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Bis[2,5,8,11,14-pentaoxabicyclo[13.4.0]-nonadeca-1(15),16,18-triene]potassium(I) bis(2-thioxo-4,5-dihydro-1,3-dithiole-4,5-dithiolato)nickel(III)

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Key indicators

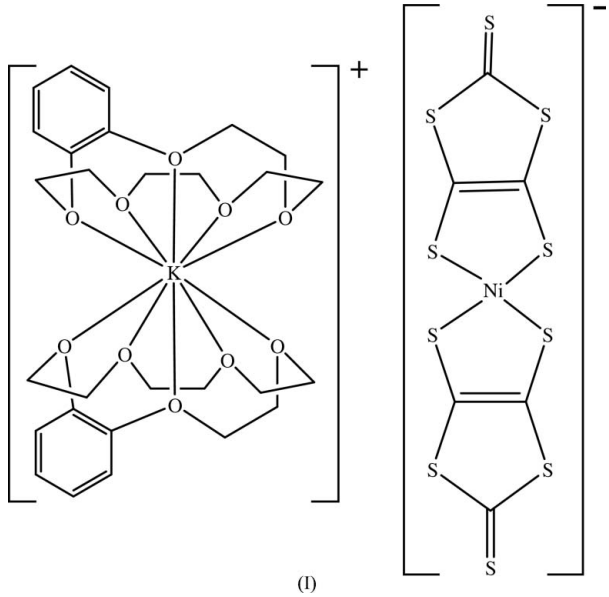
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.047
wR factor = 0.057
Data-to-parameter ratio = 15.5

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

The title compound, $[K(C_{14}H_{20}O_5)_2][Ni(C_3S_5)_2]$ or [K(benzo-15-crown-5)$_2$][Ni(dmit)$_2$], consists of K^I complex cations with benzo-15-crown-5 ligands and Ni^{III} complex anions with 4,5-dimercapto-1,3-dithiole-2-thione (dmit) ligands. The Ni^{III} atom is coordinated by four S atoms from two dmit ligands in a distorted square-planar geometry, while the K^+ cation is coordinated by ten O atoms from two crown ether ligands.

Comment

As part of an investigation of organic–inorganic hybrid crystalline solids containing crown-ether-coordinated cations as building blocks (Dou *et al.*, 2004; Dong *et al.*, 2005), we prepared the title compound, (I), and present its crystal structure here.



(I)

The crystal structure of (I) consists of K^+-crown complex cations and Ni^{III} complex anions. The molecular structure is shown in Fig. 1. Within the complex cation, K^+ is coordinated by ten O atoms from two benzo-15-crown-5 molecules. The K—O distances range from 2.788 (4) to 3.014 (3) Å. This is consistent with the values found in [K(18-crown-6)][Ni(dmit)$_2$] (Wang *et al.*, 2002).

The charge balance suggests that the Ni atom occurs as Ni^{III} and not Ni^{II} in the complex anion, which agrees with the situations in $(Bu_4N)[Ni(dmit)_2]$ (Lindqvist *et al.*, 1982) and [K(18-crown-6)][Ni(dmit)$_2$] (Wang *et al.*, 2002). Within the complex anion, the Ni^{III} atom is coordinated by four S atoms from two dmit ligands with a tetrahedrally distorted square-planar geometry, the dihedral angle between two dmit mean

planes being 15.18 (7)°. The Ni—S bond distances (Table 1) are consistent with those found in $(Bu_4N)[Ni(dmit)_2]$ (Lindqvist *et al.*, 1982).

The packing is shown in Fig. 2. The S2···S10($\frac{3}{2}-x$, $y+\frac{1}{2}$, $\frac{1}{2}-z$) separation of 3.444 (2) Å and the S4···S5($\frac{3}{2}-x$, $y+\frac{1}{2}$, $\frac{1}{2}-z$) separation of 3.573 (2) Å are the shortest contacts between complex anions.

Experimental

A 1,2-dichloroethane (10 ml) solution of benzo-15-crown-5 (1 mmol) was mixed with an aqueous solution (5 ml) of $NiCl_2·6H_2O$ (0.5 mmol) and an ethanol solution (5 ml) of K_2dmit (0.5 mmol). The mixture was stirred for 2 h at room temperature and then filtered. The precipitate was separated and dissolved in a mixture of ethanol and diethyl ether (1:1 (*v*/*v*). Colourless single crystals of (I) were obtained by slow evaporation of the solution (m.p. 523 K). Analysis calculated for $C_{34}H_{40}KNiO_{10}S_{10}$: C 39.76, H 3.93, S 31.22%; found: C 39.71, H 3.96, S 31.19%.

