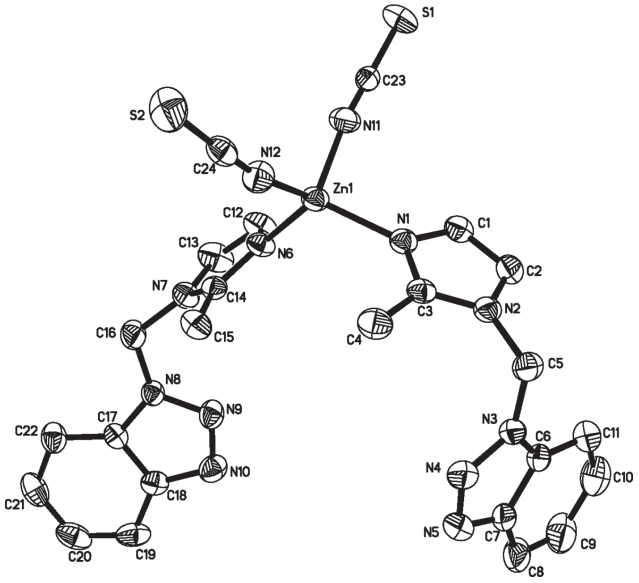
Hai-Yan Yang*, Yao-Min Zhao, Fang He, Hui Li, Hong-Fang Wang and Ying-Hua Li

Crystal structure of bis{1-[(benzotriazol-1-yl)-methyl]-1-*H*-1,3-(2-methyl-imdazol)-κ*N*}-dithiocyano-κN-zinc(II) $C_{24}H_{22}N_{12}S_2Zn$



https://doi.org/10.1515/ncrs-2018-0047
Received March 12, 2018; accepted June 12, 2018; available online June 30, 2018

Abstract

$C_{24}H_{22}N_{12}S_2Zn$, monoclinic, *C*2/*c* (no. 15), $a = 27.6123(9)$ Å, $b = 9.6238(4)$ Å, $c = 21.1748(8)$ Å, $\beta = 101.088(4)°$, $Z = 8$, $V = 5521.8(4)$ Å^3, $R_{gt}(F) = 0.0481$, $wR_{ref}(F^2) = 0.1047$, $T = 291(2)$ K.

CCDC no.: 1848839

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

Source of material

A methanol solution (4 mL) of 1-((benzotriazol-1-yl)methyl)-1-*H*-1,3-(2-methyl-imdazol) (0.1 mmol) was added dropwise into a methanol solution (2 mL) of $ZnSO_4$ (0.05 mmol), giving a clear solution. KSCN (0.1 mmol) in 4 mL of methanol was added dropwise to the above solution. The resulting solution was left at room temperature. After three days, colorless crystals were obtained and dried in air.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.2 × 0.16 × 0.15 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.08 mm^{-1}
Diffractometer, scan mode:	Xcalibur, Eos, Gemini, *ω*-scans
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11687, 5632, 0.028
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3654
$N(param)_{refined}$:	354
Programs:	CrysAlis^{PRO} [1], SHELX [2], OLEX2 [3]

Experimental details

H atoms were generated geometrically to ride their parent atoms with $U_{iso}(H) =$ x times $U_{eq}(C)$, where $x = 1.5$ for methyl H and $x = 1.2$ for other H atoms.

Discussion

The multidentate *N*-heterocyclic compounds, such as imidazole, triazole, tetrazole and their derivative, have rich coordination sites and can result in coordination polymers [4–8]. We synthesized a *N*-heterocyclic compound, 1-[(benzotriazol-1-yl)methyl]-1-*H*-1,3-(2-methyl-imdazol)(bmi), and studied its coordination behavior with metal halides ($ZnCl_2$, $CoCl_2$, CdI_2, $HgCl_2$, $HgBr_2$) [9–11]. As a continuation, we use this N-heterocyclic compound, to react with $ZnSO_4$ in the presence KSCN, generating the title complex.

Single crystal X-ray diffraction analysis reveals that it is a mononuclear complex. As is shown in the figure, the Zn atom is four-coordinated by two bmi ligands (with Zn—N bond lengths of 1.994(2) Å and 1.999(2) Å), and two SCN-ligands (with Zn-N bond lengths of 1.970(3) Å and 1.946(3) Å) in a distorted tetrahedral geometry. The bond angles around the Zn atom vary from 101.28(11)° (N11-Zn1—N1) to 117.66(10)° (N1—Zn1—N6).

