Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-[3-(3-Pyridyloxy)pyrazin-2-yloxy]-pyridinium perchlorate

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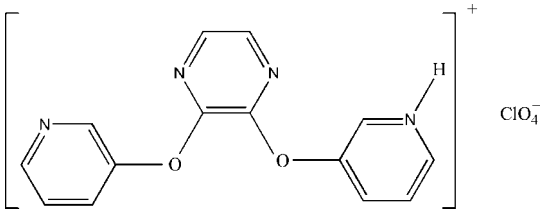
Received 17 July 2007; accepted 24 July 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.064; wR factor = 0.233; data-to-parameter ratio = 14.9.

In the crystal structure of the title compound, $C_{14}H_{11}N_4O_2^+\cdot ClO_4^-$, the cations are connected by intermolecular N—H···N hydrogen bonds to form one-dimensional chains along the a axis, while weak intermolecular C—H···O hydrogen bonds connect the cations to the perchlorate anions. In the cation, the dihedral angles between the pyrazine ring and the two pyridine rings are 71.00 (16) and 64.59 (16)°, and the dihedral angle between the two pyridine rings is 12.02 (16)°.

Related literature

For a related structure, see: McMorran & Steel (2002). For related literature, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{14}H_{11}N_4O_2^+\cdot ClO_4^-$
M_r = 366.72
Monoclinic, $P2_1/c$
a = 11.557 (3) Å
b = 9.815 (2) Å
c = 13.955 (3) Å
β = 100.444 (4)°
V = 1556.8 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 298 (2) K
0.23 × 0.18 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.937, T_{max} = 0.955
9181 measured reflections
3365 independent reflections
2041 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.233
S = 0.78
3365 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H2···N3^{i}	0.86	1.81	2.669 (4)	175
C12—H12···O5^{ii}	0.93	2.54	3.410 (5)	155

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2464).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
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 doi:10.1107/S1600536807036367

supplementary materials

Acta Cryst. (2007). E**63**, o3651 [**doi:10.1107/S1600536807036367**]

3-[3-(3-Pyridyloxy)pyrazin-2-yloxy]pyridinium perchlorate

J. M. Li

Comment

Compounds containing pyrazine and pyridine rings have play an important role in coordination chemistry (McMorran & Steel, 2002) and it was originally hoped to synthesize a Zn^{II} complex of 2,3-bis(pyridin-3-yloxy)pyrazine and phosphorate as multi-dentate ligands, but instead the title compound was obtained and its crystal structure is reported herein.

The asymmetric unit is shown in Fig. 1. Geometric parameters are in the usual rangs (Allen *et al.*, 1987). In the cation, the dihedral angles between the pyrazine ring and the two pyridine rings are 71.00 (16)° and 64.59 (16)°, and the dihedral angle between the two pyridine rings is 12.02 (16)°. In the crystal structure, intermolecular N—H···N hydrogen bonds link cation molecules to form one-dimensional chains along the *a* axis. In addition, weak C—H···O hydrogen bonds connect perchlorate anions bound to the cations.

Experimental

5 ml me thanol solution of 2,3-bis(pyridin-3-yloxy)prazine (0.0521 g, 0.196 mmol) was added into 10 ml H_2O solution containing $Zn(ClO_4)_2·6H_2O$ (0.1430 g, 0.384 mmol) and $NaH_2PO_4·2H_2O$ (0.0602 g, 0.386 mmol), and the mixture was stirred for a few minutes. Yellow single crystals were obtained after the solution had been allowed to stand at room temperature for one week.

Refinement

The H atoms were placed in calculated positions and refined as riding, with C—H = 0.93 Å, U_{iso}(H) = 1.2$_{eq}$(C), N—H = 0.86 Å, U_{iso}(H) = 1.2$_{eq}$(N). The larger than normal anisotropic displacement parameters associated with the O atoms of the perchlorate anion may be due to a small amount of disorder with repect to these atoms. The disorder was not modelled.

