) -x + 1, -y, z.]

3,3′-Carbonyldipyridinium diperchlorate

Crystal data

$C_{11}H_{10}N_2O^{2+}\cdot 2ClO_4^-$
M_r = 385.11
Orthorhombic, $P2_12_12$
a = 8.5315 (3) Å
b = 15.1772 (6) Å
c = 5.6107 (2) Å
V = 726.50 (5) Å^3
Z = 2
F(000) = 392
D_x = 1.760 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 256 reflections
θ = 2.3–26.2°
μ = 0.50 mm^{-1}
T = 296 K
Block, colorless
0.40 × 0.30 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{\min}$ = 0.835, $T_{\max}$ = 0.905
6343 measured reflections
1285 independent reflections
1235 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.021
$\theta_{\max}$ = 25.0°, $\theta_{\min}$ = 2.7°
h = −10→10
k = −18→16
l = −6→6

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.073
S = 1.10
1285 reflections
111 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
$w = 1/[\sigma^2(F_o^2) + (0.0315P)^2 + 0.4015P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.34 e Å^{-3}
$\Delta\rho_{\min}$ = −0.21 e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.019 (3)

Absolute structure: Flack (1983), 503 Friedel pairs
Flack parameter: 0.10 (10)

Special details

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 $F^2 > 2\sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1257 (3)	0.13417 (17)	0.4051 (5)	0.0416 (6)
H1	0.0408	0.1642	0.3402	0.050*
C2	0.1807 (3)	0.05924 (17)	0.2988 (4)	0.0372 (6)
H2	0.1340	0.0381	0.1604	0.045*
C3	0.3062 (3)	0.01520 (14)	0.3985 (4)	0.0317 (5)
H3	0.3449	−0.0357	0.3274	0.038*
C4	0.3745 (3)	0.04756 (14)	0.6063 (4)	0.0287 (5)
C5	0.3158 (3)	0.12400 (15)	0.7048 (4)	0.0334 (5)
H5	0.3609	0.1474	0.8419	0.040*
C6	0.5000	0.0000	0.7390 (6)	0.0308 (7)
N1	0.1948 (2)	0.16399 (13)	0.6032 (4)	0.0386 (5)
H7	0.1591	0.2113	0.6677	0.046*
O1	0.5000	0.0000	0.9548 (4)	0.0431 (6)
Cl1	0.30796 (6)	0.32979 (4)	1.13244 (10)	0.03518 (19)
O2	0.2552 (3)	0.31908 (15)	0.8930 (4)	0.0720 (7)
O3	0.1779 (3)	0.33349 (15)	1.2909 (4)	0.0647 (6)
O4	0.4064 (2)	0.25628 (13)	1.1946 (4)	0.0539 (6)
O5	0.3940 (3)	0.41004 (13)	1.1460 (5)	0.0622 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0318 (12)	0.0470 (14)	0.0461 (16)	0.0021 (11)	−0.0001 (12)	0.0095 (13)
C2	0.0350 (13)	0.0444 (14)	0.0323 (12)	−0.0082 (12)	−0.0038 (11)	0.0022 (10)
C3	0.0339 (11)	0.0321 (12)	0.0293 (11)	−0.0041 (10)	0.0037 (12)	−0.0010 (10)
C4	0.0293 (11)	0.0299 (11)	0.0269 (11)	−0.0019 (9)	0.0043 (11)	0.0025 (10)
C5	0.0348 (12)	0.0336 (12)	0.0319 (12)	−0.0013 (11)	0.0021 (11)	−0.0022 (9)
C6	0.0346 (18)	0.0289 (17)	0.0288 (18)	−0.0017 (15)	0.000	0.000
N1	0.0378 (10)	0.0327 (10)	0.0452 (12)	0.0081 (10)	0.0046 (10)	−0.0007 (10)
O1	0.0499 (15)	0.0537 (16)	0.0259 (13)	0.0073 (14)	0.000	0.000
Cl1	0.0335 (3)	0.0328 (3)	0.0393 (3)	−0.0005 (2)	0.0001 (3)	−0.0034 (3)
O2	0.0990 (17)	0.0660 (14)	0.0511 (12)	0.0026 (13)	−0.0214 (12)	−0.0129 (12)
O3	0.0620 (13)	0.0551 (12)	0.0770 (15)	0.0139 (12)	0.0314 (11)	0.0109 (12)
O4	0.0398 (10)	0.0402 (10)	0.0817 (16)	0.0070 (8)	−0.0062 (10)	−0.0008 (10)

O5	0.0604 (12)	0.0403 (11)	0.0858 (16)	−0.0138 (9)	−0.0075 (14)	0.0029 (12)

Geometric parameters (Å, º)

C1—N1	1.337 (3)	C5—N1	1.327 (3)
C1—C2	1.368 (4)	C5—H5	0.9300
C1—H1	0.9300	C6—O1	1.210 (4)
C2—C3	1.381 (4)	C6—C4$^{\mathrm{i}}$	1.491 (3)
C2—H2	0.9300	N1—H7	0.8600
C3—C4	1.393 (3)	Cl1—O3	1.423 (2)
C3—H3	0.9300	Cl1—O5	1.4242 (19)
C4—C5	1.379 (3)	Cl1—O2	1.426 (2)
C4—C6	1.491 (3)	Cl1—O4	1.439 (2)
N1—C1—C2	119.5 (2)	C4—C5—H5	120.2
N1—C1—H1	120.2	O1—C6—C4	119.97 (14)
C2—C1—H1	120.2	O1—C6—C4$^{\mathrm{i}}$	119.97 (14)
C1—C2—C3	119.5 (2)	C4—C6—C4$^{\mathrm{i}}$	120.1 (3)
C1—C2—H2	120.3	C5—N1—C1	123.1 (2)
C3—C2—H2	120.3	C5—N1—H7	118.5
C2—C3—C4	119.5 (2)	C1—N1—H7	118.5
C2—C3—H3	120.2	O3—Cl1—O5	109.56 (14)
C4—C3—H3	120.2	O3—Cl1—O2	110.31 (16)
C5—C4—C3	118.7 (2)	O5—Cl1—O2	108.08 (15)
C5—C4—C6	117.9 (2)	O3—Cl1—O4	109.53 (13)
C3—C4—C6	123.2 (2)	O5—Cl1—O4	110.44 (12)
N1—C5—C4	119.7 (2)	O2—Cl1—O4	108.90 (14)
N1—C5—H5	120.2		
N1—C1—C2—C3	0.4 (4)	C5—C4—C6—O1	34.0 (2)
C1—C2—C3—C4	0.2 (3)	C3—C4—C6—O1	−141.03 (17)
C2—C3—C4—C5	−1.0 (3)	C5—C4—C6—C4$^{\mathrm{i}}$	−146.0 (2)
C2—C3—C4—C6	174.0 (2)	C3—C4—C6—C4$^{\mathrm{i}}$	38.97 (17)
C3—C4—C5—N1	1.2 (3)	C4—C5—N1—C1	−0.6 (4)
C6—C4—C5—N1	−174.1 (2)	C2—C1—N1—C5	−0.2 (4)

Symmetry code: (i) −*x*+1, −*y*, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H7···O2	0.86	2.22	2.907 (3)	136
N1$^{\mathrm{ii}}$—H7$^{\mathrm{ii}}$···O4	0.86	2.34	2.967 (2)	130

Symmetry code: (ii) *x*+1/2, −*y*+1/2, −*z*+2.