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Bis[1-benzyl-4-(dimethylamino)-pyridinium] bis(2,2-dicyanoethylene-1,1-dithiolato-κ^2S,S')nickelate(II)

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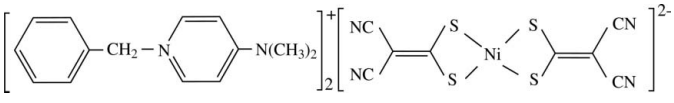
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.005 Å; R factor = 0.037; wR factor = 0.128; data-to-parameter ratio = 14.1.

A new ion-pair complex, $(C_{14}H_{17}N_2)_2[Ni(C_4N_2S_2)_2]$ or $(BzDMAP)_2[Ni(i\text{-}mnt)_2]$, where BzDMAP is 1-benzyl-4-(dimethylamino)pyridinium and i-mnt is 2,2-dicyanoethylene-1,1-dithiolate, has been prepared. The $[Ni(i\text{-}mnt)_2]^{2-}$ anion, which is located on an inversion center, exhibits a planar structure. The crystal packing is governed by short C—H···N and C—H···S contacts between anions and cations and by π–π stacking interactions between the phenyl groups of the cations (the centroid–centroid distance is 3.802 Å; symmetry code: $-x+2, -y+2, -z$).

Related literature

For the i-mnt complex of square-planar geometry with substituted pyridinium as counter-ion, see: Liu *et al.* (2006).



Experimental

Crystal data

$(C_{14}H_{17}N_2)_2[Ni(C_4N_2S_2)_2]$
M_r = 765.66
Triclinic, $P\overline{1}$
a = 8.0456 (12) Å
b = 9.6829 (15) Å
c = 13.202 (2) Å
α = 76.134 (2)°
β = 78.312 (2)°
γ = 68.834 (2)°
V = 923.5 (2) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.79 mm^{-1}
T = 291 (2) K
0.38 × 0.22 × 0.20 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.798, T_{max} = 0.858
4642 measured reflections
3180 independent reflections
2431 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.128
S = 1.07
3180 reflections
225 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11B···N1^{i}	0.97	2.62	3.368 (4)	134
C12—H12···N1^{ii}	0.93	2.59	3.477 (4)	161
C16—H16···S1^{iii}	0.93	2.86	3.750 (4)	160

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: GK2073).

References

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Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Liu, M.-G., Li, X.-Y., Lin, L.-F. & Ni, C.-L. (2006). *Acta Cryst.* E**62**, m2919–m2921.
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supplementary materials

Acta Cryst. (2007). E**63**, m1762 [**doi:10.1107/S1600536807025251**]

Bis[1-benzyl-4-(dimethylamino)pyridinium] bis(2,2-dicyanoethylene-1,1-dithiolato-κ^2S,S')nickelate(II)

C.-W. Feng, X.-R. Li, Y. Hou and C.-L. Ni

Comment

As shown in Fig.1 the asymmetric unit of the title compound consists of one $[BzDMAP]^+$ cation and one-half of $Ni(i\text{-}mnt)_2$ anion located on an inversion center. The nickel(II) ion is coordinated by four S atoms from two i-mnt ligands and the complex anion $Ni(i\text{-}mnt)_2$ is in square planar configuration. The $[BzDMAP]^+$ cation adopts a conformation where both the phenyl ring and the pyridine rings are twisted with respect to the C10\C11\N3 reference plane with the dihedral angles of 86.4 (3) and 89.7 (3)°, respectively.

Molecules are linked by C—H···N and C—H···S hydrogen bonds between anions and cations and π···π stacking interaction between cations (Fig. 2).

Experimental

The title compound was prepared by the direct reaction of $NiCl_2{\cdot}6H_2O$, K_2(i-mnt)and (BzDMAP)Br in methanol. The brown block single crystals were obtained by slow evaporation of a CH_3CN solution at room temperature for about two weeks.

