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ORIGINAL PAPER

Two Complexes from One Reaction of Nickel(II) Ion with a New Schiff Base Ligand

Hong Yan Wang · Pu Su Zhao · Jie Song · Rong Qing Li

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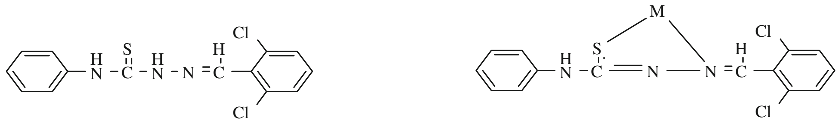
Abstract A new Schiff base ligand of 1-(2, 6-dichlorobenzylidene)-4-phenylthiosemicarbazide($C_{14}H_{11}Cl_2N_3S$) has been synthesized, which crystallizes in monoclinic, space group *C2/c* with $a = 18.551(7)$ Å, $b = 6.963(3)$ Å, $c = 23.185(9)$ Å and $\beta = 93.122(6)^\circ$. The reaction of this ligand with $NiAc_2 \cdot 4H_2O$ gives two different Ni(II) complexes, one is general traditional mononuclear and another is unexpected trinuclear, where another quadridentate ligand of 1-(amino-*N*-phenylmethanethio)-4-phenyl-thiosemicarbazide($C_{14}H_{14}N_4S_2$) is found. The mononuclear complex ($C_{32}H_{26}Cl_4N_8NiS_2$) crystallizes in triclinic, space group *P*−1 with $a = 11.071(2)$ Å, $b = 11.157(2)$ Å, $c = 15.302(3)$ Å, $\alpha = 76.214(2)^\circ$, $\beta = 87.902(2)^\circ$ and $\gamma = 79.586(3)^\circ$. The trinuclear complex ($C_{60}H_{50}Cl_4N_{16}Ni_3S_6$) crystallizes in orthorhombic, space group *Pbcn* with $a = 19.673(3)$ Å, $b = 16.094(3)$ Å and $c = 20.465(4)$ Å. In above three compounds, there are some hydrogen bonds which help to construct the three dimensional net works and stabilize the molecular structures.

Keywords Schiff base · Mononuclear Ni(II) complex · Trinuclear Ni(II) complex · Hydrogen bond

Introduction

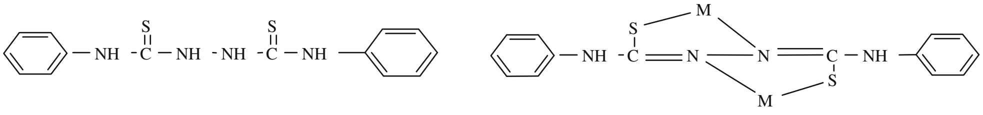
Reflecting their usual relative ease of synthesis and versatile metal complexing properties, Schiff base and its metallic complexes have been extensively investigated for more than a century and have been employed in many areas that include magnetochemistry [1, 2], non-linearoptics [3], photophysical studies [4], analytical chemistry [5] and materials chemistry [6]. Some Schiff base metal complexes have also been used as biological models [7] and as catalysts for organic reactions [8]. On the other hand, thiosemicarbazides are interesting because they form highly stable and intensely colored complexes which are used for spectrophotometric determination of metal ions in different media [9, 10] and showed catalytic activity [11, 12]. They also have potentially beneficial as antibacterial and anticancer agents [13, 14]. Pushed by aforementioned reasons, in our laboratory, we have designed and synthesized two thiosemicarbazide derivatives containing Schiff base group and their complexes [15, 16]. As one of our products, the ligand of 1-(2, 6-dichlorobenzylidene)-4-phenylthiosemicarbazide (HL) has been synthesized and its crystal structure has also been obtained. Traditionally, the HL is regarded as a bisdentae chelate ligand and the S atom from C=S bond as well as the N atom form C=N will be two potential sites to bite with a metallic ion, which will give a mononuclear complex (Fig. 1). However, unexpected, when the HL reacted with $NiAc_2$, two different Ni(II) complexes are obtained, one is a general mononuclear complex (**1**) and another is a tirnulear complex (**2**). In the trinuclear Ni(II) complex, a new quadridentate ligand of 1-(amino-*N*-phenylmethanethio)-4-phenyl-thiosemicar-bazide (H_2Q) has been found (Fig. 1), which along with the original ligand of HL to take part into coordinating with three Ni^{2+}centers. Herein, we will give a report about the synthesis of the Schiff base ligands and the two complexes as well as their characterizations and crystal structures.

