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Crystallographic report

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

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The centrosymmetric $[Zn_2\{CH_3OC_6H_4P(OC_5H_9)S_2\}_4]$, features an eight-membered $Zn_2S_4P_2$ ring as a result of two bidentate bridging thiolate ligands; the remaining ligands are chelating. Copyright © 2005 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; dithiophosphonate; zinc(II)

COMMENT

Centrosymmetric $[Zn_2\{CH_3OC_6H_4P(OC_5H_9)S_2\}_4]$ with two bidentate bridging, leading to the formation of a $Zn_2S_4P_2$ ring, and two chelating ligands, conforms to the common structural motif adopted by Zn(1,1-dithiolate)$_2$ complexes (Fig. 1).[1]

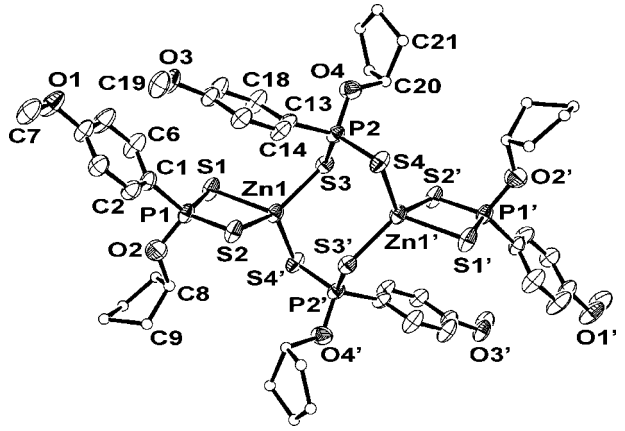

Figure 1. A view of $[Zn_2\{CH_3OC_6H_4P(OC_5H_9)S_2\}_4]$; hydrogen atoms omitted for clarity. Key geometric parameters: Zn–S1 2.4671(15), Zn–S2 2.3358(14), Zn–S3 2.3211(16), Zn–S4 2.3633(15), Zn–S4′ 2.3633(15) Å; S1–Zn–S3 108.19(16), S3–Zn–S4 103.70(6), S2–Zn–S3 131.20(6), S2–Zn–S4′ 118.90(6), S1–Zn–S2 85.52(5), S1–Zn–S4′ 102.87(6)°. Symmetry operation on primed atoms: $1-x$, $1-y$, $1-z$.

EXPERIMENTAL

The complex was prepared in 91% yield by the reaction of $CH_3OC_6H_4P(OC_5H_9)(S)(SNH_4)$[2] and $ZnSO_4{\cdot}7H_2O$ in water. Colourless crystals were obtained from a mixture of chloroform and isopropyl alcohol (3 : 1); m.p.: 169 °C. Anal. (calc.) for $C_{48}H_{64}O_8P_4S_8Zn_2$: C, 45.38 (45.03); H, 4.74 (5.04); S, 19.67 (20.04)%. IR data (cm^{-1}): 541 (PS_{sym}) and 653 (PS_{asym}). ^{1}H NMR (DMSO-d_6) δ (ppm): 7.87 (dd, 8H, $^3J_{PH} = 13.71$ Hz, $J_{HH} = 8.67$ Hz), 6.96 (dd, 8H, $^4J_{PH} = 2.30$ Hz, $J_{HH} = 8.80$ Hz), 5.02 (m, 4H), 3.78 (s, 12H, CH_3O–), 1.58 (m, 32H). ^{13}C NMR (DMSO-d_6) δ (ppm): 134.25 (C-1, $^1J_{PC} =$ 119 Hz), 132.38 (C-2, $^2J_{PC} = 13.60$ Hz), 113.80 (C-3, $^3J_{PC} = 15.10$ Hz), 161.70 (C-4, $^4J_{PC} = 3.04$ Hz), 78.40 (C-5, $^2J_{PC} = 7.20$ Hz, O–CH–, Cp), 34.50 (C-6, $^3J_{PC} = 4.20$ Hz, Cp), 56.17 (CH_3O–), 23.78 (C-8, Cp). ^{31}P NMR (DMSO) δ (ppm): 99.69. Intensity data were collected at 293(2) K on Siemens Smart CCD diffractometer for a crystal $0.15 \times 0.15 \times 0.30$ mm^3. $C_{48}H_{64}O_8P_4S_8Zn_2$, $M = 1280.20$, triclinic, $P\bar{1}$, $a = 11.330(2)$, $b = 12.385(2)$, $c = 12.511(2)$ Å, $\beta = 73.984(3)°$, $V =$ 1.5148(5) nm^3, $Z = 1$, $R = 0.064$ (6057 data with $I > 2\sigma(I)$), $R = 0.099$ (all 8585 data, $\theta_{max} = 26.4°$). Programs used: SHELX97, ORTEP. CCDC deposition number: 215 075. The cyclopenteyl group (C8–C12)

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