Yang Liguo*, Wang Xin, Niu Yongsheng, Song Haixiang, Tian Dayong and Gao Linna

Crystal structure of (1,2-dicarba-*closo*-dodecaborane-1,2-dithiolato $\kappa^2 S, S'$)-bis(1,10-phenanthroline $\kappa^2 N, N'$)zinc(II), $C_{26}H_{26}B_{10}Zn_4S_2$

Table 1: Data collection and handling.

Crystal:	White block
Size:	0.27 × 0.21 × 0.13 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.97 mm ⁻¹
Diffractometer, scan mode:	SMART detector, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	15258, 5384, 0.062
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2805
$N(\text{param})_{\text{refined}}$:	388
Programs:	Bruker [1], SHELX [2, 3]

Source of material

Solvents, which were employed, were dried with appropriate drying agents and distilled under dinitrogen prior to use. Dichloromethane was used without purification. The 1,2-dithiolato-1,2-dicarba-*closo*-dodecaborane was prepared according to the literature method [4].

$Zn(OAc)_2 \cdot 4H_2O$ (54.9 mg, 0.25 mmol) was added to a suspension of 1,2-(Hs)₂-1,2-C₂B₁₀H₁₀ (55.1 mg, 0.25 mmol) and 1,10-phenanthroline (49.6 mg, 0.25 mmol) in dichloromethane (10 mL). The mixture was stirred for 6 h at room temperature under the protection of dry nitrogen. Then a colourless solution formed. Crystals were obtained by slow evaporation of a dichloromethane-*n*-hexane solution. (30.4 mg, 22.9%). m.p. 288–289 K. FTIR (KBr) ν (cm⁻¹): 2981, 2961, 2921, 2864; 2611, 2601, 2583, 2553.

Experimental details

All H atoms were placed geometrically and treated as riding on their parent atoms, with B–H 1.10, C–H 0.96, with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(B)$, $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(C)$.

Comment

Transition metal complexes containing chelating 1,2-dicarba-*closo*-dodecaborane-1,2-dithiolato ligands have attracted interest to take advantage of the unique molecular structure of carborane in recent years [5–7]. Although there are some mononuclear complexes with carboranyl ligands [8–10] the zinc complex with the dithiolato ligands has not been reported.

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Abstract

$C_{26}H_{26}B_{10}Zn_4S_2$, monoclinic, $P2_1/n$, $a = 14.3766(13)$ Å, $b = 11.6360(11)$ Å, $c = 18.9460(15)$ Å, $\beta = 104.9630(10)^\circ$, $V = 3061.9(5)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0451$, $wR_{\text{ref}}(F^2) = 0.0999$, $T = 298(2)$ K [1–3].

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

*Corresponding author: Yang Liguo, College of Chemistry and Environmental Engineering, Anyang Institute of Technology, Anyang 455000, Henan, P.R. China, e-mail: lgyang@ayit.edu.cn

Wang Xin, Niu Yongsheng, Song Haixiang and Tian Dayong: College of Chemistry and Environmental Engineering, Anyang Institute of Technology, Anyang 455000, Henan, P.R. China

