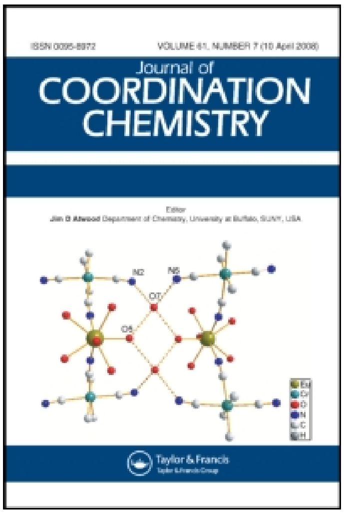

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Synthesis, crystal structure and magnetic properties of <i>N</i>-benzesulfonyl-L-glutamic acid-bridged binuclear nickel(II) complex

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Synthesis, crystal structure and magnetic properties of *N*-benzesulfonyl-L-glutamic acid-bridged binuclear nickel(II) complex

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N-benzesulfonyl-L-glutamic acid-bridged binuclear nickel(II) complex, $[Ni_2(Bs\text{-}Glu)_2(bipy)_2(H_2O)_2]\cdot 3H_2O$ (Bs-Glu = N-benzesulfonyl-L-glutamic acid dianion, bipy = 2,2′-bipyridine), has been synthesized and characterized structurally and magnetically. It crystallizes in the triclinic space group $P1$. The α-carboxyl group coordinates to Ni(II) in a monodentate mode, while the γ-carboxyl group coordinates in a chelating mode. In the temperature range 2–300 K, magnetic measurements show that the exchange interaction of the two Ni(II) ions is antiferromagnetic, and the values for J, g are $-2.47\,\mathrm{cm^{-1}}$ and 2.18, respectively.

Keywords: Nickel(II) complex; *N*-benzesulfonyl-L-glutamic acid; Crystal structure; Magnetic properties

1. Introduction

Continuing interest in the structures and properties of binuclear metal complexes derives from their serving as models of the active sites of certain metalloproteins and from the desire to understand the factors that affect physicochemical phenomena such as magnetic interactions between paramagnetic centers [1–2]. Thus coordination chemistry of binuclear metal complexes has received a great deal of attention. Investigation of magnetic properties of such complexes correlated with structure is important in the field of molecular magnetism [3–13]. Metal-organic frameworks built up by transition metal cations and dicarboxylate anions are also of interest in magnetic materials. Dicarboxylates are frequently used in the design of polynuclear complexes as efficient linkers, able to mediate magnetic interactions. In general terephthalate, oxalate, fumarate, etc. are used [14–16]; use of L-glutamic acid is not common. For *N*-benzesulfonyl-L-glutamic acid, substitution of the amino nitrogen by Ar–SO_2– decreases the affinity of the amino group for the metal, In such cases, coordination takes place only through the carboxylate, especially at low pH, opening the possibility to obtain structures with different carboxylate bridges [17–18]. Therefore, it would be

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interesting to study the magnetic properties complexes derived from *N*-benzesulfonyl-L-glutamic acid. In this work, we report a ternary complex of Ni(II) with *N*-benzesulfonyl-L-glutamic acid and 2,2′-bipyridine. The *N*-benzesulfonyl-L-glutamic acid ligand mediates weak antiferromagnetic exchange between Ni(II) ions in this complex.

2. Experimental

2.1. *General*

All reagents used in the syntheses were of analytical grade. C, H and N analyses were determined on a Perkin–Elmer 240 elemental analyzer. The IR spectrum was recorded in KBr discs on a Shimadzu IR-408 IR spectrophotometer in the 4000–600 cm^{-1} range.

Variable-temperature magnetic susceptibilities were measured using a MPMS-7 SQUID magnetometer. Diamagnetic corrections were made with Pascal's constants for all constituent atoms.

