Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[2-(2-pyridylsulfanyl)ethyl]ammonium perchlorate

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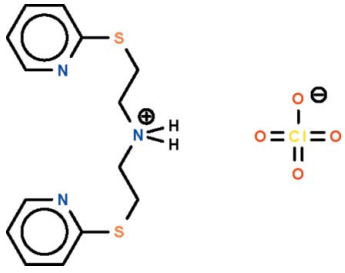
Received 21 July 2010; accepted 10 August 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; disorder in solvent or counterion; R factor = 0.071; wR factor = 0.214; data-to-parameter ratio = 14.5.

The cation and anion of the title salt, $C_{14}H_{18}N_3S_2^+\cdot ClO_4^-$, lie on a twofold rotation axis. The cation is a W-shaped entity with the aromatic rings at the ends; the ammonium NH_2^+ group is a hydrogen-bond donor to the pyridyl N atoms. The perchlorate ion has one O atom disordered over two sites in a 0.50:0.50 ratio.

Related literature

For the structure of tris[2-(2-pyridylsulfanyl)ethyl]ammonium perchlorate, see: An *et al.* (2010).



Experimental

Crystal data

$C_{14}H_{18}N_3S_2^+\cdot ClO_4^-$
M_r = 391.88
Monoclinic, $P2/n$
a = 8.1265 (6) Å
b = 9.2291 (7) Å
c = 11.9872 (9) Å
β = 97.534 (7)°
V = 891.28 (12) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 4.31 mm^{-1}
T = 293 K
0.15 × 0.15 × 0.10 mm

Data collection

Oxford Xcalibur Sapphire-3 diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.345, T_{max} = 1.000
3124 measured reflections
1736 independent reflections
1427 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.214
S = 1.11
1736 reflections
120 parameters
9 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N1	0.86	2.11	2.832 (5)	141

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The authors thank the Special Foundation for Nanotechnology of the Shanghai Committee for Science and Technology (grant No. 1052nm00600), the Foundation of the Science and Technology Programme of Shanghai Maritime University (grant No. 20100128), the State Key Laboratory of Pollution Control and Resource Reuse Foundation (grant No. PCRRF09001) and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2303).

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supporting information

Acta Cryst. (2010). E**66**, o2335 [https://doi.org/10.1107/S1600536810032216]

Bis[2-(2-pyridylsulfanyl)ethyl]ammonium perchlorate

Guo-Qing Wang, Cong-Hui Ma, Xiao-Feng Li, Wen-Ge Li and Seik Weng Ng

S1. Comment

We are engaged in the study of metal complexes of di- and tri-(pyridylsulfanyl)alkylamines as such compounds owing to their flexible nature. We reported earlier the synthesis of tris[2-(2-pyridylsulfanyl)ethyl]amine, whose reaction with copper perchlorate gave instead tris[2-(2-pyridylsulfanyl)ethyl]ammonium perchlorate (An *et al.*, 2010). The two-legged bis[2-(2-pyridylsulfanyl)ethyl]amine, in the present study, reacted with copper perchlorate to afford the corresponding ammonium perchlorate (Scheme and Fig. 1).

S2. Experimental

Bis(2-chloroethyl)ammonium hydrochloride (8.92 g, 0.05 mol) in ethanol (100 ml) was added to a solution (353 K) of 2-mercaptopyridine (12.23 g, 0.11 mol) and potassium hydroxide (6.17 g, 0.11 mol) in ethanol (200 ml). The mixture was heated at 353 K for 8 h. The solvent was removed to yield a yellow oil; this was column chromatographed with ethly acetate/petroleum ether (3/5 *v*/*v*) as eluent; yield 65%. ^{1}H NMR ($CDCl_3$, 400 MHz, p.p.m.): 3.316–3.349 (*t*, 4H), 2.959–2.992 (*t*, 4H), 6.924–6.960 (*m*, 2H), 7.154–7.173 (*m*, 2H), 7.416–7.459 (*m*, 2H), 8.382–8.393 (*m*, 2H).

The title salt was obtained from the reaction of bis[2-(2-pyridylsulfanyl)ethyl]amine (0.5 mmol, 0.146 g) and copper perchlorate (0.5 mmol, 0.132 g) in ethanol. Colorless crystals were separated from the blue solution after three days. CH&N elemental analysis, calculated for $C_{14}H_{18}O_4N_3S_2Cl$: C 42.91, H 4.63, N 10.72%; Found: C 42.73, H 4.35, N 11.02%.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.97 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 times U_{eq}(C). The ammonium H-atom was similarly positioned [N—H 0.86 Å] and its temperature factor tied by a factor of 1.2.

The perchlorate ion is disordered about the twofold rotation axis with respect to one O atom; two O atoms were assigned half-occupancy. The Cl—O distances were restrained to within 0.005 Å of each other, as were the O···O separations.

