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Dipyridinium 2,2′-dithiodinicotinate

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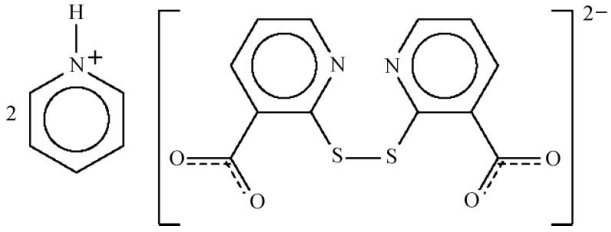
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.009 Å; R factor = 0.092; wR factor = 0.269; data-to-parameter ratio = 12.7.

The dianion of the title salt, $2C_5H_6N^+\cdot C_{12}H_6N_2O_4S_2^{2-}$, lies on a special position of 2 site symmetry that relates one thionicotinate part to the other, and the dihedral angle between the niotinate planes is 89.2 (2)°. The pyridinium cations are hydrogen bonded to the carboxylate group by way of N—H···O links.

Related literature

The structure is a non-merohedral twin; for the program to model twinned crystal structures, see: Spek (2003). For 1,1′-dithio-2,2′-dinicotinic acid, see: Zhu *et al.* (2002). For the methyl, ethyl and *n*-butyl esters, see: Cindrić *et al.* (2001); Toma *et al.* (2004).



Experimental

Crystal data

$2C_5H_6N^+\cdot C_{12}H_6N_2O_4S_2^{2-}$
M_r = 466.52
Monoclinic, $C2/c$
a = 7.9621 (3) Å
b = 12.3354 (4) Å
c = 21.5057 (8) Å
β = 95.917 (2)°
V = 2100.9 (1) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 123 K
0.28 × 0.16 × 0.08 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.923, T_{max} = 0.977
6726 measured reflections
1855 independent reflections
1496 reflections with $I > 2\sigma(I)$
R_{int} = 0.101

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.092
$wR(F^2)$ = 0.269
S = 1.59
1855 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2	0.88	1.71	2.586 (7)	174

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; 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, 2009).

We thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2170).

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supplementary materials

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Dipyridinium 2,2'-dithiodinicotinate

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Experimental

The title compound was isolated as one of the by-products when 2-(3,5-di-*tert*-butyl-4-hydroxybenzylsulfanyl)nicotinic acid (0.37 g, 1 mmol) and thiocarbohydrazide (0.10 g, 1 mmol) were reacted in pyridine (10 ml) for 3 h. The product from a cool mixture was collected and recrystallized from pyridine

Refinement

The specimen used in the diffraction measurements is a multiply-twinned crystal; twinning was evident when examined by the RLATT routine of the data collection software, with a major of about 60%. The diffraction images were integrated on the major component.

The structure initially refined to an *R*> index of 13%. The structure is a non-merohedral twin, as suggested by *PLATON* (Spek, 2003). The intensities were de-twinned by the *TwinRotMat* routine.

The carbon- and nitrogen-bound H-atoms were placed in calculated positions (C—H 0.95 Å, N–H 0.88 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 times *U*(C,*N*).

Figures

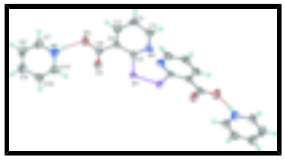

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of 2(C_5H_6N) ($C_{12}H_6N_2O_4S_2$) at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

Dipyridinium 2,2'-dithiodinicotinate

Crystal data

$2C_5H_6N^+ \cdot C_{12}H_6N_2O_4S_2^{2-}$	F_{000} = 968
M_r = 466.52	D_x = 1.475 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 1585 reflections
a = 7.9621 (3) Å	θ = 3.1–24.0º
b = 12.3354 (4) Å	μ = 0.29 mm^{-1}
c = 21.5057 (8) Å	*T* = 123 K
β = 95.917 (2)º	Chip, light yellow
V = 2100.9 (1) Å^3	0.28 × 0.16 × 0.08 mm

