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Bis{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)zinc(II)

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The title complex, $[K(C_{14}H_{20}O_5)]_2[Zn(C_3S_5)_2]$ or $[K(B\text{-}15\text{-}C\text{-}5)_2]_2[Zn(dmit)_2]$ (B-15-C-5 = benzo15-crown-5 and dmit = 4,5-dimercapto-1,3-dithiole-2-thione), contains two $\{[K(B\text{-}15\text{-}C\text{-}5)_2]_2\}^+$ complex cations and one $[Zn(dmit)_2]^{2-}$ complex anion in the asymmetric unit. The complex has a three-dimensional network structure assembled by intermolecular S···S and weak C—H···S interactions.

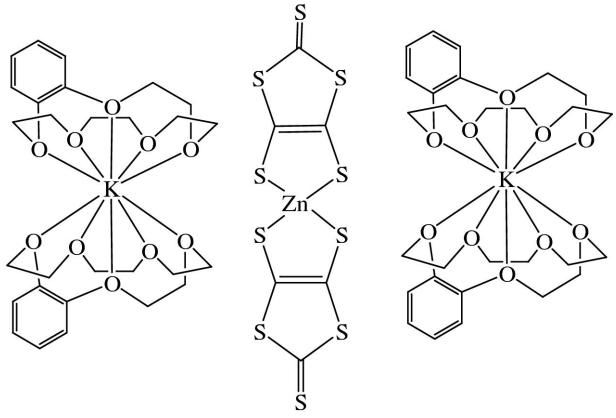
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.009 Å
R factor = 0.051
wR factor = 0.067
Data-to-parameter ratio = 15.2

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

Comment

The crystal structure of $[K(B\text{-}15\text{-}C\text{-}5)_2]_2[Zn(dmit)_2]$ (B-15-C-5 = benzo-15-crown-5 and dmit = 4,5-dimercapto-1,3-dithiole-2-thione), (I), contains two $\{[K(B\text{-}15\text{-}C\text{-}5)_2]_2\}^+$ complex cations, and one $[Zn(dmit)_2]^{2-}$ complex anion in the asymmetric unit (Fig. 1). The Zn atom is coordinated by four S atoms from two dmit ligands, the S4—Zn1—S10, S10—Zn1—S5, S5—Zn1—S9, S4—Zn1—S5 and S10—Zn1—S9 angles show the Zn atom exists in a tetrahedral geometry, with a dihedral angle of 94.3 (7)° between S4—Zn1—S5 and S9—Zn1—S10. The dihedral angle between S4—C59—C58—S5 and S1—C59—C58—S2—C57 is 1.98 (18)°, and that between S9—C61—C62—S10 and S7—C61—C62—S6—C60 is 1.0 (2)°. The Zn—S bond distances lie in the range 2.3159 (14)–2.3414 (13) Å (Table 1).



(I)

In the two crystallographically distinct $[K(B\text{-}15\text{-}C\text{-}5)_2]^+$ cations, each K atom is coordinated by ten O atoms from two benzo-15-crown-5 molecules and the environment of each K atom is the same. The K—O distances range from 2.783 (3) to 3.117 (3) Å, with a mean value of 2.903 Å, which is consistent with the corresponding value in the compound $(Bu_4N)\{[K(18\text{-}C\text{-}6)][Cd_4(dmit)_2(dmt)_3]\}$ (dmt = 4,5-dimercapto-1,2-dithiole-3-thione; Wang *et al.*, 2003). Furthermore, from Fig. 2 it can be

