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Crystallographic report

Bis(*O*-methoxyethyldithiocarbonato)(4,7-dimethyl-1,10-phenanthroline)cadmium(II)

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The structure of mononuclear $Cd(S_2COCH_2CH_2OCH_3)_2(4,7\text{-}Me_2phen)$ shows an N_2S_4 donor set about cadmium that defines a coordination geometry intermediate between octahedral and trigonal prismatic. Copyright © 2003 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; cadmium; dithiocarbonate; organometallic; imine

COMMENT

Two distinct structural motifs are known for the cadmium xanthates.[1] In one motif, adopted by R = Et,[2] R = i-Pr[3–5] and R = *n*-Bu,[6] two-dimensional sheets, comprising interconnected 16-membered rings, are formed as a result of all xanthates being bridging. This results in four-coordinate, distorted tetrahedral cadmium centres. In the other motif, adopted by R = Me[7] and R = $CH_2CH_2OCH_3$,[8] square-planar cadmium geometries are found as a result of the presence of chelating xanthate ligands; mononuclear units aggregate into loosely associated chains facilitated by supramolecular Cd···S interactions. Despite the different structures found for the parent compounds, adduct formation leads to similar mononuclear species as seen in the title compound and related species.[9,10] The cadmium atom in $Cd(S_2COCH_2CH_2OCH_3)_2(4,7\text{-}Me_2phen)$ exists within an N_2S_4 donor set with a coordination geometry intermediate between octahedral and trigonal prismatic (Fig. 1).

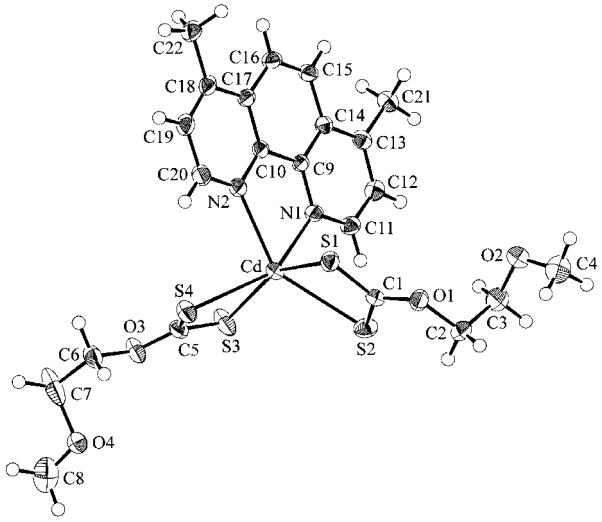

Figure 1. Molecular structure of $Cd(S_2COCH_2CH_2OCH_3)_2(4,7\text{-}Me_2phen)$. Key geometric parameters: Cd–S1 2.6028(6), Cd–S2 2.7960(7), Cd–S3 2.6401(7), Cd–S4 2.7018(6), Cd–N1 2.3394(18), Cd–N2 2.4064(18) Å; S1–Cd–S2 66.880(18), S1–Cd–S3 154.79(2), S1–Cd–S4 102.750(19), S1–Cd–N1 102.53(4), S1–Cd–N2 92.59(4), S2–Cd–S3 97.41(2), S2–Cd–S4 121.06(2), S2–Cd–N1 88.02(5), S2–Cd–N2 145.96(4), S3–Cd–S4 67.517(19), S3–Cd–N1 96.20(4), S3–Cd–N2 109.84(5), S4–Cd–N1 147.29(4), S4–Cd–N2 88.89(4), N1–Cd–N2 69.55(6)°.

EXPERIMENTAL

To a stirred chloroform–acetonitrile (50 ml) solution of $Cd(S_2COCH_2CH_2OCH_3)_2$ (0.2 g)[8] was added a stoichiometric amount of 4,7-Me_2phen (Aldrich). The mixture was refluxed for 2 h, filtered and the solvent removed *in vacuo*. The precipitate was recrystallized by the slow evaporation of a chloroform solution of the compound to yield colourless crystals suitable for X-ray analysis; m.p. 137–138 °C.

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IR (KBr): ν(C–S) 1058 and ν(C–O) 1180 cm^{-1}. Intensity data were collected at 183 K on a Bruker AXS SMART CCD diffractometer for a block 0.31 × 0.39 × 0.52 mm^3. $C_{22}H_{26}CdN_2O_4S_4$, M = 623.1, monoclinic, $P2_1/n$, a = 8.9770(8), b = 23.381(2), c = 11.7006(10) Å,

$\beta = 91.519(2)°$, $V = 2455.0(4)$ Å^3, $Z = 4$, 7157 unique data (θ_{max} 30.1°), $R = 0.043$ (all data), $wR = 0.092$ (all data), $\rho_{max} = 1.41$ e$^-$ Å^{-3} (near cadmium). Two conformations were detected for the O4 atom, in the ratio 0.55 : 0.45 (from refinement). Programs used: teXsan, DIRDIF, SHELXL, and ORTEP. CCDC deposition number: 199840.

Acknowledgements

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