H. Y. Wang · P. S. Zhao (✉) · J. Song · R. Q. Li
Jiangsu Key Laboratory for Chemistry of Low-Dimensional Materials, Huaiyin Normal University, Huaian 223300, Jiangsu, People's Republic of China
e-mail: zhaopusu@163.com



HL

Potential biding modelof HL



H_2Q

Potential biding modelof H_2Q

Fig. 1 The potential binding model of the ligands HL and H_2Q

Experimental

Materials and Instrument

Elemental analyses for carbon, hydrogen and nitrogen were performed by a Perkin-Elmer 240C elemental instrument. The melting points were determined on a Yanaco MP-500 melting point apparatus. IR spectra (4000–400 cm^{-1}), as KBr pellets, were recorded on a Nicolet FT-IR spectrophotometer. ^1H NMR spectroscopy (DMSO-*d*6) were recorded on Avance Mercury plus-400 instrument with TMS as an internal standard.

Synthesis of the HL and Two Complexes

All chemicals were obtained from a commercial source and used without further purification.

The HL was synthesized as follows: 4-phenylthiosemicarbazide (8.35 g, 0.05 mol) and 2,6-dichlorobenzaldehyde (8.75 g, 0.05 mol) were mixed in acetonitrile solution (200 mL) and stirred with refluxing. Eight hour later, light-yellow solids were observed and then, the reaction was stopped and the mixture was cooled to room temperature. The yellow solids were obtained by filtration and dried at room temperature. Yield: 88%. Mp. 192.0–193.2 °C. Anal. Calcd. for $C_{14}H_{11}Cl_2N_3S$: C, 51.86; H, 3.42; N, 12.96. Found: C, 51.68; H, 3.39; N, 12.81. ^1H NMR (400 MHz, DMSO): δ 2.51 (s, H, $-$N**H**), 3.34 (s, H, $-$N**H**), 7.17–7.65 (m, 8H, Ar**H**–), 8.42(s, H, $-$C**H**). IR (KBr): υ 3450, 3337, 3124, 2981, 1536, 1505, 1448, 1426, 1268, 1203, 1076, 776, 695, 528 cm^{-1}; Crystals suitable for an X-ray structure determination were obtained by slowly evaporating an ethanol solution of the compound in air.

The mononuclear Ni(II) complex **(1)** and trinulclear Ni(II) complex **(2)** were then obtained: to a warm solution of HL (3.24 g, 10.0 mmol) in acetonitrile (40 mL) was added with stirring $NiAc_2 \cdot 4H_2O$ (1.25 g, 5.0 mmol) and the mixture was refluxed for 2 h. The yellow–brown solution was filtered, and the filtrate was evaporated by heating until about 30 mL solution was left and then, the left solution was cooled to room temperature. Four days later, two kinds of crystals suitable for an X-ray structure determination were obtained by slowly evaporating an acetonitrile solution in air, one kind of crystals was green and anther kind was dark-green. For the green one (yield 87%), Anal. Calcd. for $C_{32}H_{26}Cl_4N_8NiS_2$: C, 48.82; H, 3.33; N, 14.24. Found: C, 48.68; H, 3.39; N, 14.41. IR (KBr): υ 3430, 1596, 1507, 1432, 1314, 1249, 1191, 1044, 775, 689 cm^{-1}. For the dark-green one (yield 4%), Anal. Calcd. for $C_{60}H_{50}Cl_4N_{16}Ni_3S_6$: C, 47.87; H, 3.35; N, 14.89. Found: C, 47.71; H, 3.24; N, 14.62. IR (KBr): υ 3430, 1596, 1536, 1505, 1433, 1385, 1314, 1249, 1081, 1047, 996, 762, 689, 485 cm^{-1}.