Refinement

All H atoms were placed in geometrically calculated positions (C—H = 0.93–0.97 Å) and refined as riding on their parent atoms with U_{iso} = 1.2 U_{eq}(C)

Figures

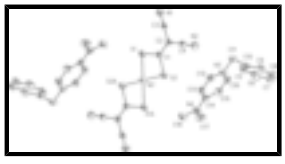

Fig. 1. The molecular structure of the title compound, with atom labels and 30% probability displacement ellipsoids for non-H atoms.

Fig. 2. The packing of the title compound viewed down the *c* axis. Hydrogen atoms have been omitted for clarity.

Bis[1-benzyl-4-(dimethylamino)pyridinium] bis(2,2-dicyanoethylene-1,1-dithiolato-κ^2*S*,*S*')nickelate(II)

Crystal data

$(C_{14}H_{17}N_2)_2[Ni(C_4N_2S_2)_2]$	$Z = 1$
$M_r = 765.66$	$F_{000} = 398$
Triclinic, $P\overline{1}$	$D_x = 1.377$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.0456$ (12) Å	Cell parameters from 2430 reflections
$b = 9.6829$ (15) Å	$\theta = 2.3$–26.9º
$c = 13.202$ (2) Å	$\mu = 0.79$ mm^{-1}
$\alpha = 76.134$ (2)º	$T = 291$ (2) K
$\beta = 78.312$ (2)º	Block, brown
$\gamma = 68.834$ (2)º	0.38 × 0.22 × 0.20 mm
$V = 923.5$ (2) Å^3	