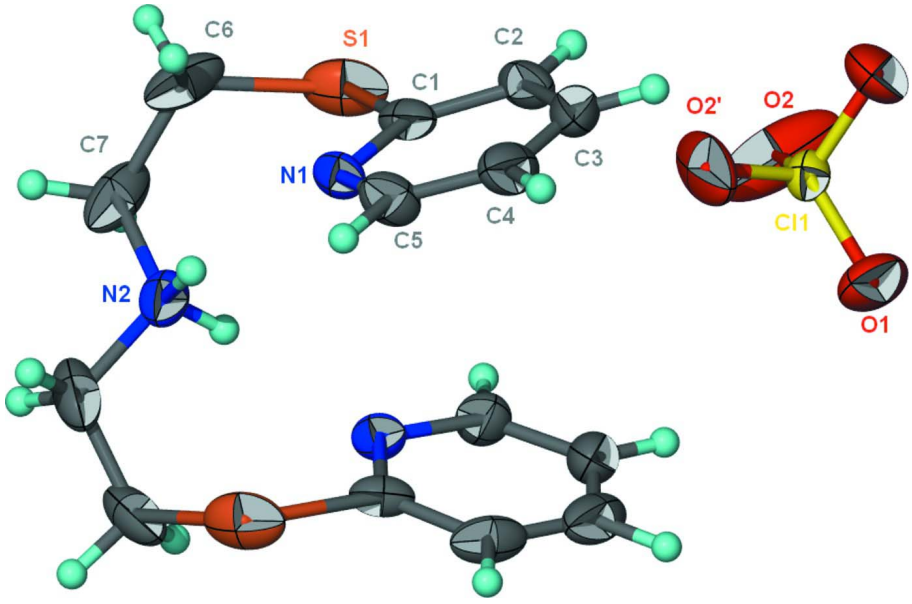

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $[C_{14}H_{18}N_3S_2]^+[ClO_4]^-$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. For the cation, the unlabeled atoms are related to the labeled ones by symmetry 1/2-*x*, *y*, 3/2-*z*.

Bis[2-(2-pyridylsulfanyl)ethyl]ammonium perchlorate

Crystal data

$C_{14}H_{18}N_3S_2^+\cdot ClO_4^-$
$M_r = 391.88$
Monoclinic, *P*2/*n*
Hall symbol: -P 2yac
$a = 8.1265\ (6)$ Å
$b = 9.2291\ (7)$ Å
$c = 11.9872\ (9)$ Å
$\beta = 97.534\ (7)°$
$V = 891.28\ (12)$ Å^3
$Z = 2$
$F(000) = 408$
$D_x = 1.460$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 1783 reflections
$\theta = 3.7–72.5°$
$\mu = 4.31$ mm^{-1}
$T = 293$ K
Prism, yellow
0.15 × 0.15 × 0.10 mm

Data collection

Oxford Xcalibur Sapphire-3 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.0855 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
$T_{min} = 0.345$, $T_{max} = 1.000$
3124 measured reflections
1736 independent reflections
1427 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 72.7°$, $\theta_{min} = 4.8°$
$h = -6\rightarrow9$
$k = -11\rightarrow10$
$l = -14\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.071$
$wR(F^2) = 0.214$
$S = 1.11$
1736 reflections
120 parameters
9 restraints

0 constraints
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

$w = 1/[\sigma^2(F_o^2) + (0.1333P)^2 + 0.2886P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.66$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.077 (7)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	0.7500	−0.07951 (11)	0.7500	0.0585 (5)	
S1	0.15849 (15)	0.6905 (2)	0.51373 (9)	0.1143 (7)	
O1	0.7006 (6)	−0.1628 (4)	0.8355 (3)	0.1259 (15)	
O2	0.6180 (11)	−0.001 (2)	0.6944 (18)	0.231 (14)	0.50
O2′	0.8847 (16)	0.0079 (15)	0.7855 (10)	0.184 (11)	0.50
N1	0.4045 (4)	0.6093 (3)	0.6688 (2)	0.0637 (8)	
N2	0.2500	0.8558 (5)	0.7500	0.0789 (13)	
H2	0.3345	0.8007	0.7463	0.095*	
C1	0.2997 (4)	0.5641 (5)	0.5824 (3)	0.0703 (10)	
C2	0.2947 (7)	0.4216 (6)	0.5433 (4)	0.0947 (16)	
H2A	0.2197	0.3938	0.4817	0.114*	
C3	0.4023 (10)	0.3248 (6)	0.5977 (6)	0.108 (2)	
H3	0.4022	0.2290	0.5736	0.130*	
C4	0.5080 (8)	0.3672 (5)	0.6857 (5)	0.1001 (16)	
H4	0.5815	0.3016	0.7244	0.120*	
C5	0.5067 (6)	0.5111 (5)	0.7187 (3)	0.0844 (12)	
H5	0.5823	0.5401	0.7796	0.101*	
C6	0.2658 (7)	0.8617 (6)	0.5449 (5)	0.115 (2)	
H6A	0.3843	0.8432	0.5566	0.137*	
H6B	0.2433	0.9242	0.4796	0.137*	
C7	0.2201 (7)	0.9415 (5)	0.6454 (6)	0.114 (2)	
H7A	0.2841	1.0304	0.6551	0.137*	
H7B	0.1036	0.9677	0.6318	0.137*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0546 (7)	0.0504 (6)	0.0705 (7)	0.000	0.0085 (4)	0.000
S1	0.0748 (8)	0.1960 (17)	0.0662 (7)	0.0179 (8)	−0.0137 (5)	0.0078 (7)
O1	0.178 (4)	0.106 (2)	0.101 (3)	0.005 (3)	0.046 (3)	0.039 (2)
O2	0.107 (12)	0.31 (3)	0.29 (2)	0.088 (14)	0.074 (13)	0.21 (2)
O2′	0.185 (18)	0.188 (15)	0.198 (14)	−0.139 (14)	0.098 (13)	−0.118 (12)
N1	0.0638 (17)	0.0756 (18)	0.0492 (14)	−0.0023 (13)	−0.0020 (12)	−0.0074 (12)
N2	0.077 (3)	0.061 (2)	0.103 (4)	0.000	0.030 (3)	0.000
C1	0.0612 (19)	0.104 (3)	0.0463 (16)	−0.0205 (18)	0.0108 (13)	−0.0068 (17)
C2	0.100 (3)	0.119 (4)	0.069 (2)	−0.050 (3)	0.025 (2)	−0.036 (3)
C3	0.155 (5)	0.076 (3)	0.108 (4)	−0.032 (3)	0.071 (4)	−0.020 (3)