Gao Linna: College of Chemical and Environmental Engineering, Shandong University of Science and Technology, Qingdao 266590, P.R. China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Zn1	0.78564(3)	0.29522(4)	0.04003(3)	0.05495(17)
N1	0.8058(2)	0.1992(3)	−0.05845(19)	0.0543(8)
N2	0.8066(2)	0.1107(3)	0.0727(2)	0.0562(9)
N3	0.7309(3)	0.4452(3)	−0.03250(16)	0.0526(9)
N4	0.6271(2)	0.2699(3)	0.00060(17)	0.0551(9)
S1	0.76439(8)	0.36279(9)	0.15525(6)	0.0615(3)
S2	0.95305(8)	0.34981(9)	0.06878(6)	0.0595(3)
C1	0.8764(3)	0.4242(3)	0.1974(2)	0.0566(11)
C2	0.9713(3)	0.4174(3)	0.1541(2)	0.0550(11)
C3	0.8055(3)	0.0681(4)	0.1380(3)	0.0701(13)
H3	0.7865	0.1159	0.1711	0.084*
C4	0.8316(3)	−0.0449(4)	0.1587(3)	0.0791(14)
H4	0.8267	−0.0731	0.2036	0.095*
C5	0.8643(3)	−0.1129(4)	0.1120(3)	0.0843(15)
H5	0.8842	−0.1876	0.1255	0.101*
C6	0.8683(3)	−0.0712(4)	0.0436(3)	0.0667(12)
C7	0.8376(3)	0.0420(3)	0.0262(2)	0.0547(11)
C8	0.8372(3)	0.0880(4)	−0.0440(2)	0.0550(11)
C9	0.8696(3)	0.0202(4)	−0.0940(3)	0.0649(12)
C10	0.9022(3)	−0.0929(4)	−0.0741(3)	0.0849(16)
H10	0.9234	−0.1381	−0.1073	0.102*
C11	0.9030(4)	−0.1361(4)	−0.0086(3)	0.0862(16)
H11	0.9266	−0.2099	0.0034	0.103*
C12	0.8650(3)	0.0690(5)	−0.1625(3)	0.0782(14)
H12	0.8846	0.0266	−0.1977	0.094*
C13	0.8320(3)	0.1782(4)	−0.1775(3)	0.0762(14)
H13	0.8287	0.2110	−0.2229	0.091*
C14	0.8031(3)	0.2401(4)	−0.1240(3)	0.0669(12)
H14	0.7806	0.3147	−0.1350	0.080*
C15	0.7833(3)	0.5309(4)	−0.0483(2)	0.0615(12)
H15	0.8500	0.5276	−0.0311	0.074*
C16	0.7419(4)	0.6256(4)	−0.0899(2)	0.0723(14)
H16	0.7804	0.6846	−0.0996	0.087*
C17	0.6451(4)	0.6306(4)	−0.1159(2)	0.0771(15)
H17	0.6170	0.6935	−0.1435	0.092*
C18	0.5870(4)	0.5418(4)	−0.1016(2)	0.0614(12)
C19	0.6343(3)	0.4497(4)	−0.0588(2)	0.0551(11)
C20	0.5783(3)	0.3562(3)	−0.0417(2)	0.0498(10)
C21	0.4783(3)	0.3582(4)	−0.0681(2)	0.0605(12)
C22	0.4331(4)	0.4530(5)	−0.1114(3)	0.0796(14)
H22	0.3665	0.4544	−0.1291	0.095*
C23	0.4859(4)	0.5404(5)	−0.1270(2)	0.0801(14)
H23	0.4548	0.6012	−0.1553	0.096*
C24	0.4265(3)	0.2663(5)	−0.0499(2)	0.0726(13)
H24	0.3598	0.2646	−0.0663	0.087*
C25	0.4750(4)	0.1789(4)	−0.0077(2)	0.0762(14)
H25	0.4417	0.1166	0.0046	0.091*
C26	0.5747(3)	0.1846(4)	0.0164(2)	0.0652(12)
H26	0.6067	0.1250	0.0455	0.078*
B1	0.9233(4)	0.4192(5)	0.2894(3)	0.0784(18)
H1	0.8859	0.3799	0.3272	0.094*
B2	0.8852(4)	0.5497(5)	0.2449(3)	0.0708(16)
H2	0.8223	0.5962	0.2533	0.085*
B3	0.9124(4)	0.5450(4)	0.1590(3)	0.0547(13)
H3A	0.8660	0.5868	0.1104	0.066*

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
B4	0.9734(4)	0.3336(5)	0.2300(3)	0.0675(15)
H4A	0.9670	0.2394	0.2275	0.081*
B5	0.9946(4)	0.5448(6)	0.3119(3)	0.0891(19)
H5A	1.0036	0.5889	0.3646	0.107*
B6	0.9882(4)	0.6230(5)	0.2316(3)	0.0784(16)
H6	0.9927	0.7174	0.2313	0.094*
B7	1.0391(4)	0.5380(4)	0.1739(3)	0.0660(15)
H7	1.0761	0.5772	0.1359	0.079*
B8	1.0767(4)	0.4073(5)	0.2185(3)	0.0712(15)
H8	1.1392	0.3600	0.2100	0.085*
B9	1.0497(4)	0.4094(6)	0.3047(3)	0.0891(19)
H9	1.0941	0.3641	0.3526	0.107*
B10	1.0907(4)	0.5355(5)	0.2694(3)	0.0845(18)
H10A	1.1621	0.5722	0.2943	0.101*

The figure depicts a perspective of the complex showing the atom numbering. The environment of the Zn atom is six coordinated, in which two positions are occupied by the two sulphur atoms, four positions are occupied by the nitrogen atoms of two 1,10-phenanthroline ligand. The bond lengths of Zn1—S1 and Zn1—S2 are 2.413 Å and 2.411 Å, respectively. The bond lengths of Zn1—N1, Zn1—N2, Zn1—N3 and Zn1—N4 are 2.253 Å, 2.233 Å, 2.235 Å and 2.223 Å, respectively.

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