Data collection

Bruker SMART APEX CCD diffractometer	2431 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.019$
Monochromator: graphite	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -5 \rightarrow 9$
$T_{min} = 0.798$, $T_{max} = 0.858$	$k = -11 \rightarrow 11$
4642 measured reflections	$l = -14 \rightarrow 15$
3180 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H-atom parameters constrained
$wR(F^2) = 0.128$	$w = 1/[\sigma^2(F_o^2) + (0.08P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} = 0.001$
3180 reflections	$\Delta\rho_{max} = 0.40$ e Å^{-3}
225 parameters	$\Delta\rho_{min} = -0.21$ 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}
Ni1	0.5000	0.0000	0.5000	0.0519 (2)
S1	0.37050 (10)	0.05129 (8)	0.35743 (6)	0.0583 (2)
S2	0.63405 (10)	0.15279 (9)	0.39411 (6)	0.0613 (2)
N1	0.2591 (4)	0.2793 (3)	0.0912 (2)	0.0764 (8)
N2	0.6676 (5)	0.4409 (4)	0.1454 (2)	0.0951 (10)
N3	0.8270 (3)	0.6225 (3)	0.25242 (19)	0.0604 (6)
N4	0.8293 (3)	0.3522 (3)	0.5458 (2)	0.0673 (7)
C1	0.4950 (3)	0.1708 (3)	0.3041 (2)	0.0499 (6)
C2	0.4823 (4)	0.2637 (3)	0.2104 (2)	0.0525 (7)
C3	0.3600 (4)	0.2714 (3)	0.1435 (2)	0.0566 (7)
C4	0.5878 (4)	0.3613 (3)	0.1738 (2)	0.0634 (8)
C5	1.1483 (5)	0.7158 (4)	0.0913 (2)	0.0750 (9)
H5	1.1762	0.6217	0.0739	0.090*
C6	1.2810 (5)	0.7788 (5)	0.0786 (3)	0.0907 (11)
H6	1.3980	0.7271	0.0527	0.109*
C7	1.2422 (6)	0.9167 (5)	0.1037 (3)	0.0901 (11)
H7	1.3328	0.9582	0.0958	0.108*
C8	1.0716 (6)	0.9932 (4)	0.1402 (3)	0.0893 (11)
H8	1.0448	1.0881	0.1560	0.107*
C9	0.9375 (5)	0.9307 (4)	0.1541 (3)	0.0746 (9)
H9	0.8210	0.9831	0.1802	0.089*
C10	0.9749 (4)	0.7903 (3)	0.1295 (2)	0.0611 (7)
C11	0.8306 (5)	0.7209 (4)	0.1471 (2)	0.0785 (10)
H11A	0.7151	0.7998	0.1416	0.094*
H11B	0.8514	0.6619	0.0931	0.094*
C12	0.9269 (4)	0.4776 (4)	0.2663 (2)	0.0625 (8)
H12	0.9952	0.4379	0.2077	0.075*
C13	0.9340 (4)	0.3853 (3)	0.3610 (2)	0.0616 (7)
H13	1.0074	0.2851	0.3666	0.074*
C14	0.8300 (4)	0.4407 (3)	0.4516 (2)	0.0564 (7)
C15	0.7280 (4)	0.5943 (4)	0.4341 (2)	0.0678 (8)
H15	0.6574	0.6383	0.4905	0.081*
C16	0.7305 (4)	0.6790 (4)	0.3371 (3)	0.0709 (8)
H16	0.6625	0.7807	0.3286	0.085*
C17	0.7161 (5)	0.4124 (5)	0.6370 (3)	0.0962 (12)
