Synthesis and Characterization of a Novel Benziloxime Ligand and Its Iron(III) and Nickel(II) Complexes

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The synthesis of a novel benziloxime ligand, 4,4'-dibromobenzilmonoxime (DBBOH) (1) and its complexes [Fe(DBBO)₃] (2) and Na[Ni(DBBO)₃] (3) are described. The ligand (DBBOH) was prepared in MeOH from the reaction of 4,4'-dibromobenzil with hydroxylamine hydrochloride in the presence of sodium acetate.

Reaction of sodium salt of 4,4'-dibromobenzilmonoxime (DBBONa) in MeOH with $Fe(NO_3)_3.9H_2O$ and $Ni(NO_3)_2.6H_2O$ yielded [$Fe(DBBO)_3$] (**2**) as yellow and $Na[Ni(DBBO)_3]$ (**3**) as brown precipitates respectively.

The ligand, 4,4'-dibromobenzilmonoxime (DBBOH) (1) and its complexes [Fe(DBBO)₃] (2) and Na[Ni(DBBO)₃] (3) were characterized by elemental analysis, ¹H-NMR and ¹³C-NMR, IR, and electronic spectral studies.

The analysis of IR, ¹H- and ¹³C-NMR spectral data of Fe(III) and Ni(II) complexes suggest that 4,4'dibromobenzilmonoximate is bonded to iron(III) and nickel(II) ions through the oxygen atom of the carbonyl, and the nitrogen atom of the imine groups. The electronic spectral data of the complexes in CHCl₃ are in good agreement with the octahedral coordination of Fe(III) and Ni(II) ions. The ligand field parameters for these complexes, i.e. splitting energy and Racah parameter were calculated to be 21560 cm⁻¹ and 707 cm⁻¹ for [Fe(DBBO)₃] (**2**), 13440 cm⁻¹ and 610 cm⁻¹ for Na[Ni(DBBO)₃] (**3**) respectively.

Keywords: 4,4'-Dibromobenzilmonoxime; Octahedral complexes; Iron(III) ion; Nickel(II) ion; Ligand field parameters; Monooximes; IR; Electronic spectra; ¹H- and ¹³C-NMR studies.

1. INTRODUCTION

Oximes play an important role in the development of transition metal coordination chemistry due to their versatile bonding modes.¹ Benzilmonoximes are known as chelating agents and are frequently used in extractive and analytical chemistry. For example, the concentration of copper in a solution has been determined its benzilmonoxime complex by atomic absorption spectroscopy.² It is a well known extracting agent for molybdenum, tungsten, vanadium,³ and also for the determination of cobalt at microgram amount.⁴ A number of benzilmonoximates which have been reported show interesting stereochemistry.⁵ Benzilmonoximes are of special interest owing to their biological activities, and semi-conducting properties.⁶ In view of this, and our continuing interest on the synthesis of new benzilmonoximes,^{7,8} we wish to report the synthesis and characterization of 4,4'-dibromobenzilmonoxime and its complexes with Fe(III) and Ni(II) salts. The properties of the complexes were investigated by spectroscopic methods.

2. RESULTS AND DISCUSSION

The reaction of sodium 4,4'-dibromobenzilmonoximate (DBBONa) with Fe(NO₃)₃.9H₂O and Ni(NO₃)₂. 6H₂O in methanol gave [Fe(DBBO)₃] (**2**) as yellow and Na[Ni(DBBO)₃] (**3**) as brown complexes, respectively (Scheme I).

The ligand, 4,4'-dibromobenzilmonoxime (DBBOH) (1) and its complexes [Fe(DBBO)₃] (2) and Na[Ni(DBBO)₃] (3) were characterized on the basis of elemental analysis, and ¹H-NMR, ¹³C-NMR, IR, and electronic spectra data. The molar conductance of complexes 2 and 3 were 25 and 123 Ω^{-1} .cm².mol⁻¹, respectively, indicating that the iron(III) complex 2 to be non-electrolyte, but the nickel(II) complex 3 showed a 1:1 electrolyte behavior.⁹

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2.1. IR spectra

The 4,4'-dibromobenzilmonoxime ligand, showed characteristic IR bands at 3455, 1645, 1220 cm⁻¹ due to the OH, C=N, and N–O stretching vibrations of the oxime group (C=N-OH), and a sharp band at 1715 cm⁻¹ due to v(C=O) of the carbonyl group (Table 1). A strong and broad band is observed at 3455 cm⁻¹ for the free ligand due to v(N–OH) which is absent in both complexes, suggesting the deprotonation of the hydroxyl group of the oxime in the process of formation of the complexes.¹⁰