2.2. *Preparation of* $[Ni_2(Bs\text{-}Glu)_2(bipy)_2\ (H_2O)_2]\cdot 3H_2O$

A mixture of $Ni(CH_3COO)_2\cdot 4H_2O$ (1 mmol, 0.249 g) and Bs-GluH_2 (2 mmol, 0.572 g) was stirred into 15 mL aqueous solution at room temperature. Then the pH was adjusted to 5 with 1 mol L^{-1} NaOH and 5 mL ethanol solution of 2,2′-bipyridine (1 mmol, 0.158 g) was added. The reaction mixture was then heated on a water bath for 8 h at 70°C, and filtered. Blue crystals were separated from the mother liquor by slow evaporation at room temperature for 4 weeks. (Found: C, 46.19; H, 4.51; N, 7.83. $C_{42}H_{46}N_6Ni_2O_{17}S_2$ Anal. Calcd: C, 46.26; H, 4.44; N, 7.71%). Yield: 0.61 g (58%).

3. Crystal structure determination

Single-crystal data were collected using ω–2θ scans at 295(2) K on a Bruker Smart Apex II diffractometer equipped with graphite-monochromated Mo-Kα radiation. The structure was solved using direct methods and successive Fourier difference synthesis (SHELXS-97) [19], and refined using full-matrix least-squares on F^2 with anisotropic thermal parameters for all non-hydrogen atoms (SHELXL-97) [20]. Hydrogen atoms were assigned with common isotropic displacement factors and included in the final refinement by use of geometrical restraints. The final agreement factor values are $R_1 = 0.0268$, $wR_2 = 0.0524$, $w = 1/[\sigma^2(F_0)^2 + (0.0155P)^2]$ where $P = (F_0^2 + 2F_c^2)/3$. A summary of the crystallographic data is given in table 1. Selected bond distances and angles are given in table 2.

4. Results and discussion

4.1. *Crystal structure of* $[Ni_2(Bs\text{-}Glu)_2(bipy)_2\ (H_2O)_2]\cdot 3H_2O$

Structure analysis shows that the formula of the complex is $[Ni_2(Bs\text{-}Glu)_2(bipy)_2(H_2O)_2]\cdot 3H_2O$, representing the first example of Ni(II) and *N*-protected-L-glutamic acid

Table 1. Crystallographic data and processing parameters for the title complex.

Empirical formula	$C_{42}H_{46}N_6Ni_2O_{17}S_2$
Formula weight	1088.39
Temperature (K)	295(2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	$P1$
Unit cell dimensions (Å, °)	
a	7.8991(5)
b	12.2103(7)
c	13.8723(8)
α	65.625(10)
β	79.277(10)
γ	76.400(10)
Volume (Å^3)	1178.55(12)
Z	1
Calculated density (Kg m^{-3})	1.534
Absorption coefficient (mm^{-1})	0.966
$F(000)$	564
Crystal size (mm^3)	0.30 × 0.19 × 0.12
θ Range for data collection (°)	2.67–27.50
Reflections collected	10443
Independent reflection	9174 [$R_{\text{int}} = 0.0108$]
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	9174/15/654
Goodness-of-fit on F^2	0.947
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0268$, $wR_2 = 0.0528$
R indices (all data)	$R_1 = 0.0318$, $wR_2 = 0.0543$
Largest diff. peak and hole (e Å^{-3})	0.262 and −0.231

Table 2. Selected bond lengths (Å) and angles (°) for the title complex.

