Redetermination of the crystal structure of dimeric bis(*N*,*N*-diethyldithiocarbamato)zinc, $[Zn(S_2CNEt_2)_2]_2$

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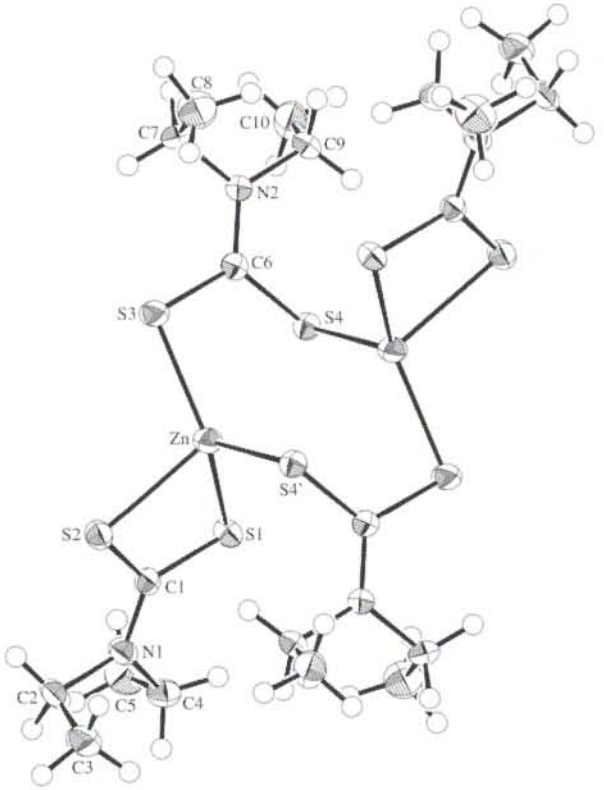

Abstract

$C_{10}H_{20}N_2S_4Zn$, monoclinic, $P12_1/n1$ (No. 14), a = 9.814(3) Å, b = 10.667(3) Å, c = 15.655(4) Å, β = 104.06(2)°, V = 1589.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.023, $wR_{ref}(F)$ = 0.025, T = 173 K.

Source of material

The title compound was prepared by reacting $Zn(SO_4)_2$ with two molar equivalents of the sodium salt of the ligand. The precipitate that formed was filtered off and crystals were obtained from the slow evaporation of a $CHCl_3$ solution of the compound; mp 448 K - 449 K.

Discussion

The structure of dimeric $[Zn(S_2CNEt_2)_2]$ [1, 2] has been redetermined with the advantages of contemporary methods. The dimeric structure is centrosymmetric and features an 8-membered -[S-C-S-Zn-]$_2$ ring in which two Zn atoms are bridged by two bidentate bridging dithiocarbamte ligands such that d(Zn—S(3), S(4)i) are 2.3354(9) a and 2.3874(8) Å, respectively; symmetry operation i: $-x,-y,1-z$. The remaining ligands are chelating, forming slightly asymmetric Zn—S bonds; d(Zn—S(1), S(2)) 2.3488(10) Å and 2.4433(9) Å, respectively. Thus, the coordination geometry for Zn is distorted tetrahedral with weak intra-ring Zn···S(4) contacts of 2.824(1) Å contributing to this distortion. The structure reported here is the common motif for $Zn(S_2CNR_2)_2$ structures [3].

Table 1. Data collection and handling.

Crystal:	pale-yellow block, size 0.13 × 0.18 × 0.24 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	20.52 cm^{-1}
Diffractometer, scan mode:	AFC7R, ω/2θ
$2\theta_{max}$:	55.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6237, 3855
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 2632
$N(param)_{refined}$:	154
Programs:	teXsan [4], DIRDIF92 [5], DIFABS [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.2767	0.3701	0.2536	0.039
H(2)	4*e*	0.1952	0.3943	0.1570	0.039
H(3)	4*e*	0.0210	0.4964	0.2132	0.046
H(4)	4*e*	0.1224	0.4873	0.3059	0.046
H(5)	4*e*	0.1631	0.5675	0.2332	0.046
H(6)	4*e*	-0.0776	0.1920	0.1480	0.038
H(7)	4*e*	-0.0330	0.3101	0.1037	0.038
H(8)	4*e*	0.1151	0.0859	0.1111	0.051
H(9)	4*e*	-0.0070	0.1181	0.0307	0.051
H(10)	4*e*	0.1273	0.2004	0.0524	0.051
H(11)	4*e*	0.4111	-0.3498	0.6621	0.036
H(12)	4*e*	0.4349	-0.2123	0.6369	0.036
H(13)	4*e*	0.3574	-0.1359	0.7509	0.056
H(14)	4*e*	0.2934	-0.2641	0.7672	0.056
H(15)	4*e*	0.4551	-0.2462	0.7913	0.056
H(16)	4*e*	0.1657	-0.4044	0.6620	0.035
H(17)	4*e*	0.0517	-0.3555	0.5822	0.035
H(18)	4*e*	0.2757	-0.5095	0.5697	0.051
H(19)	4*e*	0.1709	-0.4541	0.4879	0.051
H(20)	4*e*	0.1195	-0.5507	0.5472	0.051