The band at 1715 cm⁻¹, which was assigned to v(C=O) of ligand was shifted to a lower energy at 1685 cm⁻¹ in iron(III) and 1690 cm⁻¹ in nickel(II) complexes, indicating the participation of the carbonyl group of 4,4'-dibromobenziloxime in coordination.¹¹ Also the band at 1645 cm⁻¹ due to v(C=N) of the oxime group in ligand was shifted to lower frequencies at 1625 cm⁻¹ and 1618 cm⁻¹ in its iron(III) and nickel(II) complexes, suggests the coordination of azomethine group (C=N) with the metal ions.¹²

These facts are further supported by the appearance of new bands in the regions 492, 490, 448, and 452 cm^{-1} in

the iron(III) and Ni(II) complexes, which were assigned to the v(Fe-O), v(Ni-O) and v(Fe-N), v(Ni-N) stretching vibrations respectively.¹³

The absorption band due to C-Br (710 cm⁻¹) of the ligand does not show any shift in the spectra of the complexes (705, 707 cm⁻¹) which means it dos not take parting coordination. The band at 1220 cm⁻¹ due to v(N-O) of the ligand appeared at 1225 and 1228 cm⁻¹ in the complexes. Since no considerable change was observed for v(N-O) of the free ligand and the complexes, indicates no participation of oxygen atom of the N-O group in the coordination.

2.2. ¹H- and ¹³C-NMR spectra

The ¹H-NMR spectra of 4,4'-dibromobenzilmonoxime and its iron(III) and nickel(II) complexes show a multiplet in the aromatic region 6.7-7.80 ppm due to the phenyl groups. The singlet peak due to the N–OH proton at 10.20 ppm in 4,4'-dibromobenzilmonoxime is absent in its iron(III) and nickel(II) complexes, suggesting the deprotonation of the hydroxyl group of the oxime in the ligand.¹⁴

The ¹³C-NMR spectra for 4,4'-dibromobenzilmono-

Table 1. Important IR bands (cm⁻¹) and their assignments for 4,4'-dibromobenzilmonoxime and its Fe(III) and Ni(II) complexes

Vibration modes	v(O-H)	v(C=O)	v(C=N)	v(N-O)	v(C-Br)	v(M-O)	v(M-N)
4,4'-dibromobenzil-monoxime (1)	3455	1715	1645	1220	710		
$[Fe(DBBO)_3]$ (2)		1685	1625	1222	705	492	448
$Na[Ni(DBBO)_3]$ (3)		1690	1618	1228	707	490	452

xime show peaks at the regions 129, 131, 135, 139, 168, 198.5, and 201 ppm (Table 2). The signals at 201 and 198.5 ppm were assigned to the carbon of the carbonyl group and the carbon of the oxime group in 4,4'-dibromobenzilmonoxime ligand, respectively.¹⁵ These bands shifted upfield to 191, 193 ppm and 183.5, 185 ppm, in its iron(III) and nickel(II) complexes, respectively, due to shielding effects in its complexes, which suggest coordination of the oxygen atom of the carbonyl group and the nitrogen atom of the oxime group to metal ions.¹⁶

The resonance at 168 ppm belongs to the C4, C4' carbons carrying bromine atoms. The other two resonances which appear at 131 and 129 ppm are assigned to C2, C2', C6, C6' and 3, 3', 5, 5' carbon atoms of the phenyl rings. The resonances at 139 and 135 ppm were assigned, respectively, to the C1 carbon atom of the phenyl ring bonded to the carbonyl group and to the C1' carbon atom of the phenyl ring bonded to the oxime group.¹⁷ These bands are also present in the spectra of the iron(III) and nickel(II) complexes at almost the same positions, indicating no participation of them in coordination.

2.3. Electronic spectra

The electronic spectra of 4,4'-dibromobenzilmonoxime and its iron(III) and nickel(II) complexes in CHCl₃ Soleimani

exhibit absorption bands at 260-264 and 365-366 nm with high molar extinction coefficient (Table 3) that can be assigned to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ intra-ligand charge transfer, respectively.¹¹ These absorption bands have high intensity since they obey the Lapport selection rule. In addition to the above bands, same other bands were also observed at 384, 451, and 542 nm for its iron(III) and at 535 and 744 nm for nickel(II) complexes.

