Original Research Article

Schiff Base [(N1Z, N2Z)-N1, N2-bis (1-Phenylethylidene) ethane-1, 2-diamine] with P-Hydroxyphenyl Telluriumtribromide and their Metal Complexes: Synthesis and Characterization

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Abstract

The reaction of aliphatic diamine [ethylene diamine] with acetophenone under showed conditions yielded Schiff base ligand. The another ligand (p-hydroxyphenyl telluriumtribromide) has been added to complexes. The structures of Cu(II), Ni(II), Co(II), Zn(II) and Fe(II) with schiff base compound and organotellurium ligand have been joined by the reacting between copper bromide, hydrite nickel chloride, hydrite cobalt chloride, zinc chloride, and iron bromide and two ligands in 1:2:2 mole ratio. The free ligands and their metal structures have been withdrawn in the solid state. The spectroscopic data of the structures suggest their 1:2:2 buildings structures which are inspected by elemental analysis (CHN), FT-IR, 1H NMR spectroscopy. The spectroscopic studies proposed the octahedral structure for the all structures and the results are represented and analyzed underneath.

Keywords

Complexes, organotellurium, metal complexes, ethylenediamine, acetophenone.

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Introduction

Schiff bases and their bio-dynamic buildings have been concentrated widely over the previous decade. Schiff bases give potential locales to bio-artificially dynamic mixes. Different move and inward move metal buildings with bi, tri and tetratadentate. Schiff bases containing nitrogen and oxygen or sulfur giver particles assume a critical part in organic systems (Malik et al., 2011; Raman et al., 2007). Schiff bases got from an amino and carbonyl compound are an essential class of ligands that direction to metal particles through azomethine nitrogen and have been concentrated widely. In azomethine subordinates, the C,N linkage is crucial for natural action, a few azomethine have been accounted for to have amazing antibacterial, antifungal, anticancer and antimalarial activities (Annapoorani et al., 2013). Schiff bases are for the most part bidentate (Hine et al., 1967), tridentate (Fessenden et al., 1998), tetratadentate (Cimerman et al., 2000) or polydentate (Elmali et al., 2000) ligands equipped for shaping exceptionally stable edifices with move metals. They can just go about as
organizing ligands in the event that they bear an utilitarian gathering.

Organochalcogen science has grown quickly in the course of the last few decades (Arnold, 1995; Smith et al., 2000; Levason et al., 2002), and is credited to potential utilizations of organochalcogen mixes experiencing significant change metal catalyzed natural synthesis (Nishibayshi et al., 1996; Wirth, 2000; Kumar et al., 2008; Shi et al., 2007; Tiecco et al., 2004), as single source forerunners in metalorganic concoction vapor test (MOCVD) forms for creating materials (Steigerwald et al., 1987; Hirpo et al., 1993; Cheng et al., 1996; Anyfantis et al., 2008) in ligand chemistry (Ling et al., 2007) and biochemistry (Shi et al., 2002; Kanda et al., 1999; Kunwar et al., 2007). Uses of organochalcogen mixes have, be that as it may, been confined by troubles in manufactured procedures, sanitization and dangers of specific subordinates. To beat these challenges, sterically cumbersome substituents and chelating bunches in close closeness to selenium and tellurium have been utilized. Inward chelation has been stretched out to outline and orchestrate ligands containing both ""hard"" and ""soft"" donors (Lloyd et al., 2008; Jones et al., 1996).

Experimental

Chemicals and Apparatus

Chemicals acquired from Sigma-Aldrich, Fluka and BDH utilized without filtration. Liquefying point was dictated by utilizing open hairlike tube dissolving point mechanical assembly. 1H NMR spectra was recorded on Bruker 300 MHz spectrometers with TMS as an inner reference utilizing DMSO-d6 dissolvable. Infra-red spectra were recorded with KBr circles utilizing a FTIR spectrophotometer Shimadzu model 8400 S in reach 4000-400 cm-1. Essential examination for Carbon, Hydrogen and Nitrogen were performed by utilizing an Euro vector EA 3000A Elemental Analysis (Italy). Dissolving purposes of every single strong compound were resolved utilizing a MPS10 electrically warmed liquefying point mechanical assembly.

Synthesis of Schiff base

The following compounds were synthesized as described in literature. (4– hydroxyphenyl)tellurim tribromide (Al-Jadaan, 1990) and [(N^1 Z,N^2 Z)-N^{1},N^{2}-bis(1-phenylethylidene)ethane-1,2-diamine] was prepared by the following general method (Deyr et al., 1972).

Preparation of BAPE(C_{18}H_{20}N_{2}): Acetophenone (0.1 mole) and ethylenediamine (0.05 mole) was mixed together in 50 ml. of absolute alcohol and refluxed on water bath for about 2 hrs. The pale yellow solution obtained after reflux gave white crystalline compound on standing for 4 days in a vacuum desiccator. It was filtered under suction and washed with alcohol and finally recrystallised from absolute alcohol, yield 81%, m. p. 109 - 111°C. BAPE is soluble in many organic solvents, but insoluble in water.