H17A	0.7433	0.4977	0.6448	0.144*
H17B	0.7385	0.3362	0.6991	0.144*
H17C	0.5920	0.4428	0.6273	0.144*
C18	0.9379 (5)	0.1947 (4)	0.5619 (3)	0.0876 (10)
H18A	0.9062	0.1439	0.5184	0.131*
H18B	0.9170	0.1500	0.6344	0.131*
H18C	1.0626	0.1857	0.5435	0.131*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0539 (3)	0.0459 (3)	0.0456 (3)	−0.0108 (2)	−0.0076 (2)	0.0030 (2)
S1	0.0626 (5)	0.0545 (4)	0.0535 (5)	−0.0221 (4)	−0.0120 (3)	0.0065 (3)
S2	0.0632 (5)	0.0658 (5)	0.0522 (5)	−0.0248 (4)	−0.0151 (3)	0.0064 (3)
N1	0.089 (2)	0.090 (2)	0.0581 (17)	−0.0432 (17)	−0.0213 (15)	0.0035 (14)
N2	0.132 (3)	0.105 (2)	0.072 (2)	−0.080 (2)	−0.0298 (18)	0.0162 (17)
N3	0.0685 (15)	0.0629 (16)	0.0529 (15)	−0.0263 (13)	−0.0175 (11)	−0.0009 (12)
N4	0.0744 (17)	0.0784 (18)	0.0507 (15)	−0.0310 (15)	−0.0118 (12)	−0.0019 (13)
C1	0.0479 (15)	0.0435 (14)	0.0522 (16)	−0.0072 (12)	−0.0036 (11)	−0.0120 (13)
C2	0.0577 (16)	0.0528 (16)	0.0461 (16)	−0.0195 (13)	−0.0048 (12)	−0.0065 (13)
C3	0.0696 (19)	0.0539 (17)	0.0424 (16)	−0.0255 (15)	−0.0019 (14)	0.0022 (13)
C4	0.081 (2)	0.0644 (19)	0.0468 (17)	−0.0340 (18)	−0.0112 (14)	0.0042 (14)
C5	0.102 (3)	0.0560 (18)	0.0533 (19)	−0.0225 (19)	0.0051 (17)	−0.0027 (15)
C6	0.086 (3)	0.093 (3)	0.061 (2)	−0.018 (2)	0.0101 (18)	0.010 (2)
C7	0.101 (3)	0.096 (3)	0.074 (3)	−0.051 (3)	−0.011 (2)	0.011 (2)
C8	0.123 (3)	0.072 (2)	0.079 (3)	−0.044 (2)	−0.015 (2)	−0.007 (2)
C9	0.079 (2)	0.061 (2)	0.067 (2)	−0.0103 (17)	−0.0073 (16)	−0.0047 (16)
C10	0.079 (2)	0.0591 (18)	0.0398 (15)	−0.0207 (16)	−0.0159 (13)	0.0036 (13)
C11	0.099 (3)	0.085 (2)	0.058 (2)	−0.037 (2)	−0.0327 (17)	0.0049 (17)
C12	0.0691 (19)	0.0636 (19)	0.0571 (19)	−0.0233 (16)	−0.0019 (14)	−0.0182 (15)
C13	0.0631 (18)	0.0534 (17)	0.0622 (19)	−0.0143 (14)	−0.0066 (14)	−0.0082 (15)
C14	0.0563 (16)	0.0635 (18)	0.0546 (18)	−0.0243 (14)	−0.0109 (13)	−0.0099 (14)
C15	0.071 (2)	0.066 (2)	0.059 (2)	−0.0138 (16)	−0.0022 (15)	−0.0158 (16)
C16	0.071 (2)	0.0573 (19)	0.075 (2)	−0.0087 (16)	−0.0132 (16)	−0.0118 (17)
C17	0.108 (3)	0.132 (4)	0.051 (2)	−0.050 (3)	−0.0025 (19)	−0.012 (2)
C18	0.106 (3)	0.075 (2)	0.078 (2)	−0.033 (2)	−0.030 (2)	0.0153 (19)