Since there are no spin-allowed d \rightarrow d transitions for iron(III) with high spin d⁵ configuration, therefore, the bands at the regions 451 and 542 nm with molar extinction coefficients lower than 100 M⁻¹.cm⁻¹ can be assigned to the ligand field transitions for iron(III) with low spin (t_{2g})⁵ configuration.¹⁸

The low spin configuration of the Fe(III) complex is further confirmed by magnetic susceptibility measurement. At ambient temperature, the Fe(III) complex exhibit effective magnetic moment of 1.98 B.M. This magnetic moment value confirm that Fe(III) complex remain low spin (S = 1/2) with $(t_{2g})^5$ configuration. Mononuclear Fe(III) complexes [CpFe(dppe)-SR]PF₆ and [Fe(pyAS)₂]Bph₄ with a low spin d⁵ configuration have been reported to exhibit magnetic moment at 1.93 and 1.97 B.M. respectively.^{19,20}

According to the Tanabe-Sugano diagram for low spin d^5 configuration, the bands at the regions 542 nm

Table 2. The observed chemical shift values (ppm) for the ¹³C-NMR spectra of 4,4'-dibromobenzilmonoxime, and its Fe(III) and Ni(II) complexes

Carbon type	C3, C3' C5, C5'	C2, C2' C6, C6'	C1′	C1	C4, C4′	C=N-OH	С=О
4,4'-dibromobenzil-monoxime (1)	129	131	135	139	168	198.5	201
$[Fe(DBBO)_3]$ (2)	129	131	135.5	138	169	183.5	191
$Na[Ni(DBBO)_3]$ (3)	129	132	134.5	138.5	168	185	193

Table 3. The electronic absorption data (nm) 4,4'-dibromobenzilmonoxime, and its Fe(III) and Ni(II) complexes recorded in CHCl₃. Log ε of absorption bands are in parentheses

Transition type	Intra ligand charge transfer					
	$\pi \to \pi^*$	$n \to \pi^*$	Ligand to metal	Ligand field tra	Splitting energy/cm ⁻¹	
4,4'-dibromobenzil- monoxime (1)	260 (4.2)	365 (2.8)	enarge transfer			
[Fe(DBBO) ₃] (2)	262 (3.8)	365 (2.4)	$p\pi(O) \to eg^{*}(Fe)$ 384 (3.7)	$^{2}T_{2g} \rightarrow (^{2}A_{2g} \text{ or } ^{2}T_{1g})$ 542 (1.85)	${}^{2}\mathrm{T}_{2g} \rightarrow {}^{2}\mathrm{E}_{g}$ $451 (1.8)$	21560
Na[Ni(DBBO) ₃] (3)	264 (3.9)	366 (2.5)		${}^{3}A_{2g} \rightarrow {}^{3}T_{2g}$ 744 (1.95)	$^{3}A_{2g} \rightarrow ^{3}T_{1g}$ 535 (1.84)	13440

(18450 cm⁻¹) and 451 nm (22170 cm⁻¹) were assigned to the ${}^{2}T_{2g} \rightarrow ({}^{2}A_{2g} \text{ or } {}^{2}T_{1g})$ and ${}^{2}T_{2g} \rightarrow {}^{2}E_{g}$ transitions, respectively.²¹

When working with electronic spectra, it is often useful to determine the ratio of the energy of the second transition to the first one. In the iron(III) complex, $\frac{v_2}{v_1} = \frac{22170 \text{ cm}^{-1}}{18450 \text{ cm}^{-1}} = 1.2$, and this ratio corresponds to $\frac{\Delta_o}{B}$ being equal to 30.5 on the Tanabe-Sugano diagram. By using this value in the calculation of the Racah parameter, splitting energy, unobserved third transition, and nephelauxetic parameter for the iron(III) complex, the following results were obtained $B = 707 \text{ cm}^{-1}$, $\Delta_o = 21560 \text{ cm}^{-1}$, $v_3 = 30400 \text{ cm}^{-1}$, and $\beta = 0.642$.

The bands at the regions 744 nm (13440 cm⁻¹) and 535 nm (18690 cm⁻¹) for nickel(II) complex were assigned to the first and second ligand field transitions ${}^{3}A_{2g} \rightarrow {}^{3}T_{2g}$ and ${}^{3}A_{2g} \rightarrow {}^{3}T_{1g}$, respectively. The ratio of the second to first transition $\frac{v_2}{v_1} = \frac{18690 \text{ cm}^{-1}}{13440 \text{ cm}^{-1}} = 1.4$, which corresponds

to $\frac{\Delta_o}{B}$ being equal to 22 on the Tanabe-Sugano diagram. By

using this value in the calculation of the Racah parameter, splitting energy, unobserved third transition, and nephelauxetic parameter for the nickel(II) complex, the following results were obtained $B = 610 \text{ cm}^{-1}$, $\Delta_O = 13440 \text{ cm}^{-1}$, $v_3 = 29280 \text{ cm}^{-1}$, and $\beta = 0.608$.

