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Bis(diethyldithiocarbamato)(4,7-dimethyl-1,10-phenanthroline)cadmium(II) acetonitrile solvate

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Key indicators

Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.005 Å
R factor = 0.045
wR factor = 0.105
Data-to-parameter ratio = 27.7

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

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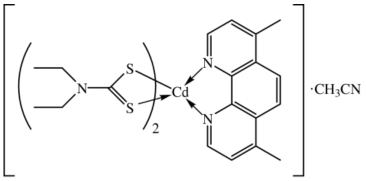
An N_2S_4 donor set defines the coordination environment about the cadmium centre in the monomeric title complex, [Cd(dedtc)$_2$(4,7-Me$_2$phen)]·C_2H_3N or [Cd{$S_2CN(CH_2CH_3)_2$}$_2$($C_{14}H_{12}N_2$)]·C_2H_3N. The disposition of the donor atoms is such as to define a polyhedron based on a trigonal prism.

Comment

The structure of the title compound, Cd(dedtc)$_2$(4,7-Me$_2$phen), (I), has been determined as part of an ongoing investigation of the structural characteristics of zinc, cadmium and mercury 1,1-dithiolates and their adducts. It is well understood that the diverse coordination patterns and molecular aggregation observed in the parent compounds (Cox & Tiekink, 1997) is moderated upon addition of Lewis base so that, usually, mononuclear or dinuclear species result. This reduced aggregation offers a considerable advantage in terms of providing more suitable precursors for chemical vapour deposition studies.



(I)

The molecular structure of (I) (Fig. 1 and Table 1) features a six-coordinate cadmium centre defined by four S atoms, derived from two dithiocarbamate ligands, and two N atoms from the diimine. The dithiocarbamate ligands adopt a chelating mode of coordination, forming essentially equivalent Cd—S bonds that lie in the relatively narrow range 2.6116 (7)–2.6976 (7) Å [average 2.6617 (4) Å]. Similarly, the Cd—N bonds [average 2.408 (1) Å] are equal within experimental error. The coordination geometry is irregular, being intermediate between octahedral and trigonal prismatic, but tending towards the latter. In the trigonal prismatic description, atoms S1, S3 and N4 define one triangular face and atoms S2, S4 and N3 the other. The dihedral angle between the two faces is 5.66 (8)°. Atoms S1—S4 lie, respectively, 0.3152 (4), −0.3299 (4), −0.3129 (4) and 0.3276 (4) Å out of their least-squares plane and the dihedral angle formed between this and the plane through atoms Cd, N3 and N4 is 88.83 (5)°.

The structures of both the 2,2′-bipyridine and 1,10-phenanthroline adducts of Cd(dedtc)$_2$ are known (Glinskaya *et al.*, 1992; Airoldi *et al.*, 1990). The coordination geometries in these structures have been described as distorted octahedral. In the 2,2′-bipyridine structure, which has crystallographic twofold symmetry, the average Cd—S and Cd—N

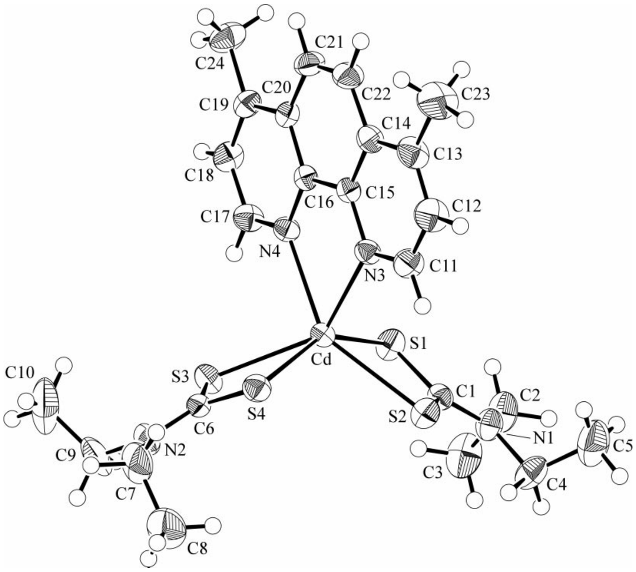


Figure 1

The molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level (Johnson, 1976).

