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N-(4-Fluorobenzyl)pyridinium bis(2-thioxo-1,3-dithiole-4,5-dithiolato)nickelate(III)

Weijiang Lou,[a] Guozhong Yang,[b] Yang Su[c] and Ruojie Tao[a]*

[a]Institute of Molecular and Crystal Engineering, School of Chemistry and Chemical Engineering, Henan University, Kaifeng, 475001, People's Republic of China, [b]Department of Building Engineering of Henan University, Kaifeng 475001, Henan, People's Republic of China, and [c]Coordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, School of Chemistry and Chemical Engineering, Nanjing University, Nanjing, 210093, People's Republic of China

Correspondence e-mail: rjtao@henu.edu.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.049
wR factor = 0.093
Data-to-parameter ratio = 15.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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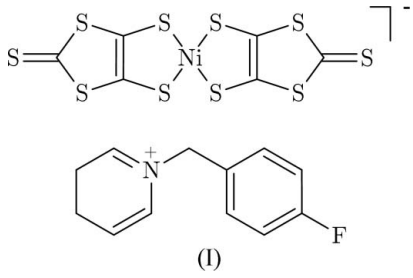
In the title ion-pair compound, $(C_{12}H_{11}FN)[Ni(C_3S_5)_2]$, the Ni^{III} atom is coordinated by four sulfur atoms of the two 2-thioxo-1,3-dithiole-4,5-dithiolate ligands in a square-planar geometry. Weak C—H···S interactions are found, resulting in a three-dimensional supramolecular network.

Comment

Extensive research has focused on the syntheses and characterization of bis-dithiolate–metal complexes and their analogs due to their properties and potential applications, for example as conducting/magnetic materials and in non-linear optics (NLO) (Cassoux, 1999). Among these, 2-thioxo-1,3-dithiole-4,5-dithiolate (dmit) metal complexes are well known as molecular conductors. In order to study the interplay of the magnetic properties, the title compound, (I), was synthesized.



(I)

The asymmetric unit (Fig. 1) is composed of an $Ni^{III}(dmit)_2^-$ anion and an *N*-(4-fluorobenzyl)pyridinium cation. The Ni^{III} ion adopts square-planar coordination geometry with four sulfur atoms of the two dmit ligands.

Experimental

4,5-Bis(thiobenzoyl)-1,3-dithiol-2-thione (812 mg, 2.0 mmol) (Wang *et al.* 1998) was suspended in dry methanol (20 ml). Sodium (92 mg, 4.0 mmol) was added to the above mixture at room temperature under nitrogen, giving a bright red solution to which $NiCl_2{\cdot}6H_2O$ (177 mg, 1 mmol) was added. After 20 min, a solution of I_2 (127 mg,

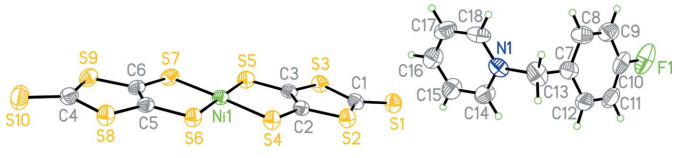


Figure 1
Perspective view of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

0.5 mmol) was added. After another 20 min, a solution of *N*-(4-fluorobenzyl)pyridinium bromide (2 mmol, 0.636 g) in methanol (10 ml) was added, and the solution was stirred for a further 30 min. The reaction product was collected by filtration. Single crystals of the title compound were obtained by evaporation of a dilute acetone solution at room temperature over a period of 1–2 weeks.

Crystal data

$(C_{12}H_{11}FN)[Ni(C_3S_5)_2]$
M_r = 639.67
Monoclinic, $P2_1/c$
a = 8.7766 (17) Å
b = 17.421 (3) Å
c = 16.544 (3) Å
β = 103.774 (4)°
V = 2456.8 (8) Å^3
Z = 4
D_x = 1.729 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.66 mm^{-1}
T = 293 (2) K
Needle, black
0.3 × 0.1 × 0.1 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan *SADABS* (Bruker, 2000)
T_{min} = 0.818, T_{max} = 0.850
12038 measured reflections
4308 independent reflections
3041 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.093
S = 1.03
4308 reflections
280 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—S6	2.1544 (11)	Ni1—S4	2.1569 (12)
Ni1—S5	2.1556 (12)	Ni1—S7	2.1615 (12)
S6—Ni1—S5	179.37 (5)	S6—Ni1—S7	92.87 (4)
S6—Ni1—S4	86.28 (4)	S5—Ni1—S7	87.75 (5)
S5—Ni1—S4	93.10 (4)	S4—Ni1—S7	178.28 (5)

All H atoms were positioned geometrically and refined using a riding model with C—H = 0.93 Å, U_{iso}(H) = 1.2U_{eq}(C) for aromatic and methylene H atoms and C—H = 0.96 Å, U_{iso}(H) = 1.5U_{eq} (C) for methyl H atoms.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); 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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