Crystal data

$[K(C_{14}H_{20}O_5)_2][Ni(C_3S_5)_2]$	D_x = 1.542 Mg m^{-3}
M_r = 1027.07	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2431 reflections
a = 18.270 (6) Å	θ = 2.5–20.1°
b = 11.800 (4) Å	μ = 1.06 mm^{-1}
c = 21.927 (8) Å	T = 293 (2) K
β = 110.648 (8)°	Block, colourless
V = 4424 (3) Å^3	0.34 × 0.23 × 0.16 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	7813 independent reflections
	3106 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.074
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	θ_{max} = 25.0°
	$h = -21 \rightarrow 21$
T_{min} = 0.715, T_{max} = 0.849	$k = -14 \rightarrow 13$
22348 measured reflections	$l = -26 \rightarrow 16$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$w = 1/[\sigma^2(F_o^2)]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.057	$(\Delta/\sigma)_{max}$ = 0.001
S = 0.85	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
7813 reflections	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
505 parameters	

Table 1
Selected bond lengths (Å).

Ni1—S4	2.1518 (14)	K1—O4	2.892 (3)
Ni1—S5	2.1586 (15)	K1—O5	2.835 (4)
Ni1—S9	2.1594 (15)	K1—O6	2.931 (3)
Ni1—S10	2.1484 (14)	K1—O7	2.965 (4)
K1—O1	2.790 (3)	K1—O8	2.905 (4)
K1—O2	2.807 (3)	K1—O9	2.788 (4)
K1—O3	3.014 (3)	K1—O10	2.822 (3)

All H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H = 0.93 (aromatic) or 0.97 Å (methylene). The U_{iso}(H) values were set at 1.2U_{eq}(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

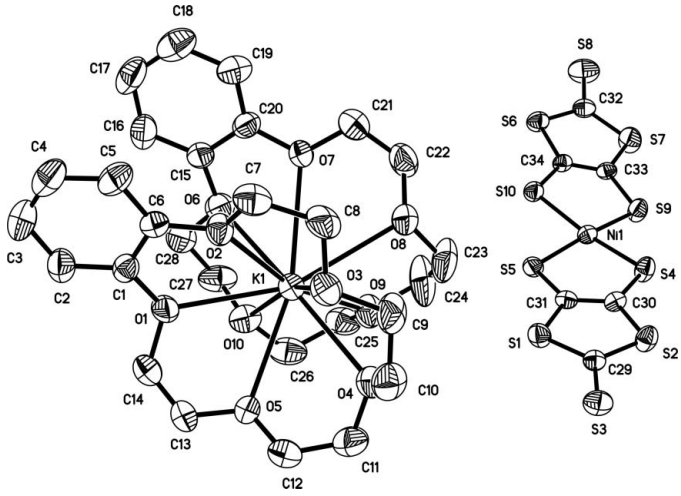


Figure 1
The structure of (I), shown with 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

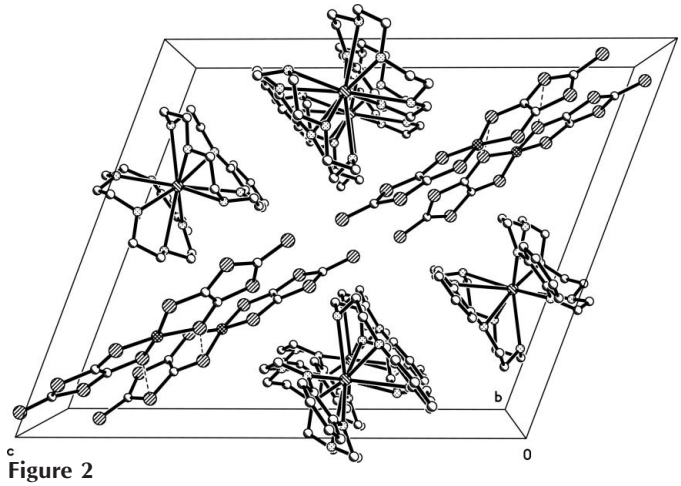


Figure 2
A packing diagram for (I), showing the S···S short contacts (dashed lines). H atoms have been omitted for clarity.

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