Figures

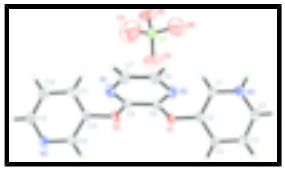

Fig. 1. The asymmetric unit showing the the atom numbering scheme with thermal ellipsoids drawn at the 30% probability level.

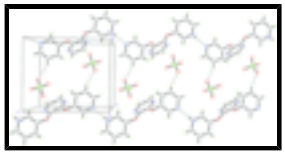

Fig. 2. Partial packing plot with hydrogen bonds shown as dashed lines.

3-[3-(3-Pyridyloxy)pyrazin-2-yloxy]pyridinium perchlorate

Crystal data

$C_{14}H_{11}N_4O_2^+\cdot ClO_4^-$	F_{000} = 752
M_r = 366.72	D_x = 1.565 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1097 reflections
a = 11.557 (3) Å	θ = 2.6–20.3º
b = 9.815 (2) Å	μ = 0.29 mm^{-1}
c = 13.955 (3) Å	T = 298 (2) K
β = 100.444 (4)º	Prism-like, yellow
V = 1556.8 (6) Å^3	0.23 × 0.18 × 0.16 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3365 independent reflections
Radiation source: fine-focus sealed tube	2041 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.047
T = 298(2) K	θ_{max} = 27.0º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −13→14
T_{min} = 0.937, T_{max} = 0.955	k = −12→12
9181 measured reflections	l = −15→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.064	H-atom parameters constrained
$wR(F^2)$ = 0.233	$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.78	$(\Delta/\sigma)_{max}$ = 0.003
3365 reflections	$\Delta\rho_{max}$ = 0.56 e Å^{-3}
226 parameters	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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}
Cl1	0.21688 (9)	0.66871 (9)	0.09517 (7)	0.0516 (3)
O2	0.50784 (18)	0.9667 (2)	0.12191 (17)	0.0451 (6)
O1	0.31002 (18)	1.0866 (2)	0.14694 (17)	0.0410 (6)
N1	0.5197 (2)	0.8455 (3)	0.2650 (2)	0.0433 (7)
N3	0.8152 (2)	0.9357 (3)	0.1091 (2)	0.0453 (7)
N4	−0.0023 (2)	1.1068 (3)	0.1394 (2)	0.0481 (7)
H2	−0.0623	1.0535	0.1327	0.058*
N2	0.3098 (2)	0.9658 (3)	0.2899 (2)	0.0425 (7)
C8	0.3580 (3)	0.9940 (3)	0.2154 (2)	0.0363 (7)
C9	0.4649 (3)	0.9316 (3)	0.2024 (2)	0.0369 (7)
C6	0.3666 (3)	0.8749 (4)	0.3551 (3)	0.0510 (9)
H6	0.3344	0.8522	0.4094	0.061*
C5	−0.0182 (3)	1.2402 (4)	0.1459 (3)	0.0497 (9)
H5	−0.0935	1.2740	0.1451	0.060*
C14	0.7113 (3)	0.9855 (4)	0.1218 (3)	0.0445 (8)
H14	0.7056	1.0766	0.1387	0.053*
C7	0.4695 (3)	0.8163 (4)	0.3429 (3)	0.0488 (9)
H7	0.5062	0.7546	0.3892	0.059*
C2	0.2006 (3)	1.1387 (3)	0.1528 (2)	0.0359 (7)
C10	0.6132 (3)	0.9047 (3)	0.1105 (2)	0.0377 (7)
C13	0.8222 (3)	0.8059 (4)	0.0853 (3)	0.0459 (9)
H13	0.8946	0.7715	0.0768	0.055*
C1	0.1045 (3)	1.0532 (3)	0.1427 (3)	0.0438 (8)
H1	0.1139	0.9595	0.1383	0.053*
C3	0.1868 (3)	1.2755 (4)	0.1582 (2)	0.0417 (8)
H3	0.2515	1.3333	0.1647	0.050*
C4	0.0738 (3)	1.3278 (4)	0.1539 (3)	0.0509 (9)
H4	0.0617	1.4213	0.1564	0.061*
C11	0.6194 (3)	0.7710 (4)	0.0858 (3)	0.0460 (9)
H11	0.5531	0.7155	0.0778	0.055*
C12	0.7274 (3)	0.7203 (4)	0.0728 (3)	0.0523 (10)
H12	0.7353	0.6295	0.0558	0.063*
O5	0.1759 (3)	0.5676 (3)	0.0247 (2)	0.0879 (11)