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn	4*e*	0.11778(3)	0.07211(3)	0.44564(2)	0.0255(1)	0.0258(1)	0.0220(1)	0.0029(1)	0.00692(9)	0.0053(1)
S(1)	4*e*	0.00957(6)	0.07188(5)	0.29432(3)	0.0290(3)	0.0224(3)	0.0238(3)	–0.0042(2)	0.0074(2)	0.0007(2)
S(2)	4*e*	0.23219(6)	0.24451(6)	0.38743(4)	0.0253(3)	0.0287(3)	0.0245(3)	–0.0051(2)	0.0047(2)	0.0023(2)
S(3)	4*e*	0.29293(6)	–0.04008(5)	0.54350(4)	0.0249(3)	0.0223(3)	0.0354(3)	–0.0036(2)	–0.0017(2)	0.0073(2)
S(4)	4*e*	0.02432(6)	–0.17252(5)	0.47061(3)	0.0234(3)	0.0232(3)	0.0189(2)	–0.0009(2)	0.0032(2)	–0.0012(2)
N(1)	4*e*	0.1018(2)	0.2668(2)	0.2175(1)	0.027(1)	0.026(1)	0.0225(9)	–0.0022(8)	0.0078(7)	0.0039(8)
N(2)	4*e*	0.2343(2)	–0.2652(2)	0.5982(1)	0.0219(9)	0.0207(9)	0.0245(9)	0.0011(7)	0.0059(7)	0.0031(8)
C(1)	4*e*	0.1128(2)	0.2024(2)	0.2913(1)	0.021(1)	0.020(1)	0.025(1)	0.0025(8)	0.0099(8)	0.0001(9)
C(2)	4*e*	0.1862(3)	0.3810(2)	0.2154(2)	0.036(1)	0.033(1)	0.030(1)	–0.010(1)	0.010(1)	0.010(1)
C(3)	4*e*	0.1168(3)	0.4934(2)	0.2446(2)	0.037(1)	0.028(1)	0.047(2)	–0.006(1)	0.004(1)	0.009(1)
C(4)	4*e*	–0.0006(3)	0.2349(2)	0.1346(1)	0.034(1)	0.035(1)	0.023(1)	0.002(1)	0.004(1)	0.006(1)
C(5)	4*e*	0.0648(3)	0.1521(3)	0.0768(2)	0.053(2)	0.049(2)	0.027(1)	–0.003(1)	0.012(1)	–0.004(1)
C(6)	4*e*	0.1888(2)	–0.1696(2)	0.5454(1)	0.023(1)	0.021(1)	0.0197(9)	0.0015(8)	0.0079(8)	–0.0021(9)
C(7)	4*e*	0.3749(2)	–0.2668(2)	0.6588(2)	0.025(1)	0.030(1)	0.033(1)	0.004(1)	0.0011(9)	0.012(1)
C(8)	4*e*	0.3697(3)	–0.2243(3)	0.7507(2)	0.043(2)	0.063(2)	0.027(1)	–0.008(1)	–0.004(1)	0.007(1)
C(9)	4*e*	0.1481(2)	–0.3770(2)	0.6026(2)	0.030(1)	0.023(1)	0.033(1)	–0.002(1)	0.008(1)	0.009(1)
C(10)	4*e*	0.1817(3)	–0.4826(2)	0.5467(2)	0.035(1)	0.023(1)	0.069(2)	–0.002(1)	0.013(1)	–0.003(1)

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