Synthesis of Cu(II), Ni(II), Co(II), Zn(II) and Fe(II) complexes

All the new structures were set up by the same methodology: a solution of [(N^1 Z,N^2 Z)-N^{1},N^{2}-bis (1-phenyl-ethyldiene) ethane-1,2-diamine] (BAPE) (1 mmol) in absolute ethanol (10 mL) was added to solution of 4-(hydroxyphenyl) tellurium tribromide (OPTB) (1 mmol) in absolute ethanol (10 mL) and the mixture was refluxed for 30 min and then NiCl_2·6H_2O, CoCl_2·6H_2O, CuBr_2, ZnCl_2 or FeBr_2 (0.5 mmol) in absolute ethanol (10 mL) was added to the ligand solution with stirring and the reaction mixture was
further stirred under reflux for 3 hrs. The obtained colored solution was left standing at room temperature to crystallize. The product was removed by filtration, washed with cooled absolute ethanol, recrystallized from methanol and dried. The exploratory and physical data of the structures are recorded in Table 1.

**Results and Discussion**

The Schiff base BAPE is formed in good yield by the condensation of acetophenone structure 1 with 1,2-diaminoethane occurred in the 1:1 ratio as in structure 2 (Scheme 1).

Schiff base is crystalline in nature and is soluble in common organic solvents. The metal – ligand complexes were synthesized and obtained in pure form. Reactions of (Cu-, Ni-, Zn-, Co- and Fe- halides) with \[(N^1Z,N^2Z)-N^1,N^2\text{-bis(1-phenylethylidene)}\text{ethane-1,2- diamine}\] (BAPE) and 4-(hydroxyphenyl)tellurium tribromide (OPTB) in 1:2:2 molar ratio in ethanol are represented in Scheme 2. It's formed well defined and crystalline complexes with of Cu(II), Ni(II), Co(II), Zn(II) and Fe(II) ions. The results of elemental analysis (C, H, N) with molecular formula and the melting points are presented in Table 1. The results obtained are in good agreement with those calculated for the suggested formula and the melting points are sharp, indicating the purity of the prepared Schiff base and their complexes. The structures of the Schiff base and their complexes under study are given below (Scheme 2). The structure of these Schiff bases is also confirmed by IR and $^1$H NMR spectra, which will be discussed in a detailed manner.

**Infrared spectra**

The IR spectra of mixing ligand (BAPE), (OPTB) and their complexes are presented in Table 2. The IR spectra of the ligands and complexes synthesized are shows two strong bands appeared at range (3099-3121) cm$^{-1}$ and (2861-2931) cm$^{-1}$ due to stretching aromatic (C-H) and aliphatic (C-H) respectively (Silerstien et al., 2005; Shriner et al., 2004). The IR spectra of the complexes were compared with of the free ligand (BAPE) and (OPTB) in order to determine the coordination sites that may be involved in chelating. There was some guide peaks in the spectra of the ligands, which were helpful in achieving this goal. The position and/or the intensities of these peaks are expected to change upon chelating. New peak is also guide peak, as M-N in chelating. This guide peak are shown in Table 2. Upon comparison This band was shifted to higher or lower wave numbers in the complexes, indicating the participation of the azomethine nitrogen in coordination (M—N). New bands are found in the spectra of the complexes in the regions (546-576) cm$^{-1}$ which are assigned to $\nu$(M-O) stretching vibrations for (OPTB) metal complexes. Several medium intensity bands appeared in the (1492-1575) cm$^{-1}$ region of the spectra are due to the stretching of various (C=C) vibrations. The IR spectra of the ligand and complexes synthesized are shows two strong bands appeared at range (1235 - 1265) cm$^{-1}$ and (1621 - 1646) cm$^{-1}$ due to stretching aliphatic (C=N) and (C=N) respectively.

The IR spectra of mixes 1-5 and ligand demonstrated a solid band in reach (1182 - 1195) cm$^{-1}$ can be credited to (C-O) security. A wide band in 3515 cm$^{-1}$ can be alluded to (O-H) bond in free ligand (OPTB) yet This crest will be vanished in IR spectra of edifices was great and clear confirmation to event of response by hydroxyl bunch in (OPTB) ligand. Accordingly; from the IR spectra it is inferred that the (BAPE) acts as an unbiased bidentate ligand facilitated to the means of azomethine N while (OPTB) ligand carries on as monodentate and deprotonated hydroxyl bunch.
Table.1 Physical and analytical data for complexes 1-5