O4	0.2440 (4)	0.7893 (3)	0.0502 (3)	0.0944 (12)
O3	0.3166 (5)	0.6217 (6)	0.1546 (5)	0.168 (2)
O6	0.1339 (5)	0.6938 (5)	0.1538 (4)	0.150 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0620 (7)	0.0423 (5)	0.0517 (6)	−0.0002 (4)	0.0137 (5)	−0.0013 (4)
O2	0.0263 (11)	0.0570 (15)	0.0549 (14)	0.0092 (10)	0.0150 (10)	0.0168 (11)
O1	0.0222 (11)	0.0449 (13)	0.0572 (14)	0.0044 (9)	0.0110 (10)	0.0112 (11)
N1	0.0259 (14)	0.0576 (18)	0.0461 (16)	0.0052 (12)	0.0059 (12)	0.0043 (13)
N3	0.0268 (14)	0.0515 (18)	0.0599 (18)	0.0016 (12)	0.0135 (13)	0.0034 (14)
N4	0.0251 (14)	0.0525 (18)	0.0676 (19)	−0.0042 (12)	0.0103 (13)	0.0030 (15)
N2	0.0340 (14)	0.0482 (17)	0.0476 (16)	0.0050 (12)	0.0132 (13)	0.0007 (13)
C8	0.0236 (15)	0.0379 (17)	0.0468 (18)	−0.0016 (13)	0.0048 (14)	−0.0015 (14)
C9	0.0260 (15)	0.0420 (18)	0.0426 (17)	0.0000 (13)	0.0061 (13)	0.0015 (14)
C6	0.050 (2)	0.059 (2)	0.0467 (19)	0.0069 (18)	0.0163 (17)	0.0038 (17)
C5	0.0296 (18)	0.049 (2)	0.070 (2)	0.0105 (15)	0.0083 (17)	0.0060 (18)
C14	0.0331 (18)	0.0378 (18)	0.065 (2)	0.0001 (14)	0.0166 (16)	−0.0034 (16)
C7	0.044 (2)	0.062 (2)	0.0399 (18)	0.0121 (17)	0.0061 (15)	0.0089 (16)
C2	0.0250 (15)	0.0397 (18)	0.0438 (17)	0.0027 (13)	0.0082 (13)	0.0042 (13)
C10	0.0277 (16)	0.0467 (19)	0.0410 (17)	0.0022 (13)	0.0125 (13)	0.0039 (14)
C13	0.0394 (19)	0.053 (2)	0.049 (2)	0.0096 (16)	0.0186 (16)	0.0029 (16)
C1	0.0297 (17)	0.0364 (18)	0.067 (2)	0.0027 (14)	0.0127 (16)	0.0034 (16)
C3	0.0307 (17)	0.0438 (19)	0.0499 (19)	−0.0007 (14)	0.0055 (15)	0.0037 (15)
C4	0.044 (2)	0.0373 (19)	0.073 (3)	0.0074 (15)	0.0140 (19)	0.0064 (17)
C11	0.043 (2)	0.049 (2)	0.046 (2)	−0.0131 (16)	0.0109 (16)	−0.0006 (16)
C12	0.066 (3)	0.045 (2)	0.052 (2)	0.0078 (18)	0.0251 (19)	−0.0021 (17)
O5	0.128 (3)	0.0568 (19)	0.081 (2)	−0.0236 (19)	0.026 (2)	−0.0157 (16)
O4	0.155 (4)	0.0531 (18)	0.078 (2)	−0.026 (2)	0.030 (2)	0.0002 (16)
O3	0.137 (4)	0.155 (5)	0.179 (5)	0.026 (4)	−0.061 (4)	0.032 (4)
O6	0.186 (5)	0.119 (4)	0.185 (5)	−0.023 (3)	0.139 (5)	−0.045 (3)

