

**(*N,N*-Diethyldithiocarbamato- $\kappa^2$ S,S')**bis-  
**(1,10-phenanthroline)sodium(I)****Tihana Mirkovic, Sreekumari P. Nair, Gregory D. Scholes and Alan J. Lough\***

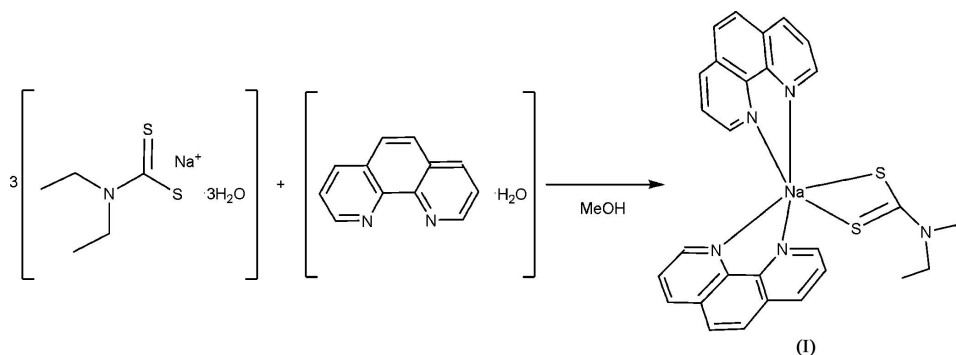
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Correspondence e-mail: [alough@chem.utoronto.ca](mailto:alough@chem.utoronto.ca)**Key indicators**Single-crystal X-ray study  
 $T = 150$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.045  
 $wR$  factor = 0.120  
Data-to-parameter ratio = 17.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title molecule,  $[\text{Na}(\text{C}_5\text{H}_{10}\text{NS}_2)(\text{C}_{12}\text{H}_8\text{N}_2)_2]$ , which has crystallographic twofold symmetry, the coordination geometry of the Na atom is intermediate between octahedral and trigonal prismatic, coordinated by two S atoms of an *N,N*-diethyldithiocarbamate ligand and four chelating N atoms of two 1,10-phenanthroline ligands. In the crystal structure, weak  $\text{C}-\text{H}\cdots\text{S}$  and  $\pi-\pi$  stacking interactions form chains of rings.

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Dithiocarbamate ligands have an ability to complex with a wide variety of transition metals while demonstrating flexibility in coordination mode. An example of the versatility of the thiolate ligand is the dinuclear gold complex  $[\text{Au}_2(\text{C}_{14}\text{H}_{14}\text{P})(\text{C}_5\text{H}_{10}\text{NS}_2)]$  in which diethyldithiocarbamate acts as a bridge between transition metal centres (Bardaji *et al.*, 1994). On the other hand, the monodentate bonding mode of the dithiocarbamate ligand is observed in  $[\text{Au}(\text{C}_{18}\text{H}_{33}\text{P})(\text{C}_5\text{H}_{10}\text{NS}_2)]$  (Ho & Tiekink, 2001), as well as in the sodium complex  $\text{Na}[\text{S}_2\text{CN}(\text{CH}_3)_2]\cdot 2\text{H}_2\text{O}$  (Oskarsson & Ymén, 1983).



The molecular structure of the title compound, (I), is shown in Fig. 1 and selected bond lengths are given in Table 1. The molecule lies on a crystallographic twofold axis which runs through atom Na1 and C13–N3 of the dithiocarbamate ligand. The chelating phenanthroline ligands are bonded in a bidentate fashion through two N atoms, with Na–N distances comparable to those in related complexes (Marek *et al.*, 2003).

In the crystal structure, molecules are connected into dimeric units (Fig. 2) *via* weak  $\text{C}-\text{H}\cdots\text{S}$  interactions (Table 2), forming  $R_2^2(14)$  rings (Bernstein *et al.*, 1995) which are, in turn, reinforced by  $\pi-\pi$  stacking interactions, with a perpendicular distance of 3.368 Å [ $\text{Cg}\cdots\text{Cg}^{\text{iii}} = 3.5584$  (12) Å, where Cg is the centroid of atoms C7–C11/N2; symmetry code: (iii)  $\frac{3}{2} - x, \frac{1}{2} - y, 1 - z$ ]. The dimeric units propagate along [101], forming one-dimensional chains of rings.

## Experimental

The dropwise addition of a sodium diethyldithiocarbamate trihydrate [Na(Ddtc)·3H<sub>2</sub>O] solution (6 mmol in 10 ml of anhydrous methanol) to a solution of 1,10-phenanthroline monohydrate [Phen·H<sub>2</sub>O] (2 mmol in 10 ml of anhydrous methanol) resulted in a white precipitate. Upon recrystallization of the product from anhydrous methanol, colourless crystals of (I) suitable for X-ray analysis were obtained.