C4	0.143 (5)	0.076 (3)	0.089 (3)	0.022 (3)	0.043 (3)	0.008 (2)
C5	0.098 (3)	0.090 (3)	0.062 (2)	0.016 (2)	−0.0026 (19)	−0.005 (2)
C6	0.100 (3)	0.137 (4)	0.113 (4)	0.027 (3)	0.037 (3)	0.071 (4)
C7	0.095 (3)	0.082 (3)	0.174 (5)	0.024 (3)	0.052 (4)	0.054 (3)

Geometric parameters (Å, º)

Cl1—O2′	1.381 (4)	C2—C3	1.357 (8)
Cl1—O1^{i}	1.383 (3)	C2—H2A	0.9300
Cl1—O1	1.383 (3)	C3—C4	1.329 (10)
Cl1—O2	1.391 (4)	C3—H3	0.9300
S1—C1	1.763 (4)	C4—C5	1.387 (7)
S1—C6	1.820 (7)	C4—H4	0.9300
N1—C1	1.319 (4)	C5—H5	0.9300
N1—C5	1.319 (5)	C6—C7	1.500 (9)
N2—C7	1.475 (6)	C6—H6A	0.9700
N2—C7^{ii}	1.475 (6)	C6—H6B	0.9700
N2—H2	0.8600	C7—H7A	0.9700
C1—C2	1.395 (7)	C7—H7B	0.9700
O2′—Cl1—O1^{i}	104.9 (8)	C2—C3—H3	120.1
O2′—Cl1—O1	113.1 (4)	C3—C4—C5	118.7 (5)
O1^{i}—Cl1—O1	112.4 (3)	C3—C4—H4	120.6
O2′—Cl1—O2	111.9 (4)	C5—C4—H4	120.6
O1^{i}—Cl1—O2	102.5 (12)	N1—C5—C4	123.8 (5)
O1—Cl1—O2	111.4 (4)	N1—C5—H5	118.1
O2^{i}—Cl1—O2	117 (2)	C4—C5—H5	118.1
C1—S1—C6	102.3 (2)	C7—C6—S1	115.4 (3)
C1—N1—C5	116.3 (4)	C7—C6—H6A	108.4
C7—N2—C7^{ii}	115.2 (6)	S1—C6—H6A	108.4
C7—N2—H2	108.5	C7—C6—H6B	108.4
C7^{ii}—N2—H2	108.5	S1—C6—H6B	108.4
N1—C1—C2	123.3 (4)	H6A—C6—H6B	107.5
N1—C1—S1	118.1 (3)	N2—C7—C6	112.9 (4)
C2—C1—S1	118.6 (3)	N2—C7—H7A	109.0
C3—C2—C1	118.1 (4)	C6—C7—H7A	109.0
C3—C2—H2A	121.0	N2—C7—H7B	109.0
C1—C2—H2A	121.0	C6—C7—H7B	109.0
C4—C3—C2	119.8 (5)	H7A—C7—H7B	107.8
C4—C3—H3	120.1		
C5—N1—C1—C2	−0.5 (5)	C2—C3—C4—C5	−0.9 (8)
C5—N1—C1—S1	179.2 (3)	C1—N1—C5—C4	−0.3 (6)
C6—S1—C1—N1	25.3 (3)	C3—C4—C5—N1	1.0 (8)
C6—S1—C1—C2	−155.0 (3)	C1—S1—C6—C7	−94.7 (4)
N1—C1—C2—C3	0.6 (6)	C7^{ii}—N2—C7—C6	154.1 (5)

S1—C1—C2—C3	−179.1 (3)	S1—C6—C7—N2	58.4 (5)
C1—C2—C3—C4	0.1 (7)		

Symmetry codes: (i) −*x*+3/2, *y*, −*z*+3/2; (ii) −*x*+1/2, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···N1	0.86	2.11	2.832 (5)	141