Geometric parameters (Å, °)

Cl1—O3	1.372 (5)	C6—H6	0.9300
Cl1—O4	1.402 (3)	C5—C4	1.357 (5)
Cl1—O5	1.417 (3)	C5—H5	0.9300
Cl1—O6	1.391 (4)	C14—C10	1.369 (5)
O2—C9	1.352 (4)	C14—H14	0.9300
O2—C10	1.396 (4)	C7—H7	0.9300
O1—C8	1.361 (4)	C2—C3	1.356 (5)
O1—C2	1.381 (4)	C2—C1	1.378 (5)
N1—C9	1.295 (4)	C10—C11	1.362 (5)
N1—C7	1.353 (4)	C13—C12	1.366 (5)
N3—C13	1.323 (4)	C13—H13	0.9300
N3—C14	1.337 (4)	C1—H1	0.9300
N4—C5	1.328 (5)	C3—C4	1.395 (5)
N4—C1	1.334 (4)	C3—H3	0.9300

N4—H2	0.8600	C4—H4	0.9300
N2—C8	1.296 (4)	C11—C12	1.387 (5)
N2—C6	1.356 (4)	C11—H11	0.9300
C8—C9	1.420 (4)	C12—H12	0.9300
C6—C7	1.359 (5)		
O3—Cl1—O4	108.9 (3)	C10—C14—H14	119.4
O3—Cl1—O5	108.8 (3)	C6—C7—N1	121.6 (3)
O4—Cl1—O5	110.7 (2)	C6—C7—H7	119.2
O3—Cl1—O6	107.3 (4)	N1—C7—H7	119.2
O4—Cl1—O6	110.3 (3)	C3—C2—O1	119.2 (3)
O5—Cl1—O6	110.7 (3)	C3—C2—C1	120.5 (3)
C9—O2—C10	116.3 (2)	O1—C2—C1	119.9 (3)
C8—O1—C2	117.8 (2)	C14—C10—C11	120.5 (3)
C9—N1—C7	116.9 (3)	C14—C10—O2	117.1 (3)
C13—N3—C14	118.8 (3)	C11—C10—O2	122.4 (3)
C5—N4—C1	121.7 (3)	N3—C13—C12	122.8 (3)
C5—N4—H2	119.2	N3—C13—H13	118.6
C1—N4—H2	119.2	C12—C13—H13	118.6
C8—N2—C6	116.9 (3)	N4—C1—C2	119.1 (3)
N2—C8—O1	121.7 (3)	N4—C1—H1	120.4
N2—C8—C9	121.4 (3)	C2—C1—H1	120.4
O1—C8—C9	116.9 (3)	C2—C3—C4	118.7 (3)
N1—C9—O2	121.3 (3)	C2—C3—H3	120.6
N1—C9—C8	121.7 (3)	C4—C3—H3	120.6
O2—C9—C8	117.0 (3)	C5—C4—C3	118.9 (3)
C7—C6—N2	121.5 (3)	C5—C4—H4	120.5
C7—C6—H6	119.2	C3—C4—H4	120.5
N2—C6—H6	119.2	C10—C11—C12	118.0 (3)
N4—C5—C4	121.0 (3)	C10—C11—H11	121.0
N4—C5—H5	119.5	C12—C11—H11	121.0
C4—C5—H5	119.5	C13—C12—C11	118.8 (3)
N3—C14—C10	121.2 (3)	C13—C12—H12	120.6
N3—C14—H14	119.4	C11—C12—H12	120.6
C6—N2—C8—O1	−178.2 (3)	C8—O1—C2—C1	−63.0 (4)
C6—N2—C8—C9	0.5 (5)	N3—C14—C10—C11	−0.1 (5)
C2—O1—C8—N2	−7.4 (4)	N3—C14—C10—O2	−177.8 (3)
C2—O1—C8—C9	173.9 (3)	C9—O2—C10—C14	−109.9 (3)
C7—N1—C9—O2	−179.7 (3)	C9—O2—C10—C11	72.4 (4)
C7—N1—C9—C8	0.9 (5)	C14—N3—C13—C12	0.2 (5)
C10—O2—C9—N1	0.3 (4)	C5—N4—C1—C2	−0.3 (5)
C10—O2—C9—C8	179.8 (3)	C3—C2—C1—N4	−0.7 (5)
N2—C8—C9—N1	−1.0 (5)	O1—C2—C1—N4	−173.7 (3)
O1—C8—C9—N1	177.7 (3)	O1—C2—C3—C4	173.4 (3)
N2—C8—C9—O2	179.5 (3)	C1—C2—C3—C4	0.4 (5)
O1—C8—C9—O2	−1.8 (4)	N4—C5—C4—C3	−2.0 (6)
C8—N2—C6—C7	0.1 (5)	C2—C3—C4—C5	1.0 (5)
C1—N4—C5—C4	1.7 (6)	C14—C10—C11—C12	0.1 (5)
C13—N3—C14—C10	−0.1 (5)	O2—C10—C11—C12	177.7 (3)