Bond lengths			
Ni(1)–O(3)	2.0298(19)	Ni(2)–O(1)	2.121(2)
Ni(1)–N(4)	2.051(2)	Ni(2)–O(2)	2.126(2)
Ni(1)–N(3)	2.053(2)	Ni(2)–N(1)	2.034(3)
Ni(1)–O(14)	2.087(2)	Ni(2)–O(9)	2.0428(19)
Ni(1)–O(8)	2.103(2)	Ni(2)–N(2)	2.077(3)
Ni(1)–O(7)	2.1636(19)	Ni(2)–O(13)	2.083(2)
Bond angles			
O(3)–Ni(1)–N(4)	93.59(9)	O(8)–Ni(1)–O(7)	61.77(8)
O(3)–Ni(1)–N(3)	172.88(10)	N(1)–Ni(2)–O(9)	92.93(9)
N(4)–Ni(1)–N(3)	79.55(10)	N(1)–Ni(2)–N(2)	79.53(10)
O(3)–Ni(1)–O(14)	90.93(8)	O(9)–Ni(2)–N(2)	171.67(10)
N(4)–Ni(1)–O(14)	98.19(9)	N(1)–Ni(2)–O(13)	97.25(9)
N(3)–Ni(1)–O(14)	91.93(9)	O(9)–Ni(2)–O(13)	91.53(8)
O(3)–Ni(1)–O(8)	86.80(8)	N(2)–Ni(2)–O(13)	92.92(9)
N(4)–Ni(1)–O(8)	101.13(9)	N(1)–Ni(2)–O(1)	161.79(9)
N(3)–Ni(1)–O(8)	92.63(9)	O(9)–Ni(2)–O(1)	91.85(9)
O(14)–Ni(1)–O(8)	160.65(8)	N(2)–Ni(2)–O(1)	94.29(9)
O(3)–Ni(1)–O(7)	91.05(8)	O(13)–Ni(2)–O(1)	100.18(9)
N(4)–Ni(1)–O(7)	162.02(9)	N(1)–Ni(2)–O(2)	101.01(9)
N(3)–Ni(1)–O(7)	94.94(9)	O(9)–Ni(2)–O(2)	85.92(8)
O(14)–Ni(1)–O(7)	99.09(8)	N(2)–Ni(2)–O(2)	92.01(9)
O(1)–Ni(2)–O(2)	61.82(8)	O(13)–Ni(2)–O(2)	161.66(8)

or aspartic acid. The two Ni(II) ions in the complex are six-coordinate with a distorted octahedral geometry, as shown in figure 1. The six atoms coordinated to Ni(II) come from two nitrogen atoms of bipyridine and two oxygen atoms of γ-COO$^-$ group of Bs-Glu ligand, one oxygen atom of α-COO$^-$ group and one water molecule. The Ni–N and Ni–O bond distances are in the range 2.032–2.077 Å and 2.028–2.093 Å, respectively. The 2,2′-bipyridine molecule is planar with an angle between the pyridine planes of 0.5°. The Ni(1) and Ni(2) ions are linked to form a binuclear structure where Bs-Glu is the bridging ligand. The two carboxylate groups of Bs-glu display different coordination modes. The α-COO$^-$ is monodentate to Ni(II) and the γ-COO$^-$ group coordinates bidentate with another Ni(II). Thus, the bridging action of Bs-Glu is accomplished via its monodentate coordination and bidentate chelate action with another metal ion. Such a bridging mode has not been reported in transition metal complexes of glutamic acid. The intradimer Ni $\cdots$ Ni distance is 7.057 Å. Only one crystal structure of a nickel complex with *N*-benzesulfonyl-amino acids has been reported, i.e., $[Ni(bipy)_2(tsglyNO)] \cdot 2H_2O$ (tsglyNO = *N*-p-tolylsulfonylglycinato-N,O) [21]. The complex is mononuclear and the Ni(II) is six-coordinate with four nitrogen atoms of the two bipy, the deprotonated sulfonamide nitrogen and one carboxylic oxygen of the tsgly dianion.

Formation of binuclear units created 16-membered rings, which are linked by intramolecular hydrogen-bonding (O(13)–H(1W) $\cdots$ O(7) and O(14)–H(4W) $\cdots$ O(1), with bond distances of 2.775(3) and 2.809(3) Å and bond angles of 166(2) and160(2)°, respectively) to result in smaller six-membered rings. The alternating 16-membered and six-membered rings link to form a 1-D chain along the *c*-axis, as shown in figure 2. The shortest intermolecular distance of Ni $\cdots$ Ni is 5.486 Å. Also, there exist complicated hydrogen bonds among the crystalline water molecules, the coordinating water molecules, the carboxylato O and sulfonamide O atoms, which contribute to the stability of the complex. There also exist orderly aromatic ring stacking interactions between 2,2′-bipyridines; the centroid distance between the 2,2′-bipyridine molecules