$Z = 4$

Data collection

Bruker SMART APEX diffractometer	1855 independent reflections
Radiation source: fine-focus sealed tube	1496 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.101$
$T = 123$ K	$\theta_{max} = 25.0^{o}$
ω scans	$\theta_{min} = 1.9^{o}$
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	$h = -9\rightarrow9$
$T_{min} = 0.923$, $T_{max} = 0.977$	$k = -14\rightarrow14$
6726 measured reflections	$l = -25\rightarrow25$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.092$	H-atom parameters constrained
$wR(F^2) = 0.269$	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 5P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.59$	$(\Delta/\sigma)_{max} = 0.001$
1855 reflections	$\Delta\rho_{max} = 0.47$ e Å^{-3}
146 parameters	$\Delta\rho_{min} = -0.57$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.5434 (2)	0.61088 (12)	0.29627 (7)	0.0213 (5)
O1	0.6450 (6)	0.6042 (4)	0.4204 (2)	0.0317 (12)
O2	0.5265 (6)	0.6888 (4)	0.4968 (2)	0.0336 (12)
N1	0.3073 (7)	0.7643 (4)	0.2874 (2)	0.0249 (12)
N2	0.7396 (7)	0.5816 (4)	0.5721 (3)	0.0278 (13)
H2	0.6690	0.6218	0.5478	0.033*
C1	0.2040 (9)	0.8378 (5)	0.3103 (3)	0.0299 (16)
H1	0.1300	0.8778	0.2814	0.036*
C2	0.1994 (8)	0.8583 (5)	0.3731 (3)	0.0271 (15)
H2A	0.1240	0.9102	0.3873	0.033*
C3	0.3092 (8)	0.8001 (5)	0.4142 (3)	0.0249 (14)
H3	0.3091	0.8116	0.4579	0.030*
C4	0.4190 (8)	0.7255 (5)	0.3929 (3)	0.0204 (13)
C5	0.4125 (8)	0.7097 (5)	0.3280 (3)	0.0191 (13)
C6	0.5418 (8)	0.6678 (5)	0.4383 (3)	0.0227 (14)
C7	0.7634 (9)	0.5987 (6)	0.6336 (3)	0.0311 (16)

H7	0.7030	0.6557	0.6510	0.037*
C8	0.8721 (9)	0.5368 (6)	0.6728 (3)	0.0342 (17)
H8	0.8862	0.5512	0.7164	0.041*
C9	0.9605 (9)	0.4535 (6)	0.6481 (3)	0.0325 (16)
H9	1.0335	0.4081	0.6744	0.039*
C10	0.9398 (10)	0.4379 (6)	0.5841 (3)	0.0329 (16)
H10	1.0030	0.3842	0.5651	0.039*
C11	0.8259 (9)	0.5018 (5)	0.5485 (3)	0.0280 (15)
H11	0.8078	0.4883	0.5049	0.034*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0232 (9)	0.0189 (8)	0.0212 (8)	0.0044 (6)	−0.0002 (6)	0.0000 (6)
O1	0.035 (3)	0.036 (3)	0.023 (2)	0.016 (2)	−0.003 (2)	−0.0003 (19)
O2	0.038 (3)	0.040 (3)	0.022 (2)	0.017 (2)	−0.002 (2)	0.000 (2)
N1	0.023 (3)	0.021 (3)	0.029 (3)	0.010 (2)	−0.003 (2)	0.000 (2)
N2	0.025 (3)	0.029 (3)	0.029 (3)	0.001 (2)	−0.003 (2)	0.000 (2)
C1	0.029 (4)	0.020 (3)	0.039 (4)	0.004 (3)	−0.003 (3)	0.001 (3)
C2	0.023 (3)	0.018 (3)	0.040 (4)	0.000 (3)	0.004 (3)	−0.003 (3)
C3	0.027 (3)	0.023 (3)	0.027 (3)	0.004 (3)	0.009 (3)	−0.001 (3)
C4	0.019 (3)	0.016 (3)	0.025 (3)	−0.003 (2)	−0.003 (3)	0.002 (2)
C5	0.020 (3)	0.018 (3)	0.019 (3)	0.000 (2)	0.003 (2)	0.003 (2)
C6	0.024 (3)	0.017 (3)	0.026 (3)	−0.001 (3)	−0.001 (3)	−0.001 (2)
C7	0.027 (4)	0.037 (4)	0.028 (4)	−0.002 (3)	−0.003 (3)	−0.006 (3)
C8	0.024 (4)	0.053 (5)	0.024 (3)	0.001 (3)	−0.005 (3)	−0.001 (3)
C9	0.021 (3)	0.037 (4)	0.038 (4)	−0.003 (3)	−0.005 (3)	0.004 (3)
C10	0.034 (4)	0.025 (3)	0.039 (4)	0.002 (3)	−0.002 (3)	−0.001 (3)
C11	0.028 (4)	0.029 (3)	0.027 (3)	0.002 (3)	0.002 (3)	−0.002 (3)