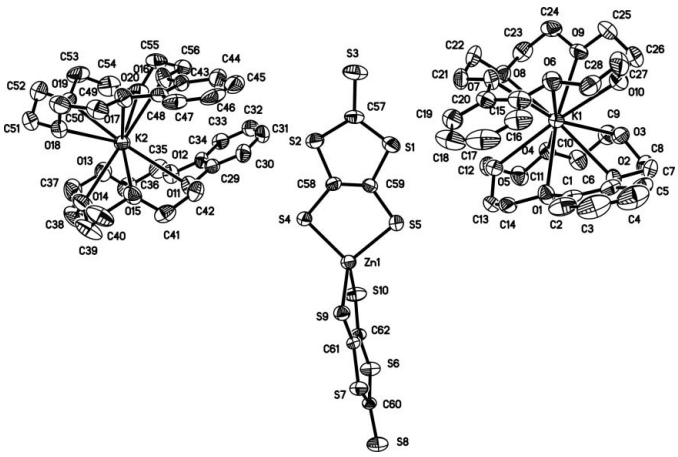


Figure 1
The structure of the title complex, showing 30% probability displacement ellipsoids. H atoms have been omitted.

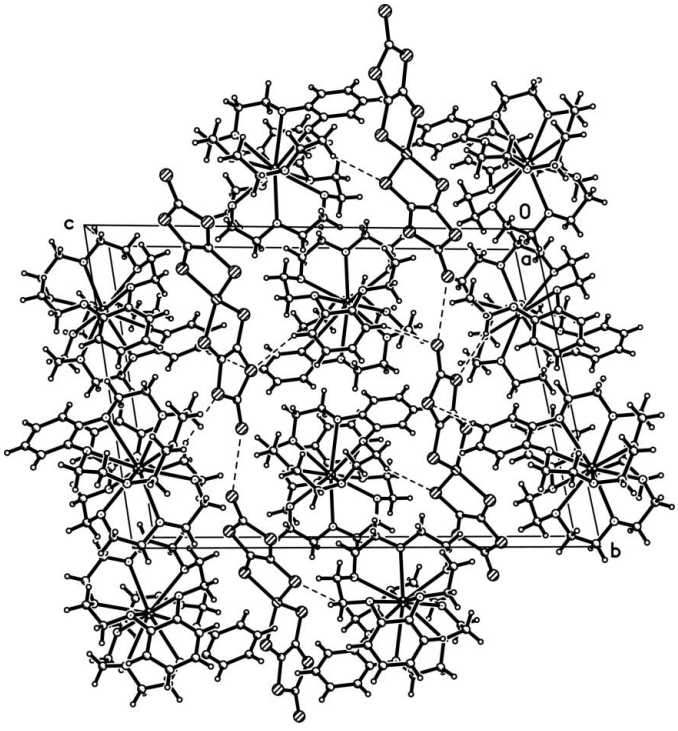


Figure 2
Crystal packing of the title complex, showing the S···S weak interactions and the C—H···S bonds (dashed lines).

seen that the complex has a three-dimensional network structure assembled by intermolecular head-to-tail S3···S8($x-1, y-1, z$) weak interactions between adjacent dmit groups, and intramolecular or intermolecular C—H···S hydrogen bonds between the dmit ligand and the benzo ring of B-15-C-5-crown-5. Bonds lengths and angles agree with accepted values; full details are given in the archived CIF.

Experimental

To a solution of B-15-C-5-crown-5 (1.00 mmol) in 1,2-dichloroethane (10 ml) was added an aqueous (5 ml) mixture of $ZnCl_2$ (0.25 mmol) and a solution of K_2dmit (0.5 mmol) in ethanol (5 ml). The reaction mixture was stirred for 2 h at room temperature and then filtered. The precipitate was dissolved in a mixture of ethanol and diethyl ether (4:1, *v*/*v*). Colorless single crystals were obtained by slowly evaporating the solution (m.p. 576 K). Analysis calculated for $C_{62}H_{80}K_2O_{20}S_{10}Zn$: C 46.27, H 5.01, S 19.92%; found: C 46.13, H 4.91, S 19.87%.