Structure Determination

The crystal structures of the HL and the complexes **1** and **2** were determined based on data collected on a Bruker Smart APEX2 CCD diffractometer with graphite-monchromated Mo-Kα radiation [$\lambda = 0.71073$ Å, $T = 296(2)$ K]. The empirical absorption correction was based on equivalent reflections, and other possible effects, such as absorption by the glass fiber, were simultaneously corrected. The structures of the three compounds were solved by direct methods and refined by least squares on F^2. The hydrogen atom positions were fixed geometrically at calculated distances and allowed to ride on the parent carbon or nitrogen atoms. All non-hydrogen atoms were anisotropically refined. The SMART software was used for collecting frames of data, indexing reflections, and determination of lattice constants; SAINT-PLUS for integration of intensity of reflections and scaling; SADABS for absorption correction; and SHELXTL for space groups and structure determinations, refinements, graphics, and structure reporting [17–19].

The crystallographic collection and refinement parameters for the three compounds are listed in Table 1.

Table 1 Summary of crystallographic results for HL and two Ni(II) complexes

Empirical formula	HL $C_{14}H_{11}Cl_2N_3S$	**1** $C_{32}H_{26}Cl_4N_8NiS_2$	**2** $C_{60}H_{50}Cl_4N_{16}Ni_3S_6$
Formula weight	324.22	787.24	1505.45
Temperature (K)	296(2)	296(2)	296(2)
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system	Monoclinic	Triclinic	Orthorhombic,
Space group	*C2/c*	*P*−1	*Pbcn*
Unit cell dimensions (Å, °)	$a = 18.551(7)$	$a = 11.071(2)$	$a = 19.673(3)$
	$b = 6.963(3)$	$b = 11.157(2)$	$b = 16.094(3)$
	$c = 23.185(9)$	$c = 15.302(3)$	$c = 20.465(4)$
		$\alpha = 76.214(2)$	
	$\beta = 93.122(6)$	$\beta = 87.902(2)$	
		$\gamma = 79.586(3)$	
Volume (Å^3)	2990(2)	1805.4(6)	6479.6(19)
Z	8	2	4
Calculated density (g/cm^3)	1.440	1.448	1.543
Absorption coefficient (mm^{-1})	0.566	0.985	1.275
F(000)	1328	804	3080
θ range for data collection (°)	1.76–25.50	1.37–25.00	1.91–25.00
Limiting indices	$-22 \le h \le 18$	$-11 \le h \le 13$	$-20 \le h \le 23$
	$-8 \le k \le 8$	$-13 \le k \le 13$	$-14 \le k \le 18$
	$-22 \le l \le 28$	$-18 \le l \le 18$	$-24 \le l \le 24$
Reflections collected/unique	6,994/2,750	12,862/6,297	29,145/5,643
R_{int}	0.0985	0.0391	0.1031
Data/restraints/parameters	2,750/12/181	6,297/0/429	5,643/0/403
Goodness-of-fit on F^2	0.927	0.962	0.950
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0603$	$R_1 = 0.0483$	$R_1 = 0.0577$
	$wR_2 = 0.1395$	$wR_2 = 0.1180$	$wR_2 = 0.1374$
R indices (all data)	$R_1 = 0.2275$	$R_1 = 0.0948$	$R_1 = 0.1370$
	$wR_2 = 0.1690$	$wR_2 = 0.1368$	$wR_2 = 0.1623$
Largest diff. peak and hole (e. Å^{-3})	0.306 and −0.279	0.484 and −0.351	0.578 and −0.442

