Zhuang-Yu Li, E. Liu, Xiao-Jing Liu, Fang-Fang Jian* and Tong-ling Liang

Crystal structure of bis(*N*,2-bis(4-ethoxybenzylidene)hydrazine-1-carbohydrazonothioato- $\kappa^2 N,S$)nickel(II) – *N,N*-dimethylformamide (1/2), $C_{44}H_{56}N_{10}S_2O_6Ni$

Abstract

$C_{44}H_{56}N_{10}S_2O_6Ni$, triclinic, $P\bar{1}$ (no. 2), $a = 9.3077(14)$ Å, $b = 9.9624(14)$ Å, $c = 14.0449(19)$ Å, $\alpha = 70.889(3)^\circ$, $\beta = 75.532(2)^\circ$, $\gamma = 87.544(3)^\circ$, $V = 1190.5(3)$ Å³, $Z = 1$, $R_{gt}(F) = 0.0528$, $wR_{ref}(F^2) = 0.1279$, $T = 293$ K.

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The molecular structure is shown in the figure ($' = 1 - x, -y, -z$). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	$0.29 \times 0.26 \times 0.25$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.55 mm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD-4, ω
$\theta_{\max}$, completeness:	25.0° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7848, 4160, 0.033
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2365
$N(\text{param})_{\text{refined}}$:	286
Programs:	CAD-4 [1, 2], Olex2 [3]

Source of material

Dissolve hydrazinecarbothiohydrazide (3 mmol) and 4-methylbenzenesulfonic acid (1 mL) in 50 mL of distilled water, add 15 mL of an ethanol solution containing 4-ethoxybenzaldehyde (6 mmol), heat and stir under reflux for 4 h: Then add nickel chloride (2 mmol), and continue heating and stirring for 2 h. After the reaction was completed, the product was filtered and washed with distilled water, and the title compound was finally obtained from the DMF solution.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Schiff bases of thiosemicarbazone have strong coordination ability due to the fact that their structures contain O, N, S and heteroatoms, and they are important in the fields of medicine

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***Corresponding author: Fang-Fang Jian**, School of Chemical Engineering and Pharmaceutics Henan, University of Science and Technology, Luoyang Hekeda Recycling Energy Co.LTD., Luoyang, Henan 471003, P.R. China, e-mail: Fangfjian@126.com

Zhuang-Yu Li and Xiao-Jing Liu: School of Chemical Engineering and Pharmaceutics Henan, University of Science and Technology, Luoyang, Henan 471003, P.R. China. <https://orcid.org/0000-0003-4445-1455> (Z.-Y. Li)

E. Liu: School of Chemical Engineering and Pharmaceutics Henan, University of Science and Technology, Luoyang Hekeda Recycling Energy Co.LTD., Luoyang, Henan 471003, P.R. China

Tong-ling Liang: Institute of Chemistry Chinese Academy of Sciences, Chemistry Chinese Academy of Sciences, Beijing, Beijing 100190, P.R. China