Geometric parameters (Å, °)

S1—C5	1.785 (6)	C3—C4	1.380 (9)
S1—S1^{i}	2.038 (3)	C3—H3	0.9500
O1—C6	1.226 (8)	C4—C5	1.405 (9)
O2—C6	1.302 (8)	C4—C6	1.490 (8)
N1—C5	1.329 (8)	C7—C8	1.375 (10)
N1—C1	1.350 (8)	C7—H7	0.9500
N2—C11	1.330 (9)	C8—C9	1.383 (10)
N2—C7	1.333 (8)	C8—H8	0.9500
N2—H2	0.8800	C9—C10	1.384 (10)
C1—C2	1.376 (9)	C9—H9	0.9500
C1—H1	0.9500	C10—C11	1.372 (10)
C2—C3	1.380 (9)	C10—H10	0.9500
C2—H2A	0.9500	C11—H11	0.9500
C5—S1—S1^{i}	102.7 (2)	C4—C5—S1	120.8 (5)
C5—N1—C1	117.8 (6)	O1—C6—O2	124.2 (6)
C11—N2—C7	117.9 (6)	O1—C6—C4	121.1 (6)

C11—N2—H2	121.0	O2—C6—C4	114.7 (5)
C7—N2—H2	121.0	N2—C7—C8	122.4 (7)
N1—C1—C2	124.0 (6)	N2—C7—H7	118.8
N1—C1—H1	118.0	C8—C7—H7	118.8
C2—C1—H1	118.0	C7—C8—C9	119.4 (7)
C1—C2—C3	117.0 (6)	C7—C8—H8	120.3
C1—C2—H2A	121.5	C9—C8—H8	120.3
C3—C2—H2A	121.5	C8—C9—C10	118.2 (6)
C4—C3—C2	121.0 (6)	C8—C9—H9	120.9
C4—C3—H3	119.5	C10—C9—H9	120.9
C2—C3—H3	119.5	C11—C10—C9	118.6 (7)
C3—C4—C5	117.6 (6)	C11—C10—H10	120.7
C3—C4—C6	119.8 (6)	C9—C10—H10	120.7
C5—C4—C6	122.6 (6)	N2—C11—C10	123.4 (6)
N1—C5—C4	122.5 (6)	N2—C11—H11	118.3
N1—C5—S1	116.7 (5)	C10—C11—H11	118.3
C5—N1—C1—C2	−1.0 (10)	$S1^{i}$—S1—C5—C4	172.6 (5)
N1—C1—C2—C3	0.6 (10)	C3—C4—C6—O1	−177.2 (6)
C1—C2—C3—C4	0.5 (9)	C5—C4—C6—O1	0.9 (9)
C2—C3—C4—C5	−1.2 (9)	C3—C4—C6—O2	4.3 (8)
C2—C3—C4—C6	177.1 (6)	C5—C4—C6—O2	−177.5 (6)
C1—N1—C5—C4	0.2 (9)	C11—N2—C7—C8	−0.4 (10)
C1—N1—C5—S1	178.5 (5)	N2—C7—C8—C9	−0.1 (11)
C3—C4—C5—N1	0.9 (9)	C7—C8—C9—C10	2.1 (11)
C6—C4—C5—N1	−177.3 (5)	C8—C9—C10—C11	−3.5 (10)
C3—C4—C5—S1	−177.4 (5)	C7—N2—C11—C10	−1.2 (10)
C6—C4—C5—S1	4.4 (8)	C9—C10—C11—N2	3.2 (11)
$S1^{i}$—S1—C5—N1	−5.7 (5)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.88	1.71	2.586 (7)	174

Fig. 1