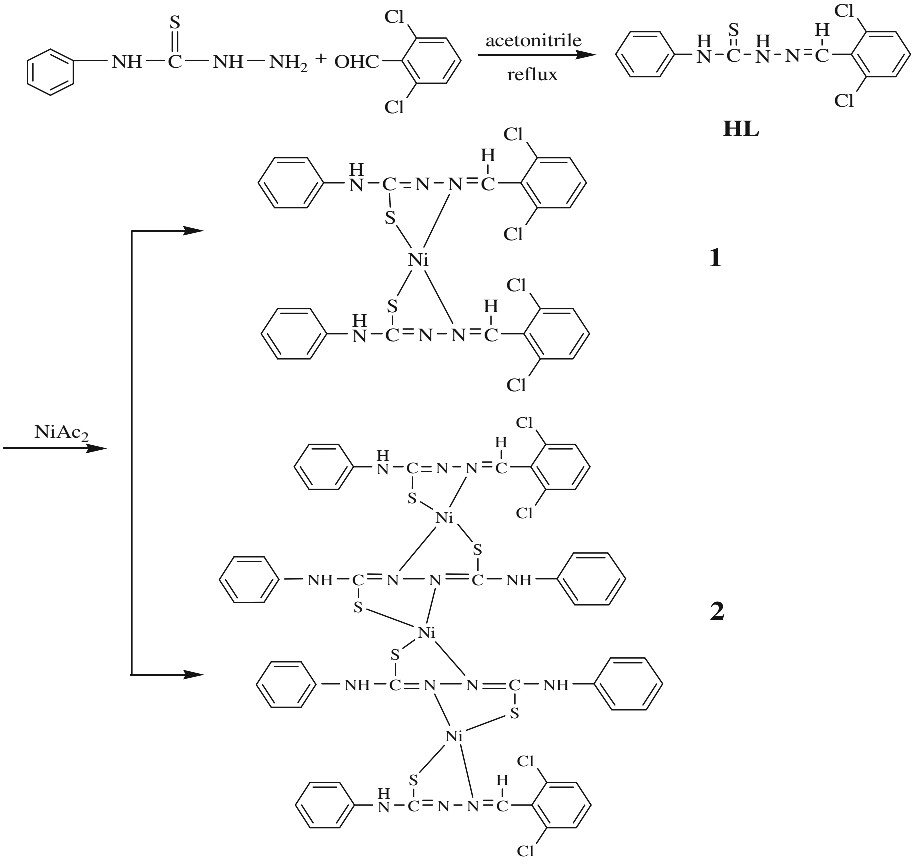
Results and Discussion

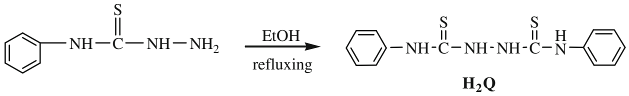
Synthesis

The synthetic path for HL and the two Ni(II) complexes is shown in Scheme 1.