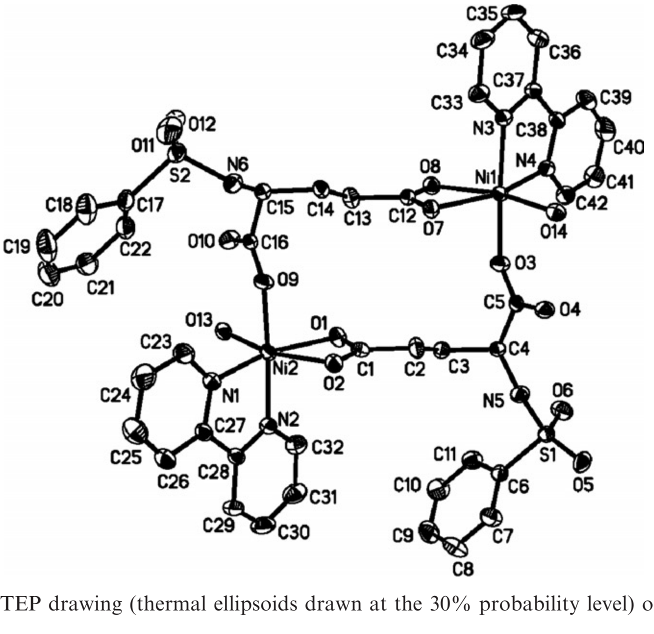

Figure 1. ORTEP drawing (thermal ellipsoids drawn at the 30% probability level) of the complex.

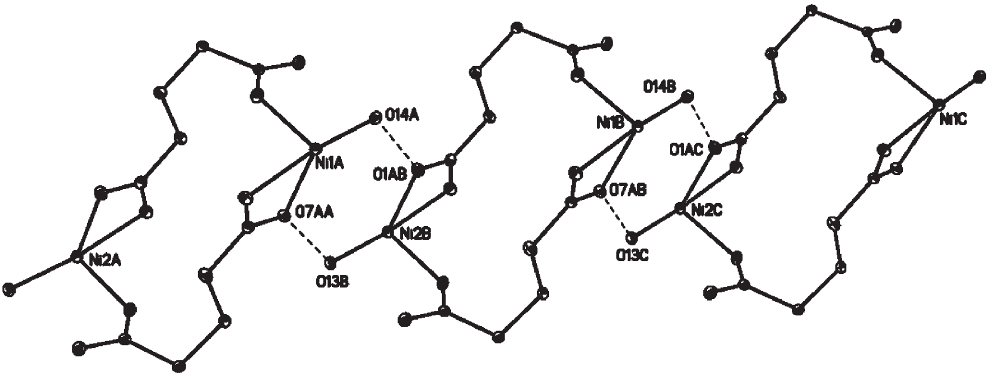

Figure 2. One-dimensional structure of the complex (benzene ring, bipyridine ring and sulfonamide groups are omitted for clarity).

belonging to adjacent chains is 3.698 Å with a dihedral angle of 5.8°. Thus the 1-D chains are linked via π–π stacking interactions to form a two-dimensional supramolecular network.

4.2. *Spectroscopic studies*

The IR spectra of the complex exhibit several characteristic strong bands: the one observed at 3421 $\mathrm{cm^{-1}}$ is attributed to N–H stretching, those at 1315 and 1159 $\mathrm{cm^{-1}}$ are due to $\upsilon_{as}(\text{–SO}_2\text{–})$ and $\upsilon_s(\text{–SO}_2\text{–})$ in complex. Of three strong characteristic peaks at 1599, 1443 and 1408 $\mathrm{cm^{-1}}$ for the complex, the one at 1599 $\mathrm{cm^{-1}}$ is attributed to $\nu_{as}(\mathrm{COO^-})$ of all the carboxylate groups, while the peaks at 1443 and 1408 $\mathrm{cm^{-1}}$ may be assigned to $\nu_s(\mathrm{COO^-})$ of the bidentate carboxylate group and the monodentate carboxylate group, respectively, since it is known that the difference $\Delta = \nu_{as} - \nu_s$ is smaller in the former (156 $\mathrm{cm^{-1}}$) than in the latter (191 $\mathrm{cm^{-1}}$) [22, 23].