Geometric parameters (Å, °)

Ni1—S1^{i}	2.2019 (8)	C7—H7	0.9300
Ni1—S1	2.2019 (8)	C8—C9	1.379 (5)
Ni1—S2^{i}	2.2037 (8)	C8—H8	0.9300
Ni1—S2	2.2037 (8)	C9—C10	1.385 (4)
S1—C1	1.732 (3)	C9—H9	0.9300
S2—C1	1.730 (3)	C10—C11	1.495 (4)
N1—C3	1.140 (4)	C11—H11A	0.9700
N2—C4	1.126 (4)	C11—H11B	0.9700
N3—C12	1.330 (4)	C12—C13	1.350 (4)
N3—C16	1.337 (4)	C12—H12	0.9300
N3—C11	1.487 (4)	C13—C14	1.413 (4)
N4—C14	1.331 (4)	C13—H13	0.9300
N4—C18	1.446 (4)	C14—C15	1.404 (4)
N4—C17	1.459 (4)	C15—C16	1.346 (4)
C1—C2	1.342 (4)	C15—H15	0.9300
C2—C3	1.421 (4)	C16—H16	0.9300

C2—C4	1.429 (4)	C17—H17A	0.9600
C5—C10	1.372 (4)	C17—H17B	0.9600
C5—C6	1.375 (5)	C17—H17C	0.9600
C5—H5	0.9300	C18—H18A	0.9600
C6—C7	1.364 (5)	C18—H18B	0.9600
C6—H6	0.9300	C18—H18C	0.9600
C7—C8	1.354 (5)		
$S1^{i}$—Ni1—S1	180.0	C5—C10—C9	118.3 (3)
$S1^{i}$—Ni1—$S2^{i}$	78.96 (3)	C5—C10—C11	121.2 (3)
S1—Ni1—$S2^{i}$	101.04 (3)	C9—C10—C11	120.5 (3)
$S1^{i}$—Ni1—S2	101.04 (3)	N3—C11—C10	111.0 (2)
S1—Ni1—S2	78.96 (3)	N3—C11—H11A	109.4
$S2^{i}$—Ni1—S2	180.00 (3)	C10—C11—H11A	109.4
C1—S1—Ni1	86.41 (10)	N3—C11—H11B	109.4
C1—S2—Ni1	86.40 (10)	C10—C11—H11B	109.4
C12—N3—C16	118.0 (3)	H11A—C11—H11B	108.0
C12—N3—C11	121.1 (3)	N3—C12—C13	123.3 (3)
C16—N3—C11	120.8 (3)	N3—C12—H12	118.3
C14—N4—C18	122.0 (3)	C13—C12—H12	118.3
C14—N4—C17	120.3 (3)	C12—C13—C14	120.1 (3)
C18—N4—C17	117.7 (3)	C12—C13—H13	120.0
C2—C1—S2	126.1 (2)	C14—C13—H13	120.0
C2—C1—S1	125.8 (2)	N4—C14—C15	123.2 (3)
S2—C1—S1	108.02 (16)	N4—C14—C13	121.9 (3)
C1—C2—C3	121.3 (3)	C15—C14—C13	115.0 (3)
C1—C2—C4	121.9 (3)	C16—C15—C14	121.1 (3)
C3—C2—C4	116.8 (3)	C16—C15—H15	119.4
N1—C3—C2	178.6 (3)	C14—C15—H15	119.4
N2—C4—C2	178.4 (4)	N3—C16—C15	122.5 (3)
C10—C5—C6	120.6 (3)	N3—C16—H16	118.7
C10—C5—H5	119.7	C15—C16—H16	118.7
C6—C5—H5	119.7	N4—C17—H17A	109.5
C7—C6—C5	120.4 (4)	N4—C17—H17B	109.5
C7—C6—H6	119.8	H17A—C17—H17B	109.5
C5—C6—H6	119.8	N4—C17—H17C	109.5
C8—C7—C6	120.0 (4)	H17A—C17—H17C	109.5
C8—C7—H7	120.0	H17B—C17—H17C	109.5
C6—C7—H7	120.0	N4—C18—H18A	109.5
C7—C8—C9	120.2 (4)	N4—C18—H18B	109.5
C7—C8—H8	119.9	H18A—C18—H18B	109.5
C9—C8—H8	119.9	N4—C18—H18C	109.5
C8—C9—C10	120.5 (3)	H18A—C18—H18C	109.5
C8—C9—H9	119.7	H18B—C18—H18C	109.5
C10—C9—H9	119.7		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11B···N1^{ii}	0.97	2.62	3.368 (4)	134
C12—H12···N1^{iii}	0.93	2.59	3.477 (4)	161
C16—H16···S1^{iv}	0.93	2.86	3.750 (4)	160

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

Fig. 1

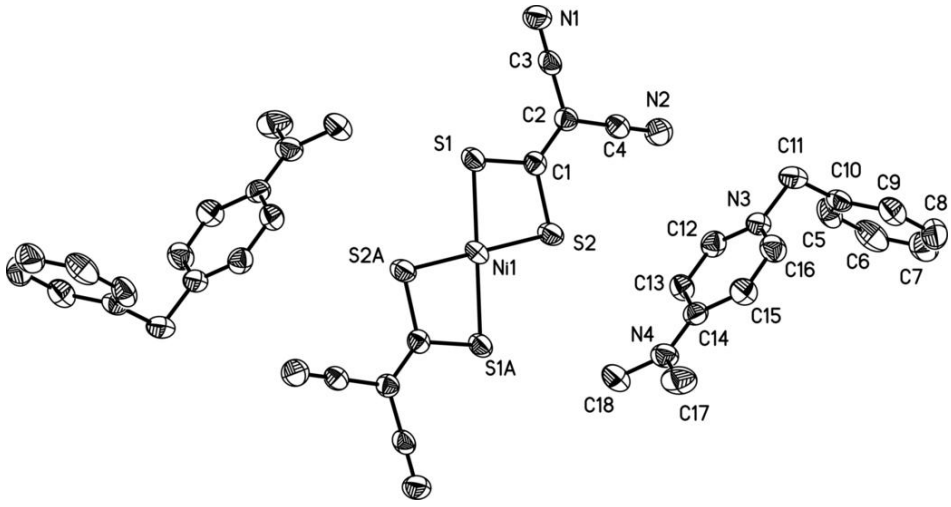
N1
C3
C2
N2
C4
S1
C1
C11
C10
C9
C8
N3
C12
C5
C6
C7
S2A
Ni1
S2
C16
C13
C15
N4
C14
S1A
C18
C17

Fig. 2