The original intention of this work is to synthesize the ligand of HL and its mononuclear Ni(II) complex. However, the experimental results show that an unexpected trinuclear Ni(II) complex is found with low yield, where there is a new ligand of H_2Q. After analyzing the structure of H_2Q, we think that, maybe, the H_2Q is a condensation product of two 4-phenylthiosemicarbazides. In order to prove above supposition, a condensation reaction of 4-phenylthiosemicarbazide was done as below: To a 100 mL flask, 4-phenylthiosemicarbazide (0.8350 g, 5 mmol) and anhydrous ethanol (50 mL) were added in. After the mixture was refluxing with stirring for 6 h, white suspended solids were obtained. Then, the reaction was stopped. The mixtures were cooled to room temperature and filtered. The white solids were washed with anhydrous ethanol and then dried in vacuum drier. Finally, white powders were obtained. Yield (0.6286 g, 83.25%). The melting point of the white solids is 177.6 ~ 178.6 °C, which is almost the same with that of H_2Q reported in earlier literature (178 ~ 179 °C) [20]. IR (KBr) *v*: 3212, 3113, 1547, 1508, 1190, 694 cm^{-1}. ^{1}H NMR (400 MHz, DMSO−*d*6): δ 9.89 (s, 2H, Ph−N**H**), 9.69 (s, 2H, −N**H**), 7.56 ~ 7.54 (d, 4H, Ph−**H**), 7.36 ~ 7.32 (t, 4H, Ph–**H**), 7.18 ~ 7.14(t, 2H, Ph–**H**); MS (*m/z*): 303 (M + 1). According to experimental results, the possible synthetic path of H_2Q is showed in Scheme 2. Namely, maybe, the H_2Q is a by-product during the synthesis of HL.

Scheme 1 Synthesis path for HL and two complexes





Scheme 2 The possible synthetic path for the ligand of H2Q

Crystal Structure Descriptions for HL and the Two Complexes

For the three compounds, the displacement ellipsoid plots with the atomic numbering scheme are shown in Figs. 2, 3, and 4 and the perspective views of the crystal packing in the unit cell are showed in Figs. 5, 6, and 7. Some selected bond lengths and bond angles for the three compounds are listed in Table 2.

The crystal structure of the HL consists of a monomeric 1-(2, 6-dichlorobenzylidene)-4-phenylthiosemicarbazide. In HL, the two phenyl rings are both located at the opposite sides of C=S bond and all of the bond lengths and bond angles are comparable with those in the similar compounds derived from aldehydes [21–26]. The dihedral angle between the two phenyl rings is 29.85(2)°.

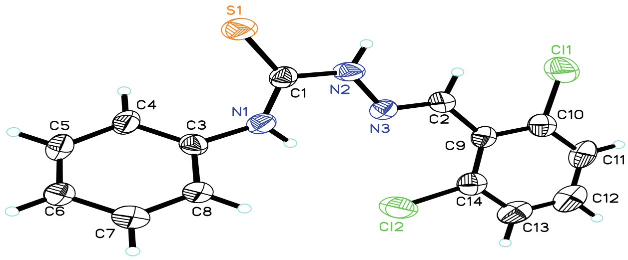


Fig. 2 The displacement ellipsoid plot for the HL with the atomic numbering scheme

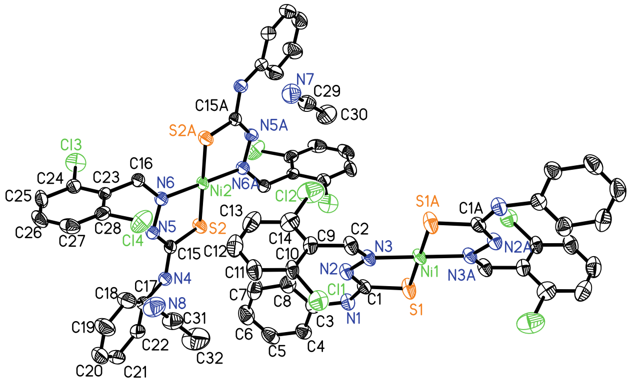


Fig. 3 The displacement ellipsoid plot for the mononuclear Ni(II) complex **1** (only the crystallographic independent atoms and the Ni coordination spheres and atoms are labeled for the clarity purpose)