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

Atom	x	y	z	U _{iso} */U _{eq}
Ni1	0.5	0.0	0.0	0.0505(2)
S1	0.44175(12)	0.07894(12)	0.13065(8)	0.0696(4)
O1	1.3063(3)	−0.3961(3)	0.0698(2)	0.0847(9)
O2	0.1682(3)	0.4763(3)	0.5961(2)	0.0769(9)
N1	0.6761(3)	−0.0660(3)	0.0461(2)	0.0497(8)
N2	0.7057(3)	−0.0372(3)	0.1300(2)	0.0554(8)
N3	0.6223(3)	0.0648(3)	0.2529(2)	0.0630(9)
H3	0.7013(3)	0.0389(3)	0.2742(2)	0.0756(11)*
N4	0.5205(3)	0.1387(3)	0.3019(2)	0.0552(8)
C1	1.5206(5)	−0.4611(5)	0.1307(4)	0.0990(17)
H1a	1.5752(5)	−0.4516(5)	0.1780(4)	0.148(2)*
H1b	1.4941(5)	−0.5596(5)	0.1470(4)	0.148(2)*
H1c	1.5809(5)	−0.4251(5)	0.0607(4)	0.148(2)*
C2	1.3794(5)	−0.3767(5)	0.1412(3)	0.0789(14)
H2a	1.4041(5)	−0.2767(5)	0.1248(3)	0.0947(16)*
H2b	1.3168(5)	−0.4121(5)	0.2115(3)	0.0947(16)*
C3	1.1802(5)	−0.3231(4)	0.0585(3)	0.0652(11)
C4	1.1164(4)	−0.2342(4)	0.1129(3)	0.0619(11)
H4	1.1613(4)	−0.2166(4)	0.1601(3)	0.0743(13)*
C5	0.9850(4)	−0.1710(4)	0.0971(3)	0.0577(10)
H5	0.9425(4)	−0.1110(4)	0.1342(3)	0.0692(12)*
C6	0.9157(4)	−0.1949(4)	0.0275(3)	0.0534(10)
C7	0.9876(5)	−0.2802(5)	−0.0283(4)	0.0853(14)
H7	0.9461(5)	−0.2950(5)	−0.0780(4)	0.1024(17)*
C8	1.1159(5)	−0.3433(5)	−0.0138(4)	0.0892(15)
H8	1.1604(5)	−0.4002(5)	−0.0529(4)	0.1070(18)*
C9	0.7770(4)	−0.1376(4)	0.0043(3)	0.0587(10)
H9	0.7569(4)	−0.1563(4)	−0.0519(3)	0.0705(13)*
C10	0.6009(4)	0.0317(4)	0.1715(3)	0.0500(10)
C11	0.5462(4)	0.1583(4)	0.3818(3)	0.0593(11)
H11	0.6293(4)	0.1197(4)	0.4045(3)	0.0712(13)*
C12	0.4480(4)	0.2396(4)	0.4375(3)	0.0546(10)
C13	0.4697(5)	0.2497(4)	0.5288(3)	0.0668(12)
H13	0.5479(5)	0.2025(4)	0.5539(3)	0.0802(14)*
C14	0.3800(5)	0.3269(5)	0.5844(3)	0.0673(12)
H14	0.3973(5)	0.3306(5)	0.6460(3)	0.0808(14)*
C15	0.2658(5)	0.3977(4)	0.5478(3)	0.0605(11)
C16	0.2388(5)	0.3886(4)	0.4576(3)	0.0647(11)
H16	0.1594(5)	0.4349(4)	0.4337(3)	0.0777(14)*
C17	0.3290(5)	0.3114(4)	0.4033(3)	0.0601(11)
H17	0.3103(5)	0.3070(4)	0.3423(3)	0.0721(13)*
C18	0.1961(5)	0.5008(5)	0.6847(3)	0.0759(13)
H18a	0.1898(5)	0.4116(5)	0.7411(3)	0.0911(15)*
H18b	0.2948(5)	0.5439(5)	0.6672(3)	0.0911(15)*
C19	0.0818(6)	0.5980(5)	0.7168(4)	0.1047(17)
H19a	0.0981(6)	0.6163(5)	0.7764(4)	0.157(2)*
H19b	0.0893(6)	0.6860(5)	0.6606(4)	0.157(2)*
H19c	−0.0154(6)	0.5543(5)	0.7342(4)	0.157(2)*
O3	−0.1136(4)	1.0328(4)	0.3243(3)	0.0936(10)
N5	0.0810(4)	0.9072(4)	0.3676(2)	0.0656(10)
C20	−0.0441(5)	0.9260(5)	0.3381(3)	0.0730(13)
H20	−0.0826(5)	0.8496(5)	0.3267(3)	0.0876(15)*
C21	0.1498(5)	1.0201(5)	0.3848(4)	0.0963(16)
H21a	0.2499(12)	1.038(2)	0.3424(18)	0.144(2)*
H21b	0.095(2)	1.1046(10)	0.367(2)	0.144(2)*
H21c	0.151(3)	0.9936(14)	0.4568(6)	0.144(2)*
C22	0.1572(6)	0.7750(5)	0.3808(4)	0.1135(19)
H22a	0.2456(6)	0.7824(5)	0.4025(4)	0.170(3)*
H22b	0.0932(6)	0.6988(5)	0.4326(4)	0.170(3)*
H22c	0.1834(6)	0.7561(5)	0.3159(4)	0.170(3)*

and analytical chemistry [4, 5]. In the field of medicine, thiourea Schiff base complexes have biological activities such as bacteriostasis [6, 7] and antitumor effects [8, 9]. In the field of optical materials, thiourea Schiff base ligands with specific structures and their complexes have shown to own good optical properties, such as fluorescent light, second-order nonlinear optics, etc. [10].

The molecular structure of the title compound is shown in the figure, bond lengths and angles are within normal ranges [11]. In the complex, the N1–N2 bond length is 1.392 Å and the N3–N4 bond length is 1.364 Å. Ni coordinates with N and S to form a five-membered chelate ring, in which the bond lengths of Ni1–N1 and Ni1–N1' are 1.923 Å, and the bond lengths of Ni1–S1 and Ni1–S1' are 2.166 Å. The five-membered ring of atoms Ni, S1, N1, N2, and C10 is in the same plane as the five-membered ring of atoms Ni, S1', N1', N2', and C10a. The dihedral angle between the plane of the aryl moiety formed by atoms C(1)–C(6) and the moiety of atoms C(12)–C(17) is 14.03°.

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