N2—C6—C7—N1	−0.2 (6)	N3—C13—C12—C11	−0.2 (6)
C9—N1—C7—C6	−0.3 (5)	C10—C11—C12—C13	0.0 (5)
C8—O1—C2—C3	124.0 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H2···N3^{i}	0.86	1.81	2.669 (4)	175
C12—H12···O5^{ii}	0.93	2.54	3.410 (5)	155

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) −*x*+1, −*y*+1, −*z*.

Fig. 1

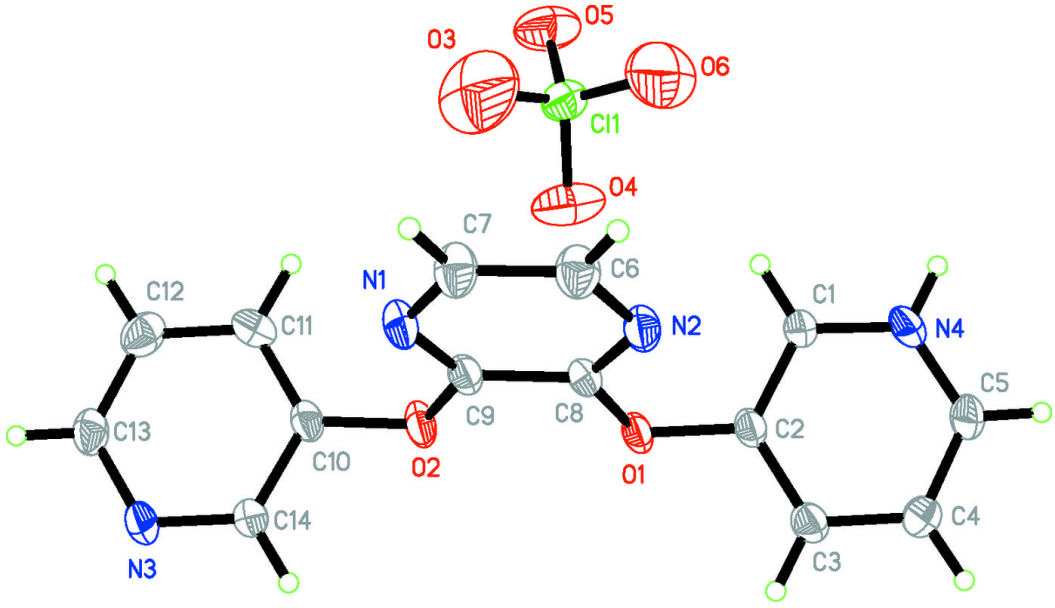
O5
O3
O6
Cl1
O4
C7
N1
C6
N2
C1
N4
C12
C11
C5
C13
C9
C8
C2
C10
O2
O1
C14
C4
N3
C3

Fig. 2