Crystal data

$[K(C_{14}H_{20}O_5)]_2[Zn(C_3S_5)_2]$	$Z = 2$
$M_r = 1609.43$	$D_x = 1.439$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 10.9614$ (12) Å	Cell parameters from 2462 reflections
$b = 16.1352$ (17) Å	$\theta = 2.2$–$18.6°$
$c = 22.026$ (2) Å	$\mu = 0.79$ mm^{-1}
$\alpha = 98.832$ (2)°	$T = 295$ (2) K
$\beta = 94.633$ (3)°	Block, colorless
$\gamma = 103.500$ (2)°	0.43 × 0.37 × 0.31 mm
$V = 3715.4$ (7) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	12981 independent reflections
φ and ω scans	4843 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.051$
$T_{min} = 0.727$, $T_{max} = 0.792$	$\theta_{max} = 25.0°$
20681 measured reflections	$h = -13 \rightarrow 10$
	$k = -19 \rightarrow 19$
	$l = -20 \rightarrow 26$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.051$	$w = 1/[\sigma^2(F_o^2)]$
$wR(F^2) = 0.067$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.81$	$\Delta\rho_{max} = 0.37$ e Å^{-3}
12981 reflections	$\Delta\rho_{min} = -0.39$ e Å^{-3}
856 parameters	

Table 1
Selected geometric parameters (Å, °).

Zn1—S4	2.3159 (14)	K2—O17	2.967 (3)
Zn1—S10	2.3178 (14)	K2—O16	2.994 (3)
Zn1—S5	2.3276 (13)	K2—O12	3.011 (3)
Zn1—S9	2.3414 (13)	K2—O11	3.069 (3)
K1—O9	2.783 (3)	S1—C57	1.697 (5)
K1—O4	2.790 (3)	S1—C59	1.738 (4)
K1—O10	2.829 (3)	S2—C57	1.727 (4)
K1—O5	2.837 (3)	S2—C58	1.749 (4)
K1—O3	2.876 (3)	S3—C57	1.645 (4)
K1—O1	2.911 (3)	S3—S8^{i}	3.5034 (19)
K1—O8	2.917 (3)	S4—C58	1.731 (4)
K1—O2	2.959 (3)	S5—C59	1.716 (5)
K1—O7	3.034 (3)	S6—C60	1.741 (4)
K1—O6	3.117 (3)	S6—C62	1.747 (4)
K2—O14	2.762 (4)	S7—C60	1.696 (4)
K2—O15	2.794 (3)	S7—C61	1.735 (4)
K2—O19	2.795 (4)	S8—C60	1.645 (4)
K2—O20	2.811 (3)	S9—C61	1.732 (4)
K2—O18	2.884 (4)	S10—C62	1.725 (4)
K2—O13	2.927 (4)		
S4—Zn1—S10	120.26 (5)	S4—Zn1—S9	116.65 (5)
S4—Zn1—S5	95.64 (5)	S10—Zn1—S9	94.80 (5)
S10—Zn1—S5	112.18 (5)	S5—Zn1—S9	119.07 (5)

Symmetry code: (i) $x-1, y-1, z$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C30—H30···S2	0.93	2.93	3.685 (5)	140
C21—H21B···S1	0.97	2.93	3.706 (5)	138
C13—H13B···S5	0.97	2.99	3.852 (5)	149
C33—H33···S3^{ii}	0.93	2.90	3.756 (6)	154
C3—H3···S2^{iii}	0.93	2.93	3.849 (9)	171
C35—H35A···S8^{iv}	0.97	2.95	3.665 (5)	131
C49—H49B···S10^{v}	0.97	2.92	3.791 (6)	150
C24—H24A···S9^{vi}	0.97	2.96	3.906 (5)	165
C7—H7A···S9^{vii}	0.97	2.97	3.538 (5)	119

Symmetry codes: (ii) $-x+1, -y+1, -z+1$; (iii) $-x+1, -y+1, -z$; (iv) $-x+2, -y+2, -z+1$; (v) $x-1, y, z$; (vi) $x, y-1, z$; (vii) $-x+2, -y+1, -z$.

Similarity restraints were applied to displacement parameters of some ligand atoms to avoid extreme values. All H atoms were positioned geometrically and treated as riding on their parent atoms, with aromatic C—H distances of 0.93 Å and methylene C—H distances of 0.97 Å. 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*.

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