4.3. *Magnetic properties*

The magnetic susceptibilities, χ_M, of the title complex were measured in the 2–300 K region, and figure 3 shows the plots of $\chi_M T$ and χ_M *versus* T. The $\chi_M T$ value at room temperature is 2.36 $\mathrm{cm^3\,K\,mol^{-1}}$, which falls in the normal range of two non-coupled Ni(II) ions [11, 12]. As the temperature is lowered, the $\chi_M T$ value decreases, which indicates the presence of antiferromagnetic interactions in the complex.

In order to quantitatively evaluate the magnetic interactions in the system, for similar binuclear Ni(II) complexes, the following equation (1) is derived from the Hamiltonian $\hat{H} = -JS_1S_2$ where J is the intra-dimer exchange constant through two Bs-Glu ligands [11].

$$\chi_M = \frac{Ng^2\beta^2}{KT}\frac{A}{B} \tag{1}$$

$$A = 2\exp[J/KT] + 10\exp[3J/KT]$$

$$B = 1 + 3\exp[J/KT] + 5\exp[3J/KT]$$

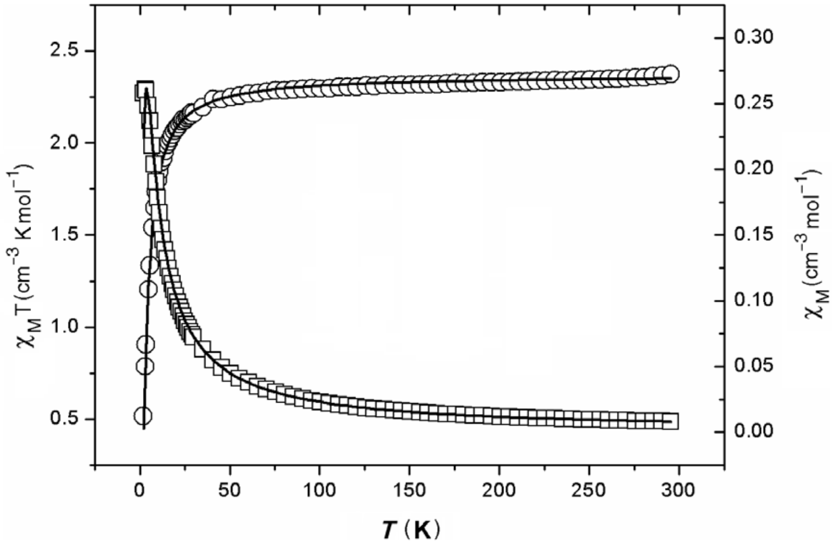

Figure 3. Temperature dependence of $\chi_M T$ and $_M$ for the title complex. The solid line represent the theoretical values based on equation (1) described in the text.

The magnetic interaction (zJ') of interbinuclear through hydrogen bonding interaction can be treated as a molecular field approximation [24].

The total magnetic susceptibility is

$$\chi_T = \frac{\chi_M}{1 - (2zJ'/N\beta^2 g^2)\chi_M} \tag{2}$$

The least-squares analysis of magnetic susceptibilities data led to $J = -2.47\,\mathrm{cm}^{-1}$, $g = 2.18$, $zJ' = -0.176\,\mathrm{cm}^{-1}$ and $R = 1.62 \times 10^{-3}$ (the agreement factor defined as $R = \sum[(\chi_M)_{obs} - (\chi_M)_{calc}]^2/\sum[(\chi_M)_{obs}]^2$. The J value indicates a weakly antiferromagnetic between the two Ni(II) ions bridged by Bs-Glu. The smaller negative zJ' value can be attributed to a very weak antiferromagnetic interaction through hydrogen bonding.