The absorption bond at 384 nm (26040 cm⁻¹) with log ε equal to 3.3 for iron(III) complex could be assigned to the charge transfer from the filled π -orbital of 4,4'-dibromobenzilmonoximate to the empty eg* orbital of iron(III) in the complex,²² which has a high intensity (Lapport selection rule).

The molar extinction coefficient of $d\rightarrow d$ transitions for trigonal prismatic complexes is more than 250 M⁻¹cm⁻¹ due to the lack of a center of symmetry. However, the molar extinction coefficient of $d\rightarrow d$ transitions for the iron(III) complex was lower than 100 M⁻¹ cm⁻¹, indicating that [Fe(DBBO)₃] and Na[Ni(DBBO)₃] complexes have octahedral geometry.

3. EXPERIMENTAL SECTION

All the chemicals used were of analytical grade. All synthetic works were carried out open to atmosphere. The

solvents used were purified, and dried according to the standard procedures.²³ The reaction progress, and the purity of the products were checked using silica gel TLC plates (CHCl₃:CH₃OH, 5:1).

3.1. Physical and spectral measurements

The elemental analysis was performed on a Heraeus Carlo Erba1108 elemental analyzer. The ¹H- and ¹³C-NMR spectra were recorded on a Brucker AV300 NMR spectrometer with the TMS as internal reference. The IR spectra were recorded on a Perkin-Elmer model 377 spectrometer using KBr pellets. The electronic spectra were obtained in CHCl₃ with a Shimadzu UV-160 spectrophotometer. Magnetic susceptibility measurements were done with a 155 Allied Research vibrating sample magnetometer at room temperature. Melting points were measured using an electrothermal Buchi 512 melting point apparatus. Molecular weights were measured on a Knauer Vapour Pressure Osmometer in CHCl₃ solution at room temperature. Molar conductivities (10⁻³ M in DMSO) were measured with CMD 8500 Laboratory Conductivity Meter. Iron and nickel were determined by spectrophotometeric method using an AA-670 Shimadzu atomic absorption-flame emission spectrophotometer.

3.2. Preparation of the ligand and its complexes

4,4'-dibromobenzilmonoxime was prepared according to a modified procedure described for the preparation of benzilmonoxime in two steps.²⁴ First, 4,4'-dibromobenzil was obtained from the reaction of 4-bromobenzaldehyde with sodium cyanide in the presence of nitric acid in methanol. Then in the second stage, the solution of the product in the same solvent was treated with hydroxylamine hydrochloride in the presence of sodium acetate to yield 4,4'-dibromobenzilmonoxime (Scheme II).

3.2.1. Preparation of 4,4'-dibromobenzil

A solution of 4-bromobenzaldehyde (925 mg, 5 mmol) in methanol (25 mL) was added dropwise to sodium cyanide (245 mg, 5 mmol) in methanol (30 mL) with stirring at room temperature. The mixture was then heated at 80 °C for 8 hours. When concentrated nitric acid (10 mL) was added to the solution a yellow residue was formed which was filtered off, and washed with cold methanol and recrystalized from ethanol to give (1.65 g, 90%) of pure $C_{14}H_8Br_2O_2$. Anal. Calcd: C, 45.68; H, 2.17; Br, 43.44. Found: C, 45.83; H, 1.98; Br, 43.61%. IR (KBr, cm⁻¹): 3052 (m), 3025 (m), 1675 (s), 1591 (w), 1441 (m), 1405 (m), 1293 (w), 704 (m). ¹H-NMR (CDCl₃, δ ppm): 6.8-7.5 (m,

Scheme II Two steps for preparation of the 4,4'-dibromobenzilmonoxime ligand



8H).