### Crystal data

[Na(C<sub>5</sub>H<sub>10</sub>NS<sub>2</sub>)(C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>]  
*M<sub>r</sub>* = 531.66  
 Monoclinic, *C*2/*c*  
*a* = 17.7050 (7) Å  
*b* = 10.5385 (4) Å  
*c* = 15.7010 (4) Å  
 $\beta$  = 115.5570 (19)°  
*V* = 2642.92 (16) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.336 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 9340 reflections  
 $\theta$  = 2.6–27.5°  
 $\mu$  = 0.25 mm<sup>-1</sup>  
*T* = 150 (1) K  
 Block, colourless  
 0.14 × 0.12 × 0.10 mm

### Data collection

Bruker–Nonius KappaCCD diffractometer  
 $\varphi$  scans and  $\omega$  scans with  $\kappa$  offsets  
 Absorption correction: multi-scan (SORTAV; Blessing, 1995)  
*T<sub>min</sub>* = 0.870, *T<sub>max</sub>* = 0.978  
 9340 measured reflections

3026 independent reflections  
 2116 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.047  
 $\theta_{max}$  = 27.5°  
*h* = −22 → 21  
*k* = −13 → 13  
*l* = −20 → 20

### Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.045  
*wR* (*F*<sup>2</sup>) = 0.120  
*S* = 1.04  
 3026 reflections  
 170 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 1.4314P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.001$   
 $\Delta\rho_{max} = 0.32 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.31 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters (Å, °).

S1–Na1	2.9158 (11)	Na1–N1	2.5451 (17)
Na1–N2	2.4628 (18)		
C13–S1–Na1	88.99 (8)	N1–Na1–S1 <sup>i</sup>	112.16 (4)
N2 <sup>i</sup> –Na1–N2	107.22 (9)	N2–Na1–S1	149.93 (5)
N2–Na1–N1	66.67 (5)	N1–Na1–S1	98.21 (4)
N2–Na1–N1 <sup>i</sup>	92.12 (6)	S1 <sup>i</sup> –Na1–S1	61.58 (3)
N1–Na1–N1 <sup>i</sup>	144.76 (9)	S1–C13–S1 <sup>i</sup>	120.45 (16)
N2–Na1–S1 <sup>i</sup>	98.85 (4)		

Symmetry code: (i)  $-x + 1, y, -z + \frac{1}{2}$ .

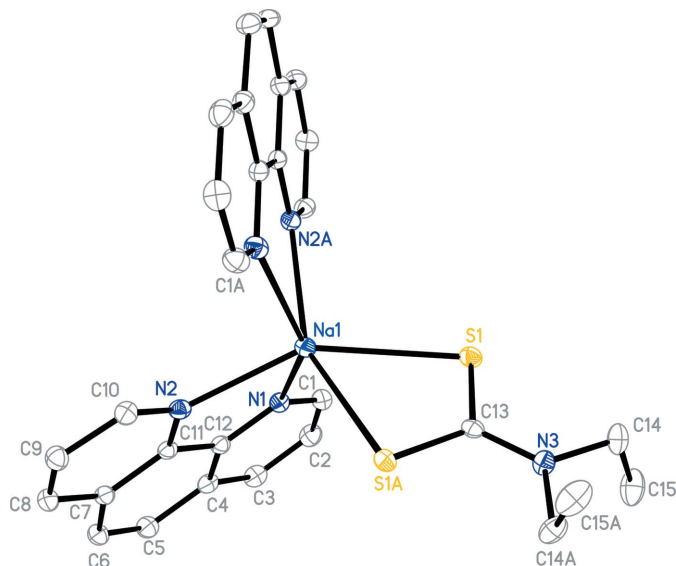
**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C8–H8A...S1 <sup>ii</sup>	0.95	2.84	3.720 (3)	155

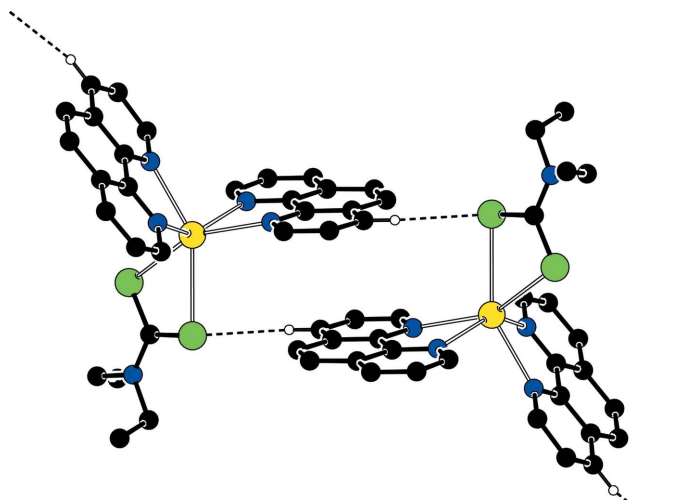
Symmetry code: (ii)  $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$ .

H atoms were placed in calculated positions, with C–H distances of 0.95, 0.99 or 0.98 Å (for methyl groups). They were included in the refinement in the riding-model approximation, with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C) or 1.5*U*<sub>eq</sub>(C) for methyl H atoms.



**Figure 1**

The molecular structure of (I), showing 30% probability displacement ellipsoids. The suffix A corresponds to symmetry code (i) in Table 1. H atoms have been omitted.



**Figure 2**

A partial packing plot (Spek, 2003) of (I), showing hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2001); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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