***Corresponding author: Hai-Yan Yang,** School of Materials and Chemical Engineering, Zhongyuan University of Technology, Zhengzhou 450007, P.R. China, e-mail: yanghy0204@163.com
Yao-Min Zhao, Fang He, Hui Li, Hong-Fang Wang and Ying-Hua Li: School of Materials and Chemical Engineering, Zhongyuan University of Technology, Zhengzhou, 450007, P.R. China

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.385078(12)	0.50497(4)	0.522998(16)	0.04907(13)
S1	0.33921(4)	0.26801(12)	0.32652(4)	0.0790(3)
S2	0.37640(5)	0.97936(10)	0.47365(6)	0.0924(4)
N1	0.45228(9)	0.4224(3)	0.54790(11)	0.0478(6)
N2	0.52652(9)	0.3620(3)	0.59665(11)	0.0505(7)
N3	0.57013(9)	0.3117(3)	0.70400(12)	0.0525(7)
N4	0.56400(11)	0.4057(3)	0.74969(15)	0.0701(8)
N5	0.56059(12)	0.3385(4)	0.80159(14)	0.0809(10)
N6	0.33499(9)	0.4546(3)	0.57588(11)	0.0493(6)
N7	0.28256(10)	0.4659(3)	0.64082(13)	0.0555(7)
N8	0.27054(9)	0.4281(3)	0.74856(12)	0.0470(6)
N9	0.31009(9)	0.3424(3)	0.75922(14)	0.0583(7)
N10	0.31506(9)	0.2922(3)	0.81713(14)	0.0640(8)
N11	0.36038(10)	0.4101(3)	0.44064(14)	0.0651(8)
N12	0.38726(10)	0.7060(3)	0.51429(14)	0.0632(8)
C1	0.46310(12)	0.2926(4)	0.52633(15)	0.0539(8)
H1	0.4421	0.2399	0.4959	0.065*
C2	0.50837(12)	0.2546(4)	0.55594(16)	0.0579(9)
H2	0.5244	0.1720	0.5502	0.069*
C3	0.49159(11)	0.4610(3)	0.59064(14)	0.0456(8)
C4	0.49678(13)	0.5938(3)	0.62662(16)	0.0638(9)
H4A	0.4930	0.5773	0.6701	0.096*
H4B	0.4719	0.6577	0.6064	0.096*
H4C	0.5288	0.6324	0.6267	0.096*
C5	0.57407(11)	0.3613(4)	0.64085(15)	0.0602(9)
H5A	0.5969	0.3025	0.6235	0.072*
H5B	0.5874	0.4548	0.6445	0.072*
C6	0.57082(11)	0.1812(4)	0.72770(15)	0.0522(8)
C7	0.56457(12)	0.1999(5)	0.79056(16)	0.0638(10)
C8	0.56277(15)	0.0869(6)	0.8306(2)	0.0916(14)
H8	0.5586	0.0986	0.8728	0.110*
C9	0.56723(16)	−0.0394(5)	0.8064(3)	0.0975(15)
H9	0.5659	−0.1167	0.8324	0.117*
C10	0.57387(15)	−0.0595(5)	0.7432(3)	0.0914(13)
H10	0.5766	−0.1496	0.7284	0.110*
C11	0.57644(13)	0.0503(4)	0.70226(19)	0.0711(10)
H11	0.5816	0.0378	0.6605	0.085*
C12	0.29925(13)	0.3552(4)	0.55824(17)	0.0697(10)
H12	0.2978	0.2936	0.5241	0.084*
C13	0.26680(13)	0.3606(4)	0.59784(18)	0.0743(11)
H13	0.2392	0.3046	0.5965	0.089*
C14	0.32429(11)	0.5195(3)	0.62634(14)	0.0471(8)
C15	0.35358(13)	0.6322(4)	0.66231(16)	0.0627(9)
H15A	0.3334	0.7137	0.6615	0.094*
H15B	0.3814	0.6527	0.6428	0.094*
H15C	0.3650	0.6036	0.7061	0.094*
C16	0.25745(13)	0.5110(4)	0.69075(16)	0.0654(10)
H16A	0.2221	0.5054	0.6751	0.079*
H16B	0.2657	0.6075	0.7010	0.079*
C17	0.24966(10)	0.4349(3)	0.80207(15)	0.0449(7)
C18	0.27829(11)	0.3478(4)	0.84522(15)	0.0514(8)
C19	0.26836(13)	0.3287(4)	0.90708(16)	0.0711(11)
H19	0.2874	0.2698	0.9368	0.085*
C20	0.22949(15)	0.4006(5)	0.92156(19)	0.0788(12)
H20	0.2218	0.3901	0.9622	0.095*
C21	0.20094(15)	0.4891(4)	0.8777(2)	0.0751(11)
H21	0.1751	0.5369	0.8902	0.090*
C22	0.20958(12)	0.5086(4)	0.81669(18)	0.0645(10)
H22	0.1901	0.5667	0.7871	0.077*
C23	0.35170(10)	0.3517(3)	0.39468(16)	0.0457(8)
C24	0.38299(12)	0.8201(4)	0.49858(16)	0.0555(9)