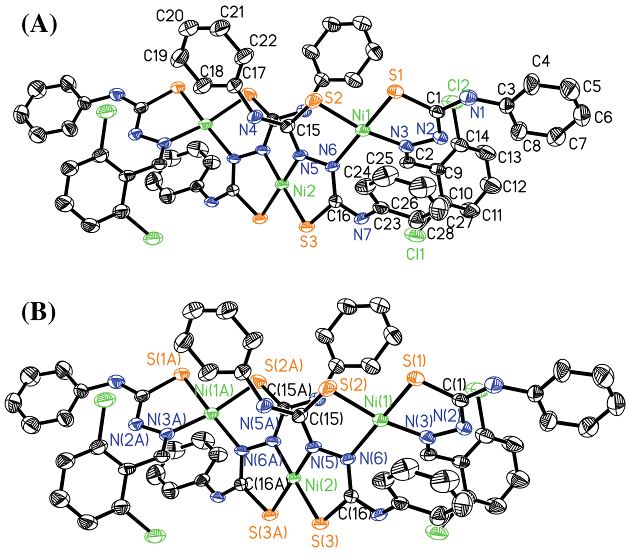


Fig. 4 The displacement ellipsoid plot for the trinuclear Ni(II) complex **2**. The solvent CH_3CN molecule is omitted for the clarity purpose. In **a** only the crystallographic independent atoms are labeled, in **b** only the Ni coordination spheres and atoms are labeled

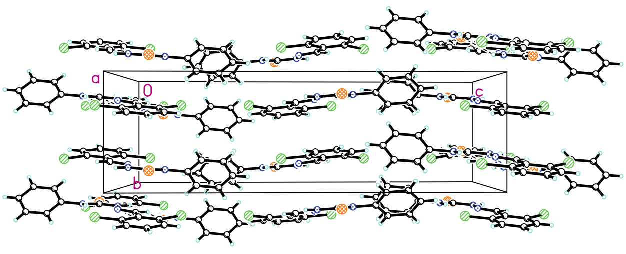


Fig. 5 Packing diagram of the unit cell along the *a*-axial for the HL

The crystal structure the mononuclear Ni(II) complex **1** contain two acetonitrile solvent molecules and two independent $[PhNHCSNNCHPhCl_2]$Ni(II) molecules with one denoting as **T1** containing Ni1 ion and phenyl ring C3–C8 and another denoting as **T2** containing Ni2 ion and phenyl ring C17–C22. In each $[PhNHCSNNCHPhCl_2]$Ni(II) molecule, the central Ni(II) ion coordinates with two deprotonated ligand of L^- with the Ni(II) ion being located at the inversion center. Each of the L^- provides one nitrogen atom from C=N group and one sulfur atom from C=S group to coordinate with the central Ni(II) ion to form five-membered chelate rings of NiN_2CS. Each of the central Ni(II) ion adopts a distorted square planar configuration with the bond angles of N3–Ni1–S1 [85.85(9) °] and N6–Ni2–S2 [84.79(9)°] being different from 90°. All of the bond lengths around Ni1 and Ni2 ions (Table 2) are consistent with those observed in the complexes of Ni(II) possessing square planar conformations [27, 28]. In **T1**, the dihedral angle between two phenyl rings of C3–C8 and C9–C14 is 77.54(1)°. In **T2**, the dihedral angle between two phenyl rings of C17–C22 and C23–C28 is 80.18(3)°. Although the **T1** and **T2** are two nearly identical molecules in the unit cell, there are some differences between them, which exist in the hydrogen bonds and intermolecular interactions. For instance, in **T1**, the hydrogen bond distance and angle of the N(1)–H(1)···N(8) are 3.0442 Å and 169.72°, respectively, while in **T2**, the hydrogen bond distance and angle of the N(4)–H(4A1)···N(7) are 3.0901 Å and 175.60°, respectively. The other differences in inter- and intra-moleuclar interactions between the **T1** and **T2** with solvent molecules can be found in Table 3.