bond distances are 2.6670 (7) and 2.430 (3) Å, respectively. These are in accord with the values in the 1,10-phenanthroline adduct, for which two molecules comprise the asymmetric unit, are 2.671 (1) and 2.410 (5) Å, respectively. This are in accord with the values determined in the present study. By contrast to the mononuclear species formed with the diimine bases, a dinuclear structure with square pyramidal cadmium centres is found in the diphosphine adduct Cd(dedtc)$_2$(μ-Ph$_2$PC$_5$H$_4$FeC$_5$H$_4$PPh$_2$)Cd(dedtc)$_2$ (Dee & Tiekink, 2002*a*).

Experimental

Yellow crystals were obtained by slow evaporation of a CHCl$_3$/CH$_3$CN (1/1) solution of Cd(dedtc)$_2$(4,7-Me$_2$phen) prepared by refluxing equimolar amounts of Cd(dedtc)$_2$ (Dee & Tiekink, 2002*b*) and 4,7-dimethyl-1,10-phenanthroline (Aldrich) in chloroform solution. The melting point of the desolvated material was 551–555 K. IR: (KBr) ν(C—S) 1030, 990 and ν(C—N) 1417 cm^{-1}.

Crystal data

[Cd(C$_5$H$_{10}$NS$_2$)$_2$(C$_{14}$H$_{12}$N$_2$)]·C$_2$H$_3$N
M_r = 658.23
Monoclinic, $P2_1/c$
a = 10.9451 (7) Å
b = 10.0886 (6) Å
c = 28.4366 (17) Å
β = 94.198 (1)°
V = 3131.6 (3) Å^3
Z = 4

D_x = 1.396 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7229 reflections
θ = 2.4–30.0°
μ = 0.99 mm^{-1}
T = 223 (2) K
Needle, pale yellow
0.55 × 0.08 × 0.08 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min}$ = 0.739, $T_{\max}$ = 0.924
25219 measured reflections

9058 independent reflections
7059 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.036
$\theta_{\max}$ = 30.0°
$h = -15 \rightarrow 15$
$k = -13 \rightarrow 14$
$l = -29 \rightarrow 39$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.105
S = 1.06
9058 reflections
327 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 + 0.3208P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 1.27 e Å^{-3}
$\Delta\rho_{\min}$ = −0.39 e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

Cd—S1	2.6116 (7)	S1—C1	1.719 (3)
Cd—S2	2.6976 (7)	S2—C1	1.720 (3)
Cd—S3	2.6630 (7)	S4—C6	1.720 (3)
Cd—S4	2.6747 (7)	S3—C6	1.720 (3)
Cd—N3	2.405 (2)	N1—C1	1.335 (3)
Cd—N4	2.410 (2)	N2—C6	1.324 (4)
S1—Cd—S2	67.80 (2)	S4—Cd—N4	114.71 (5)
S1—Cd—S3	102.21 (2)	N3—Cd—N4	67.84 (7)
S1—Cd—S4	151.09 (2)	Cd—S1—C1	88.00 (9)
S1—Cd—N3	109.38 (5)	Cd—S2—C1	85.22 (9)
S1—Cd—N4	90.79 (5)	Cd—S3—C6	86.83 (9)
S2—Cd—S3	122.05 (2)	Cd—S4—C6	86.44 (9)
S2—Cd—S4	94.11 (2)	S1—C1—S2	118.95 (15)
S2—Cd—N3	93.52 (5)	S1—C1—N1	119.8 (2)
S2—Cd—N4	145.75 (6)	S2—C1—N1	121.2 (2)
S3—Cd—S4	67.49 (2)	S3—C6—S4	119.08 (15)
S3—Cd—N3	139.54 (5)	S3—C6—N2	120.4 (2)
S3—Cd—N4	87.48 (5)	S4—C6—N2	120.5 (2)
S4—Cd—N3	93.50 (5)		

H atoms were placed in calculated positions and included with overall displacement parameters: $U_{\rm iso} = xU_{\rm eq}$(C), with x = 1 for aromatic, x = 1.25 for methylene and x = 1.5 for methyl H. The largest residual electron-density peaks are in the vicinity of the Cd atom.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *PATTY* in *DIRDIF*92 (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976).

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