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Crystallographic report

(4,7-Diphenyl-1,10-phenanthroline) bis(pyrrolinedithiocarbamato)zinc(II) chloroform solvate

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The mononuclear structure of $Zn(S_2CN(CH_2)_4)_2(4,7\text{-}Ph_2\text{-}1,10\text{-phenanthroline})$ shows the zinc atom in each of the two independent molecules comprising the asymmetric unit to exist in a distorted octahedral geometry defined by an N_2S_4 donor set. Copyright © 2003 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; zinc; dithiocarbamate; diimine adduct

COMMENT

In connection with ongoing studies into the structural chemistry of diimine adducts of the zinc-triad 1,1-dithiolates,[1–3] the title compound, $Zn(S_2CN(CH_2)_4)_2(4,7\text{-}Ph_2\text{-}1,10\text{-phen})$, was investigated. Two crystallographically independent molecules comprise the asymmetric unit; they are chemically similar but differ in their geometric parameters, most notably in the Zn—S bond distances (Fig. 1). Thus, the Zn—S distances range from 2.4524(8) to 2.5788(8) Å for the Zn1 atom and from 2.4337(8) to 2.5678(8) Å for Zn2. The octahedral geometry found for each of the zinc atoms is defined by an N_2S_4 donor set.

EXPERIMENTAL

Bright-yellow crystals were isolated from an acetonitrile/chloroform (1:1) solution containing equimolar amounts of $Zn(S_2CN(CH_2)_4)_2$ and 4,7-Ph_2-phen (Aldrich); m.p. 205–208 °C. IR (KBr, Ca^{-1}): ν(C—S) 1005, 945 and ν(C—N) 1428. Intensity data were collected at 183 K on a Bruker AXS SMART CCD diffractometer for a yellow block $0.18 \times 0.23 \times 0.49$ mm^3. $C_{34}H_{32}N_4S_4Zn{\cdot}CHCl_3$, $M = 809.6$, triclinic, $P\bar{1}$, $a = 13.1703(6)$, $b = 17.6586(9)$, $c = 17.8809(9)$ Å, $\alpha = 63.013(1)$, $\beta = 86.826(1)$, $\gamma = 75.315(1)$°, $V = 3576.1(3)$ Å^3, $Z = 4$, 20 489 unique data (θ_{max} 30.0°), $R = 0.078$ (all data), $wR = 0.169$ (all data), $\rho_{max} = 1.34$ e$^-$ Å^{-3} (near solvent molecule). Disorder in the structure was noted and resolved for the C8 atom of the pyrrolidine ring. From refinement, the major component had a site occupancy of 0.58. Some

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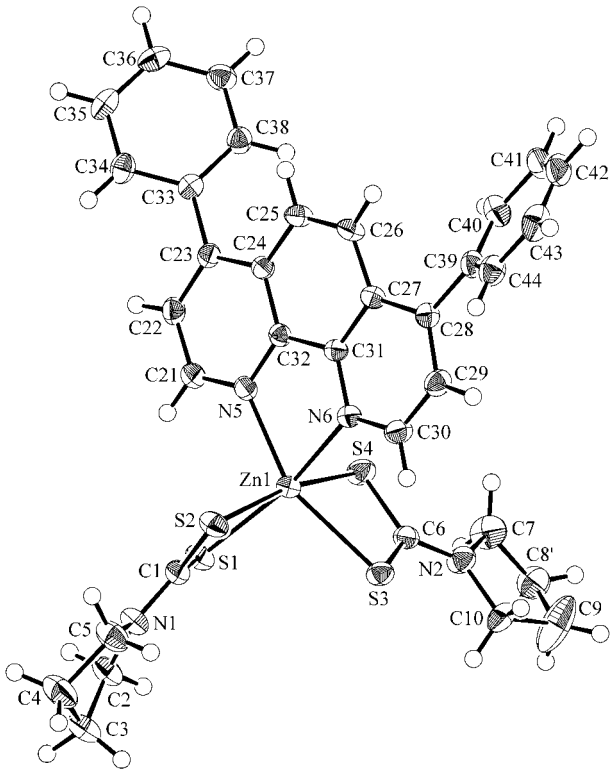

Figure 1. Molecular structure of molecule *a* in $Zn(S_2CN(CH_2)_4)_2(4,7\text{-}Ph_2\text{-}1,10\text{-phenanthroline})$; the chloroform molecule of solvation is omitted. Key geometric parameters: Zn(1)—S(1) 2.4524(8), Zn(1)—S(2) 2.5051(8), Zn(1)—S(3) 2.4993(8), Zn(1)—S(4) 2.5788(8), Zn(1)—N(5) 2.167(2), Zn(1)—N(6) 2.195(2) Å; S(1)—Zn(1)—S(2) 73.22(3), S(3)—Zn(1)—S(4) 71.28(2), N(5)—Zn(1)—N(6) 75.00(9), S(2)—Zn(1)—S(4) 168.49(3), S(1)—Zn(1)—N(6) 166.79(7), S(3)—Zn(1)—N(5) 155.07(6)°.

evidence was also found for an alternate position of the C70 chloroform molecule by rotation about the C—H axis; attempts to resolve this disorder were unsuccessful. Programs used: teXsan, DIRDIF, SHELXL, and ORTEP. CCDC deposition number: 193904.

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