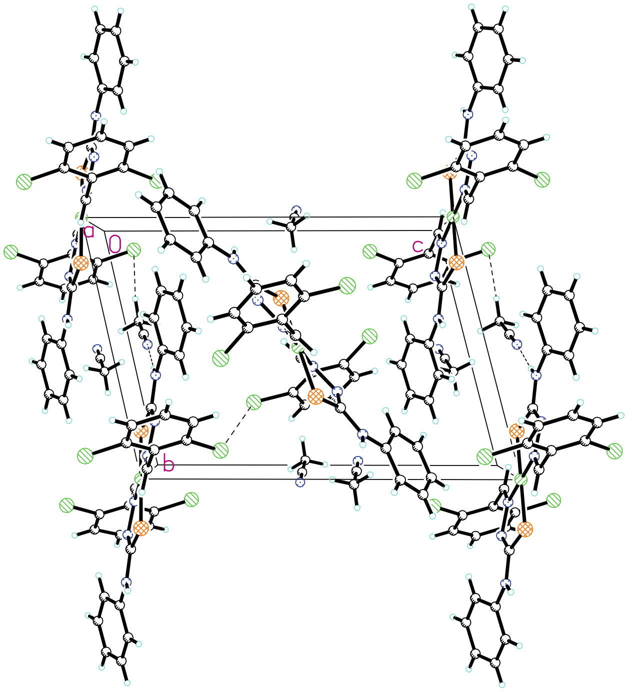


Fig. 6 Packing diagram of the unit cell along the *a*-axial for the complex **1**

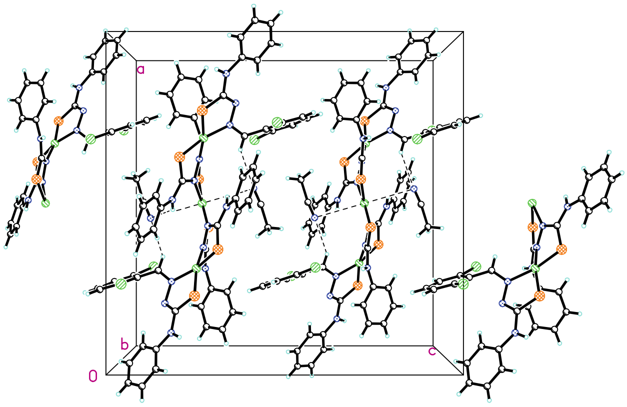


Fig. 7 Packing diagram of the unit cell along the *b*-axial for the complex **2**

In the trinuclear Ni(II) complex **2**, apart from the original ligand of HL, a new ligand of 1-(amino-*N*-phenylmethanethio)-4-phenylthiosemicarbazide (H_2Q) has been found.

Table 2 Some selected bond lengths and bond angles for the three compounds

HL	Bond length (Å)	C1–N1	1.346(6)	C2–N3	1.271(6)
		C1–N2	1.377(6)	N2–N3	1.356(5)
		C1–S1	1.646(6)	C3–N1	1.431(6)
	Bond angle (°)	N3–C2–C9	124.0(5)	N3–N2–C1	119.8(5)
		C4–C3–C8	120.2(6)	C2–N3–N2	116.8(5)
		C1–N1–C3	128.3(5)	N1–C1–N2	112.4(5)
1	Bond length (Å)	Ni1–N3	1.912(3)	Ni2–S2	2.1758(11)
		Ni1–S1	2.1635(11)	Ni2–N6	1.884(3)
	Bond angle (°)	N3–Ni1–S1	85.85(9)	N6–Ni2–S2	84.79(9)
		S1–Ni1–S1A	180.0	S2–Ni2–S2A	180.00(7)
2	Bond length (Å)	Ni1–N3	1.893(5)	Ni2–N5	1.908(4)
		Ni1–S1	2.1384(17)	Ni2–S3	2.1576(17)
		Ni1–S2	2.1478(18)	C16–S3	1.732(6)
	Bond angle (°)	N3–Ni1–S1	86.21(15)	N5–Ni2–S3	85.18(14)
		N3–Ni1–S2	163.80(15)	N5–Ni2–S3 A	174.62(14)
		N6–Ni1–S2	85.43(14)	S3A–Ni2–S3	91.53(9)
		N6–Ni1–S1	168.79(15)	N5A–Ni2–N5	98.4(3)
		N6–Ni1–N3	99.11(19)	S1–Ni1–S2	92.16(7)

