

Jian-Ding Li,<sup>a</sup> Sheng Li,<sup>b</sup> Yang Su<sup>c</sup> and Ruo-Jie Tao<sup>a\*</sup><sup>a</sup>Institute of Molecular and Crystal Engineering, School of Chemistry and Chemical Engineering, Henan University, Kaifeng 475001, People's Republic of China, <sup>b</sup>Medical College of Henan University, Kaifeng 475001, Henan, People's Republic of China, and <sup>c</sup>Coordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, School of Chemistry and Chemical Engineering, Nanjing University, Nanjing 210093, People's Republic of China

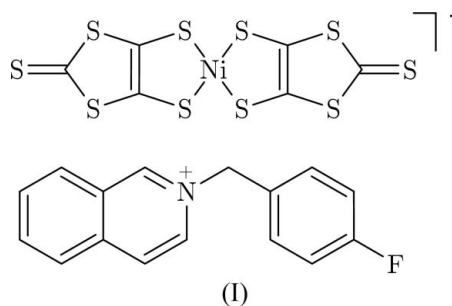
Correspondence e-mail: rjtao@henu.edu.cn

## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å  
 $R$  factor = 0.041  
 $wR$  factor = 0.093  
Data-to-parameter ratio = 14.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.***N*-(4-Fluorobenzyl)isoquinolinium bis(2-thioxo-1,3-dithiole-4,5-dithiolato)nickelate(III)**In the title compound,  $(\text{C}_{16}\text{H}_{13}\text{FN})[\text{Ni}(\text{C}_3\text{S}_5)_2]$ , the  $\text{Ni}^{\text{III}}$  ion exhibits square-planar coordination geometry formed by four S atoms from two 2-thioxo-1,3-dithiole-4,5-dithiolate (dmit) ligands.  $\pi-\pi$  interactions occur between parallel dmit ligands.Received 19 June 2006  
Accepted 4 August 2006

## Comment

Extensive research has been focused on the syntheses and characterization of bis-dithiolate metal complexes and their analogues owing to their potential applications, such as conducting and/or magnetic materials and nonlinear optics (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.

The crystal structure of (I) consists of discrete  $\text{Ni}^{\text{III}}(\text{dmit})_2^-$  anions and *N*-(4-fluorobenzyl)isoquinolinium cations (Fig. 1). The  $\text{Ni}^{\text{III}}$  ion adopts a square-planar coordination geometry with four S atoms of the two dmit ligands. The Ni—S bond lengths range from 2.1465 (10) to 2.1668 (10) Å (Table 1).  $\pi-\pi$  interactions occur between the parallel dmit ligands related by an inversion centre at  $(2 - x, 1 - y, -z)$ ; the face-to-face distance is 3.549 (5) Å.

## Experimental

4,5-Bis(thiobenzoyl)-1,3-dithiol-2-thione (812 mg, 2.0 mmol) (Wang *et al.* 1998) was suspended in methanol (20 ml). Under nitrogen, sodium (92 mg, 4 mmol) was added to the above mixture at room temperature to give a bright-red solution. To this solution,  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  (177 mg, 1 mmol) was added. After 20 min, a methanol solution (10 ml) of  $\text{I}_2$  (127 mg, 0.5 mmol) was added. After another 20 min, a methanol solution (10 ml) of *N*-(4-fluorobenzyl)isoquinolinium bromide (2 mmol, 636 mg) was added to the reaction mixture, and the mixture was stirred for a further 30 min. The product was collected by filtration. Evaporation of an acetone solution of this powder sample at room temperature over a period of two weeks gave single crystals of (I).

Crystal data

(C<sub>16</sub>H<sub>13</sub>FN)[Ni(C<sub>3</sub>S<sub>5</sub>)<sub>2</sub>]  
*M<sub>r</sub>* = 689.64  
 Monoclinic, *P*<sub>21</sub>/*n*  
*a* = 9.390 (2) Å  
*b* = 14.959 (4) Å  
*c* = 18.911 (5) Å  
 β = 94.056 (4)°  
*V* = 2649.6 (11) Å<sup>3</sup>

*Z* = 4  
*D<sub>x</sub>* = 1.729 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 μ = 1.54 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Needle, black  
 0.3 × 0.1 × 0.1 mm

Data collection

Bruker SMART APEX CCD area-  
 detector diffractometer  
 φ and ω scans  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2000)  
*T<sub>min</sub>* = 0.829, *T<sub>max</sub>* = 0.853