The carboxylate group is one of the most widely used bridging ligands for designing polynuclear complexes with interesting magnetic properties. For Cu(II) and Mn(II) systems, the number of carboxylato bridged complexes is high while the number for nickel(II) ion is low. The J values of the title complex as well as those of some other reported Ni(II) discrete complexes or polymers with carboxylato bridges are compiled in table 3. For complexes involving carboxylato bridges, we have restricted the data to Ni(II) complexes involving only carboxylato bridges. The exchange coupling through carboxylato bridges is usually weak and antiferromagenic except for the last examples. The weak coupling observed is not surprising in light of the large intramolecular metal–metal separation. The magnitude of the magnetic exchange for the above complexes mostly depends on the intramolecular distance between Ni $\cdots$ Ni besides the bridging mode and the dihedral angle. The greater the intramolecular metal–metal separation, the weaker the magnetic coupling [6, 25]. Further research

Table 3. Exchange couplings for some Ni(II) carboxylato-bridged compounds, corresponding g values and Ni–Ni distances (Å).

Compound	Bridged ligand	Type	Ni–Ni distance	g	$J\,\mathrm{cm}^{-1}$	Ref.
$[\mathrm{Ni_2(pmdtn)_2(tp)(N_3)_2]4H_2O}$	tp	Binuclear	10.800	2.18	−0.02	12
$[\mathrm{Ni_2(pmdtn)_2(tp)(H_2O)_2](ClO_4)_2}$	tp	Binuclear	10.713	2.12	−0.18	12
$[\mathrm{Ni_2(bpy)_4(tp)]\ (ClO_4)_2}$	tp	Binuclear	9.449	2.11	−0.6	6
$[\mathrm{Ni_2(phen)_2(H_2O)_2(betc)]}_n$	betc	Chain	11.014	2.21	−0.85	25
$[\mathrm{(NiL)_3(BTC)_2]8H_2O}$	BTC	Chain	11.347	2.13	−1.13	26
$[\mathrm{Ni}(\mathbf{L_2})\mathrm{(tp)]2H_2O}$	tp	Chain	11.526	2.19	−1.18	27
$[\mathrm{Ni(L)(BDC)]2H_2O}$	BDC	Chain	11.528	2.26	−1.37	26
$[\mathrm{Ni(L)(PDC)]H_2O}$	PDC	Chain	11.465	2.16	−1.47	26
$[\mathrm{Ni_2(Bs\text{-}Glu)_2\ (H_2O)_2]3H_2O}$	Bs-Glu	Binuclear	7.057	2.18	−2.47	This work
$[\mathrm{(MeOH)}(\mathbf{L_3})\mathrm{Ni_2(CCl_3COO)]}$	$\mathrm{CCl_3COO^-}$	Binuclear	3.058	2.24	−11.98	11
$[\mathrm{(maleate)_2Ni_3(bpe)_4(H_2O)_4]\ (NO_3)\cdot H_2O}$	Maleate	Chain	8.529	2.07	1.74	28

pmdtn = *N,N,N′,N′,N″*-pentamethyldiethyltriamine; tp = terephthalate; bpy = 2,2′-bipyridine; L = 3,14-dimethyl-2,6,13,17-tetraazatricyclo[14,4,$0^{1.18}$,$0^{7.12}$] docosane; BTC = 1,3,5-benzene tricarboxylate; BDC = 1,4-benzenedicarboxylate; PDC = 2,5-pyridinedicarboxylate; $\mathbf{L_2}$ = 1,3,10,12,15,18-hexaazatetracyclo[16,2,1,$1^{12.15}$,$0^{4.9}$]docosane; betc = 1,2,4,5-benzen etetracarboxylate; bpe = 1,2-*bis*(4-pyridyl)ethane.; $\mathbf{L_3} = \{(\mathrm{O^-})(\mathrm{C_6H_4CH{=}NCH_2CH_2NMe_3})\}$].

will prepare novel Ni(II) polymeric frameworks in this subfield and explore their magnetic properties.

Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 283034. Copies of this information can be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336-033; Email: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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