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NOTE

Synthesis and Magnetic Property of a Nickel(II) Complex: $[Ni(C_{14}H_{14}N_8O_2)]_4(ClO_4)_8(H_2O)_8(CH_3OH)_{0.5}$

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A new self-assembled Schiff base of nickel(II) complex with the molecular formula $[NiL]_4(ClO_4)_8(H_2O)_8(CH_3OH)_{0.5}$ {L = (6Z,15Z)-N1',N2'-*bis*(amino(pyridin-2-yl)methylene)oxalohydrazide} has been synthesized and characterized by IR spectra, elemental analysis and TG measurement. The magnetic properties investigation shows that there is weak antiferromagnetic interactions between the Ni(II) ions of the complex.

Key Words: Schiff base, Nickel(II) complex, Magnetic property.

While the development of the synthesis of Schiff-base transition metal azamacrocyclic complexes[1-3], the researches in molecular materials with magnetic properties and molecular catalysis have developed rapidly[4-6]. The design and synthesis of molecule-based magnetic materials are one of the major subjects of macrocyclic chemistry[7,8].

All the reagents were of AR grade and used without further purification. IR spectra were recorded on a Nexus-870 spectrophotometer. Elemental analysis were performed on a Elementar Vario ELZ(III) analyzer. TG-DSC analyses were obtained with a STA449F3 thermoanalyzer. Variable temperature magnetic data (1.8-300 K) were collected with Quantum Design MPMS XL5 Squid magnetometer.

Synthesis of the ligand: The ligand (6Z,15Z)-N1',N2'-bis(amino(pyridin-2-yl)methylene)oxalohydrazide (L) was prepared according to the literature[9].

Synthesis of the complex: The 0.33 g of the ligand was added to a mixture of 1.46 g $Ni(ClO_4)_2{\cdot}6H_2O$, 10 mL H_2O, 5 mL methyl cyanide and 5 mL methanol. The mixture was stirred for 2 h and filtered, the clear red coloured solution was obtained. After standing at room temperature for several days, the crimson crystals were obtained, then dried to constant weight in a vacuum dryer at room temperature. IR spectrum (KBr, cm^{-1}): ν(C=N) 1664, 1580; ν(N-H) 3350; ν(H_2O) 3167; ν(ClO_4^-) 1090, 625. Elemental analysis: calcd. (%) for $[Ni(C_{14}H_{14}N_8O_2)]_4(ClO_4)_8(H_2O)_8$

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$(CH_3OH)_{0.5}$: C, 27.19; H, 2.99; N, 17.96; Found (%) C, 27.10; H, 3.01; N, 17.83. Thermogravimetry was done at 25 to 550 ºC (5 ºC, min^{-1}) in air. TG-DSC data shows that for the complex, the number of the hydrated water molecules agree with that of elemental analysis. The magnetic susceptibility data on the complex were collected over the temperature range 1.8-300 K.

The syntheses of the title complex was achieved by the reaction of the ligand L with $Ni(ClO_4)_2 \cdot 6H_2O$ and characterized by elemental analyses, IR and TG measurement. The result is consistent with the composition of $[NiL]_4(ClO_4)_8(H_2O)_8(CH_3OH)_{0.5}$.

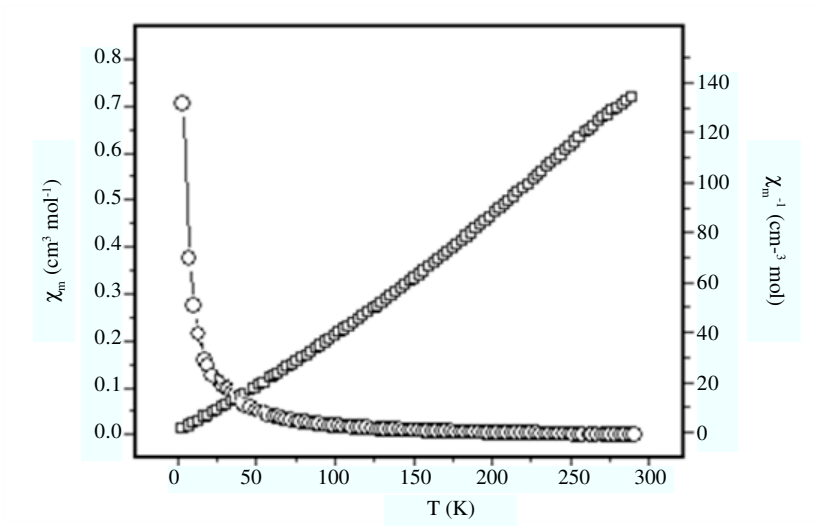

Fig. 1. χ_m-T and $1/\chi_m$-T curves of $[NiL]_4(ClO_4)_8(H_2O)_8(CH_3OH)_{0.5}$

Magnetic properties: Fig. 1 shows the plots of χ_m *versus* T and χ_m^{-1} *versus* T for the complex. With the temperature was decreased, the χ_m^{-1} value decrease gradually through out the temperature range 300 K to 1.8 K. From 30 to 111.7 K, the magnetic data can be fitted well (r = 0.99992) to the Curie-Weiss law with C = 3.445(4) cm^3 mol^{-1} K and θ = .1.74(9) K, The small negative value of Weiss constant indicates that there is a weak antiferromagnetic exchange coupling between the Ni(II) ions of the complex[10].

In summary, a novel nickel(II) complex was obtained at room temperature. It has been characterized by IR spectra, elemental analysis and TG measurement. The magnetic properties reveal that there is a weak antiferromagnetic interactions between the Ni(II) ion of the present complex.

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