3.2.2. Preparation of 4,4'-dibromobenzilmonoxime

To a solution of 4,4'-dibromobenzil (1.839 g, 5 mmol) in warm methanol (30 mL) was added a solution of sodium acetate (410 mg, 5 mmol) and hydroxylamine hydrochloride (342 mg, 5 mmol) in 50% aqueous methanol (25 mL). The reaction mixture was refluxed for 6 hours. When the solution was cooled in a beaker containing ice-water a white precipitate was formed which was filtered off, and washed with cold water. The crude material was then recrystallized from ethanol to give (1.75 g, 92%) of pure C14H9Br2NO2. Anal. Calcd.: C, 43.88; H, 2.35; N, 3.66; Br, 41.74. Found: C, 44.02; H, 2.24; N, 3.53; Br, 41.97%. IR (KBr, cm⁻¹): 3455 (s), 3063 (m), 3022 (m), 1715 (s), 1645 (m), 1438 (m), 1402 (m), 1220 (s), 710 (m). ¹H-NMR (CDCl₃, δ ppm): 6.7-7.5 (m, 8H); 10.20 (s, 1H). ¹³C-NMR (CDCl₃, δ ppm): 201, 198.5, 168, 139, 135, 131, 129; UV-Vis (CHCl₃, λ_{max}/nm , (log ε)): 260 (4.2), 365 (2.8).

3.2.3. Preparation of the complex tris(4,4'-dibromobenzilmonoximato)iron(III)

A fresh solution of sodium methoxide was prepated by dissolving metallic sodium (0.138 g, 6 mmol) in methanol (5 mL) which was added to a hot solution of 4,4'-dibromo-benzilmonoxime (2.290 g, 6 mmol) in methanol (30 mL). The resulting solution was refluxed for 4 hours until a yellow solution was obtained. Then Fe(NO₃)₃.9H₂O (0.808 g, 2 mmol) was dissolved in a minimum quantity of methanol, and was added to this solution, the mixture was refluxed for 5 hours to ensure the completion of the reaction. After cooling the solution in a ice-water container a yellow precipitate was formed which was filtered off, and washed with methanol, and recrystallized from ethanol to give (2.11 g, 88%) of pure product (C₄₂H₂₄Br₆FeN₃O₆). Anal. Calcd.: C, 41.96; H, 2.00; N, 3.50; Br, 39.91; Fe, 4.65. Found: C, 42.12; H, 1.92; N, 3.43; Br, 40.27; Fe, 4.50%. IR (KBr, cm⁻¹): 3058 (m), 3025 (m), 1685 (s), 1625 (m), 1435 (m), 1405 (m), 1225 (s), 705 (m), 492 (m), 448 (m). ¹H-NMR (CDCl₃, δ ppm): 6.6-7.6 (m, 8H). ¹³C-NMR (CDCl₃, δ ppm): 191, 182.5, 169, 138, 135.5, 131, 129; UV-Vis (CHCl₃, λ_{max} /nm, (log ϵ)): 262 (3.8), 365 (2.4), 384 (3.7), 451 (1.8), 542 (1.85).

3.2.4. Preparation of the complex sodium tris(4,4'dibromobenzilmonoximato)nickelate(II)

A fresh solution of sodium methoxide was prepated by dissolving metallic sodium (0.138 g, 6 mmol) in methanol (5 mL) which was added to a stirred hot solution of 4,4'-dibromobenzilmonoxime (2.290 g, 6 mmol) in methanol (30 mL). The resulting solution was refluxed for 4 hours until a yellow solution was obtained. A solution of Ni(NO₃)₂.6H₂O (0.580 g, 2 mmol) in methanol (25 mL) was then added to this solution. The reaction mixture was refluxed for 8 hours to ensure the completion of the reaction. The resulting solution was placed in a ice-water container until a brown precipitate was formed which was filtered off, and washed with cold water and methanol, and recrystallized from ethanol to give (2.21 g, 90%) of pure product (C₄₂H₂₄Br₆N₃NaNiO₆). Anal. Calcd.: C, 41.07; H, 1.96; N, 3.42; Br, 39.07; Ni, 4.78. Found: C, 41.35; H, 1.88; N, 3.35; Br, 39.35; Ni, 4.52%. IR (KBr, cm⁻¹): 3058 (m), 3030 (m), 1690 (s), 1618 (m), 1433 (m), 1409 (m), 1228 (s), 707 (m), 490 (m), 452 (m). ¹H-NMR (CDCl₃, δ ppm): 6.6-7.6 (m, 8H). ¹³C-NMR (CDCl₃, δ ppm): 193, 185, 168, 138.5, 134.5, 132, 129; UV-Vis (CHCl₃, λ_{max}/nm , (log ε)): 264 (3.9), 366 (2.5), 535 (1.84), 744 (1.95).

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