12831 measured reflections  
 4651 independent reflections  
 3488 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.079  
 θ<sub>max</sub> = 25.0°

Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.041  
*wR*(*F*<sup>2</sup>) = 0.093  
*S* = 1.00  
 4651 reflections  
 316 parameters

H-atom parameters constrained  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0332*P*)<sup>2</sup>]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> = 0.001  
 Δρ<sub>max</sub> = 0.59 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.47 e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

Ni1—S4	2.1668 (10)	Ni1—S9	2.1648 (9)
Ni1—S5	2.1465 (10)	Ni1—S10	2.1550 (10)
S5—Ni1—S10	84.95 (3)	S5—Ni1—S4	92.86 (4)
S5—Ni1—S9	176.65 (4)	S10—Ni1—S4	177.71 (4)
S10—Ni1—S9	93.30 (4)	S9—Ni1—S4	88.85 (4)

H atoms were placed in calculated positions, with C—H = 0.93 (aromatic) or 0.97 Å (methylene), and refined in riding mode with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C).

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); 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.

The authors express their thanks to the Natural Science Foundation of Henan Province for financial support.

References

Bruker (2000). SADABS, SMART, SAINT and SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Cassoux, P. (1999). *Coord. Chem. Rev.* **185–186**, 213–232.

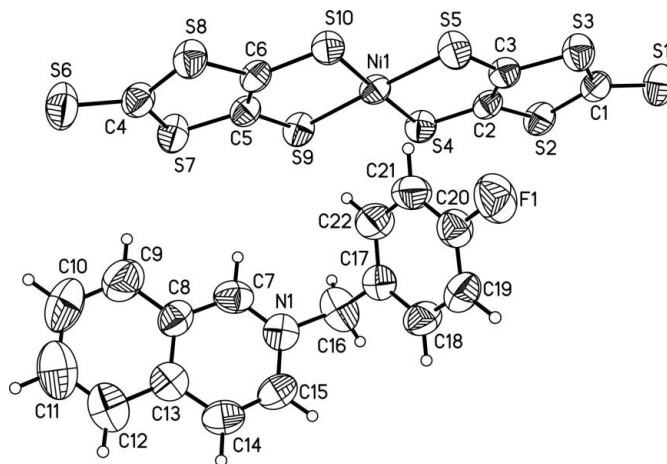


Figure 1

The asymmetric unit of (I), with 50% probability displacement ellipsoids. H atoms are represented by circles of arbitrary size.

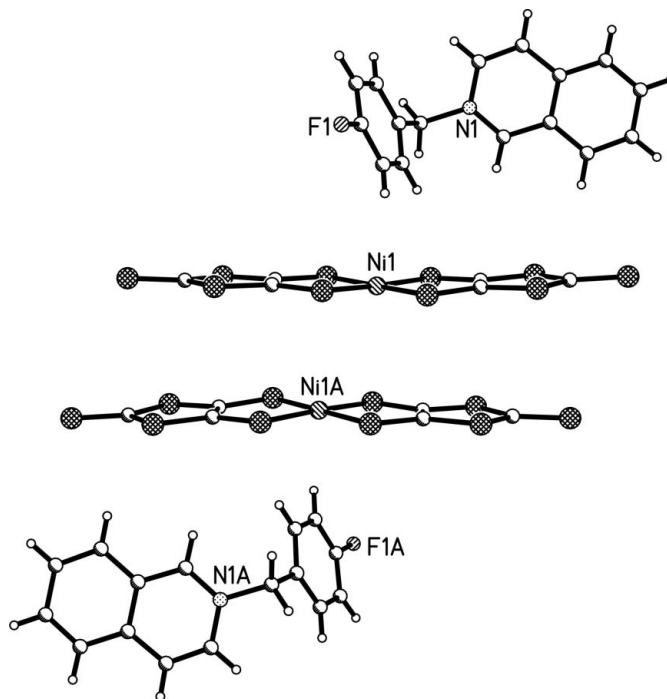


Figure 2

A diagram showing π-π stacking between the dmit ligands [symmetry code: (A) 2 - *x*, 1 - *y*, -*z*].

Wang, C. S., Batsanov, A. S., Bryce, M. R. & Howard, J. A. K. (1998). *Synthesis*, pp. 1615–1618.