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Crystallographic report

Bis{μ-[*O*-cyclopentyl(4-methoxyphenyl)dithiophosphonato]1κ:*S*,2κ:*S*-[*O*-cyclopentyl(4-methoxyphenyl)dithiophosphonato]-1κ^2*S*,*S*′}dicadmium(II)

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The centrosymmetric title compound, $[Cd_2\{CH_3OC_6H_4P(OC_5H_9)S_2\}_4]$, features an eight-membered $[-Cd-S-P-S-]_2$ ring owing to the presence of bridging dithiolate ligands. Tetrahedral coordination geometries for cadmium are completed by chelating ligands. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; dithiophosphonate; cadmium

COMMENT

The centrosymmetric $[Cd_2\{CH_3OC_6H_4P(OC_5H_9)S_2\}_4]$ structure shows the presence of two chelating and two bidentate bridging ligands, leading to the formation of an eight-membered ring; Fig. 1. This arrangement leads to a tetrahedral-coordinated cadmium atom. The structural form reported here is found in the structure of the isopropyldithiophosphate analogue[1] and, indeed, in most binary cadmium 1,1-dithiolates.[2]

EXPERIMENTAL

The complex was prepared in 89% yield by the reaction of $Cd(CH_3COO)_2 \cdot 2H_2O$ and $CH_3OC_6H_4P(OC_5H_9)(S)(SNH_4)$[3] in dry ethanol. Colourless crystals were obtained from a mixture of chloroform and methanol (1:1); m.p. 200–201 °C. Anal. (calc.) for $C_{24}H_{32}CdO_4P_2S_4$: C, 41.95 (42.47); H, 4.69 (3.95); S, 18.61 (17.69). IR data (cm^{-1}): 553 (PS_{sym}) and 649 (PS_{asym}). ^{1}H NMR (DMSO-d_6) δ (ppm): 8.88 (dd, 8H, $^3J_{PH} = 13.84$ Hz, $J_{PH} = 8.76$ Hz), 7.00 (dd, 8H, $^4J_{PH} = 2.84$ Hz, $J_{HH} = 8.85$ Hz), 5.35 (m, 4H), 3.80 (s, 12H, CH_3O–), 1.68 (m, 32H). ^{13}C NMR (DMSO-d_6) δ (ppm): 133.17 (C-1, $^1J_{PC} = 121.4$ Hz), 132.33 (C-2, $^2J_{PC} = 13.6$ Hz), 114.03 (C-3, $^3J_{PC} = 15.2$ Hz), 161.9 (C-4, $^4J_{PC} = 3.1$ Hz), 78.6 (C-5, $^2J_{PC} = 6.9$ Hz; O–CH–; Cp), 34.6 (C-6, $^3J_{PC} = 4.4$ Hz; Cp), 56.19 (CH_3O–), 23.82 (C-8; Cp). ^{31}P NMR (DMSO-d_6) δ (ppm): 103.4. Intensity data were collected at 293(2) K on an Enraf Nonius CAD-4 diffractometer for a crystal $0.20 \times 0.25 \times 0.30$ mm^3. $C_{24}H_{32}CdO_4P_2S_4$, $M = 687.08$, monoclinic, space group $P2_1/n$, $a = 11.181(4)$, $b = 24.804(3)$, $c = 11.501(2)$ Å, $\beta = 114.60(2)°$, $V = 2900.1(12)$ Å^3, $Z = 2$ (dimers), $\theta_{max} = 28.8°$, $R = 0.041$ (3612 data with $I \geq 2\sigma(I)$), $R = 0.111$ (all data; 6979). Programs used: SHELX 97, ORTEP. CCDC deposition number: 224102.

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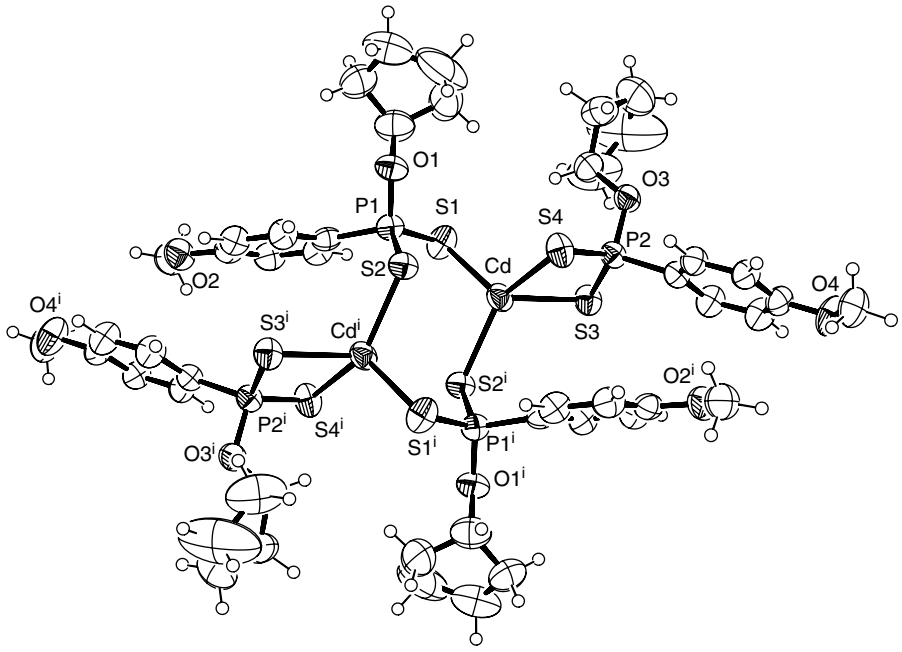

Figure 1. ORTEP plot of $[Cd_2\{CH_3OC_6H_4P(OC_5H_9)S_2\}_4]$. Key geometric parameters: Cd–S1 2.586(1), Cd–S2^i 2.521(1), Cd–S3 2.662(2), Cd–S4 2.521(1), P1–S1 1.998(2), P1–S2 2.021 Å; P2–S3 1.997(2), P2–S4 2.017(2), S1–Cd–S3 103.77(4), S2^i–Cd–S3 104.84(4), S1–P1–S2 113.57(7), S3–P2–S4 111.21°. Symmetry operation i: *–x*, *–y*, *–z*.