Symmetry transformations used to generate equivalent atoms: in **1**, A: $-x, -y + 2, -z$; in **2**, A: $-x, y, -z + 1/2$

There are two deprotonated L^- ions and two deprotonated Q^{2-} in the complex **2** and, with two S atoms and two N atoms, each of Q^{2-} ion acts as a quadridentate bridge ligand to join with two Ni(II) ions. Among the three Ni (II) ions, Ni1 and Ni1A ions coordinate with one L^- ion and one Q^{2-} ion, respectively, and they both possess distorted square planar geometries. Ni2 ion coordinates with two Q^{2-} ions and also adopts a distorted square planar conformation, with bond angles around Ni2 ion being different from 90 to 180^o (Table 2). In the complex **2**, there is a C_2 symmetric axis passing through the Ni2 ion and all of the bond lengths of **2** are also corresponding with those in the Ni(II) complexes possessing square planar conformations [27, 28]. The dihedral angles between two phenyl rings in Q^{2-} ion is $29.83(2)^o$ and the dihedral angles between two phenyl rings in L^- ion is $50.29(3)^o$.

In the crystal lattices of the three compounds, there are some intramolecular and intermolecular hydrogen bonds (Table 3 and packing diagram of the unit cell), which help to stabilize the molecular structures. It is interesting that in complex **1**, although there are two nearly identical molecules independent molecules of **T1** and **T2** along with two independent solvent molecules in the unit cell, the kinds and numbers of the hydrogen bonds and supramolecular interactions between the **T1** and **T2** with the solvent molecules are different, which result in the diversiform molecular conformations.

Table 3 Hydrogen bonds and supramolecular interactions in the four compounds

	D–H···A	Symmetry code	D···A(Å)	D–H···A(°)
HL	N1–H1···N3		2.5641	112. 11
	N2–H2···S1	$\frac{1}{2} - x, \frac{1}{2} - y, -z$	3.3912	155.05
	C2–H2A···Cl1		2.9964	110.12
	C4–H4···S1		3.2559	114.37
1	N1–H1···N8	$-1 + x, y, z$	3.0442	169.72
	N4–H4A···N7	$1 - x, -y, 1 - z$	3.0901	175.60
	C2–H2···S1	$-x, 2 - y, -z$	3.0523	121.81
	C8–H8···N2		2.8827	116.22
	C16–H16···S2	$1 - x, 1 - y, 1 - z$	3.1038	115.89
	C18–H18···N5		2.8722	120.04
	C32–H32A···Cl1	$1 - x, 1 - y, -z$	3.5696	135.07
2	N1–H1···S3	$\frac{1}{2} - x, \frac{1}{2} + y, z$	3.6892	169.29
	N4–H4A···N8		3.2953	136.75
	N4–H4A···N5	$-x, y, \frac{1}{2} - z$	3.0040	121.36
	N7–H7A···S1	$\frac{1}{2} - x, -\frac{1}{2} + y, z$	3.3540	159.51
	C2–H2···N(6		3.1086	115.20
	C2–H2···N8	$-x, y, \frac{1}{2} - z$	3.3348	143.85
	C8–H8···N2		2.9015	123.37
	C13–H13···S2	$\frac{1}{2} - x, \frac{1}{2} - y, -\frac{1}{2} + z$	3.4619	136.89
	C22–H22···S2		3.1183	129.81

Supplementary Material

CCDC-758516 for HL, CCDC-758517 for **1** and CCDC-758518 for **2** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax + 44(0)1223-336033; email: deposit@ccdc.ccam.ac.uk].

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