Acknowledgements: The authors are grateful to Zhongyuan University of Technology for financial support and thank Professor Hong-Wei Hou of Zhengzhou University for his help.

References

1. Agilent Technologies: CrysAlis^{PRO} Software system, version 1.171.35.15, Agilent Technologies UK Ltd, Oxford, UK (2011).
2. Sheldrick, G. M.: A short history of *SHELX*. Acta Crystallogr. **A64** (2008) 112–122.
3. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: a complete structure solution, refinement and analysis program. J. Appl. Cryst. **42** (2009) 339–341.
4. Seth, S.; Matzger, A. J.: Coordination polymerization of 5,5′-dinitro-2*H*,2*H*′-3,3′-bi-1,2,4-triazole leads to a dense explosive with high thermal stability. Inorg. Chem. **56** (2017) 561–565.
5. Yang, Y.; Jiang, F. L.; Liu, C. P.; Chen, L.; Gai, Y. L.; Pang, J. D.; Su, K. Z.; Wan, X. Y.; Hong, M. C.: Self-assembly syntheses, structural characterization, and luminescent properties of lanthanide coordination polymers constructed by three triazole-carboxylate ligands. Cryst. Growth Des. **16** (2016) 2266–2276.
6. Yang, H.-Y.; Li, L.-K.; Wu, J.; Hou, H.-W.; Xiao, B.; Fan, Y.-T.: 3D coordination framework with uncommon two-fold interpenetrated $\{3^3.5^9.6^3\}$-lcy net and coordinated anion exchange. Chem. Eur. J. **15** (2009) 4049–4056.
7. Weiss, D. T.; Haslinger, S.; Jandl, C.; Pothig, A.; Cokoja, M.; Kühn, F. E.: Application of open chain tetraimidazolium salts as precursors for the synthesis of silver tetra(NHC) complexes. Inorg. Chem. **54** (2015) 415–417.
8. Cabeza, J. A.; García-Álvarez, P.; Pérez-Carreno, E.; Pruneda, V.: Synthesis and reactivity of cationic triruthenium clusters derived from 2-methyl- and 4-methylpyrimidines: from conventional cyclometalated ligands to novel types of *N*-heterocyclic carbenes. Chem. Eur. J. **19** (2013) 3426–3436.
9. Yang, H.-Y.; Li, Y.-H.; Chen, H.: Crystal structure of bis{1-[(benzotriazol-1-yl)methyl]-1-*H*-1,3-(2-methyl-imdazol)}-diiodidoccadmium(II), $[Cd(C_{11}H_{11}N_5)_2I_2]$, $C_{22}H_{22}CdI_2N_{10}$. Z. Kristallogr. NCS **227** (2012) 527–528.

10. Yang, H.-Y.; Li, Y.-H.: Crystal structure of bis{1-[(benzotriazol-1-yl)methyl]-1-H-1,3-(2-methyl-imdazol)}dibromidomercury(II), $[Hg(C_{11}H_{11}N_5)_2Br_2]$, $C_{22}H_{22}HgBr_2N_{10}$. Z. Kristallogr. NCS **229** (2014) 83–84.

11. Yang, H.-Y.: Crystal structure of aquadichloridobis(1-((2-methyl-1*H*-imidazol-1-yl)methyl)-1*H*-benotriazole-*N*)mercury(II), $C_{22}H_{24}HgCl_2N_{10}O$. Z. Kristallogr. NCS **231** (2016) 687–688.