| Comp. | Color | Melting point (°C) | Analysis (%)a | Yield % |
|-------|-------|--------------------|---------------|---------|
|       |       |                    | C             | H       | N       |
| Cu(BAPE)$_2$(OPTB)$_2$ [1] | brown | 210-212            | 38.15 (38.45) | 3.20 (3.55) | 3.71 (3.97) | 57 |
| Fe(BAPE)$_2$(OPTB)$_2$ [2] | Dark brown | 190-192 | 38.35 (38.81) | 3.22 (3.65) | 3.73 (3.90) | 61 |
| Zn(BAPE)$_2$(OPTB)$_2$ [3] | Yellowish-brown | 201-203 | 38.11 (38.91) | 3.20 (3.31) | 3.70 (3.80) | 62 |
| Ni(BAPE)$_2$(OPTB)$_2$ [4] | Brown | 235-237 | 38.28 (38.56) | 3.21 (3.34) | 3.72 (3.93) | 58 |
| Co(BAPE)$_2$(OPTB)$_2$ [5] | brown | 205-207 | 38.27 (38.79) | 3.21 (3.10) | 3.72 (3.55) | 69 |

a calculated values are in parentheses

Table.2 IR Spectroscopic data for Ligand and compounds 1-5

| Ligand/complexes IR (cm$^{-1}$) | $\lambda_{max}$ in nm ( $\varepsilon$ in M$^{-1}$cm$^{-1}$) |
|---------------------------------|-----------------------------------------------------|
| BAPE                           | 3099cm$^{-1}$ v(C-H) Ar., 2931 cm$^{-1}$ v(C-H) aliph., 1566 cm$^{-1}$ v(C=C) Ar., 1645 cm$^{-1}$ v(C=N), 1265 cm$^{-1}$ v(C-N) aliph. |
| Cu(BAPE)$_2$(OPTB)$_2$ [1]     | 3102cm$^{-1}$ v(C-H) Ar., 2875 cm$^{-1}$ v(C-H) Aliph., 1563 cm$^{-1}$ v(C=C) Ar., 1192 cm$^{-1}$ v(C-O) Ar., 1621 cm$^{-1}$ v(C=N), 1235 cm$^{-1}$ v(C-N) Aliph., 435 cm$^{-1}$ v(M-N), 571 cm$^{-1}$ v(M-O). |
| Fe(BAPE)$_2$(OPTB)$_2$ [2]     | 3100 cm$^{-1}$ v(C-H) Ar., 2861 cm$^{-1}$ v(C-H) Aliph., 1575 cm$^{-1}$ v(C=C) Ar., 1182 cm$^{-1}$ v(C-O) Ar., 1633 cm$^{-1}$ v(C=N), 1237 cm$^{-1}$ v(C-N) Aliph., 455 cm$^{-1}$ v(M-N), 546 cm$^{-1}$ v(M-O). |
| Zn(BAPE)$_2$(OPTB)$_2$ [3]     | 3012 cm$^{-1}$ v(C-H) Ar., 2901 cm$^{-1}$ v(C-H) Aliph., 1498 cm$^{-1}$ v(C=C) Ar., 1190 cm$^{-1}$ v(C-O) Ar., 1647 cm$^{-1}$ v(C=N), 1239 cm$^{-1}$ v(C-N) Aliph., 466 cm$^{-1}$ v(M-N), 551 cm$^{-1}$ v(M-O). |
| Ni(BAPE)$_2$(OPTB)$_2$ [4]     | 3121 cm$^{-1}$ v(C-H) Ar., 2910 cm$^{-1}$ v(C-H) Aliph., 1492 cm$^{-1}$ v(C=C) Ar., 1193 cm$^{-1}$ v(C-O) Ar., 1632 cm$^{-1}$ v(C=N), 1245 cm$^{-1}$ v(C-N) Aliph., 455 cm$^{-1}$ v(M-N), 576 cm$^{-1}$ v(M-O). |
| Co(BAPE)$_2$(OPTB)$_2$ [5]     | 3120 cm$^{-1}$ v(C-H) Ar., 2895 cm$^{-1}$ v(C-H) Aliph., 1545 cm$^{-1}$ v(C=C) Ar., 1195 cm$^{-1}$ v(C-O) Ar., 1641 cm$^{-1}$ v(C=N), 1243 cm$^{-1}$ v(C-N) Aliph., 462 cm$^{-1}$ v(M-N), 581 cm$^{-1}$ v(M-O). |
Scheme 1 Preparative method for base [(N1Z,N2Z)-N1,N2-bis(1-phenylethylidene)ethane-1,2-diamine]

Fig. 2 Preparative methods for complexes (1:2:2) molar ratio

Where $\text{MX}_2\times \text{H}_2\text{O} = \text{CuBr}_2$, NiCl$_2$.6H$_2$O; ZnCl$_2$, CoCl$_2$.6H$_2$O or FeCl$_2$
Table 3 1H NMR Spectroscopic data for Ligand and compounds 1-5

| Ligand/ complexes         | Chemical shift (ppm)                                      |
|---------------------------|-----------------------------------------------------------|
| BAPE                      | 7.31 - 7.92 (m, 10H, aromatic), 5.33 (s, 4H, CH₂-N), 1.96 (s, 6H, CH₃). |
| Cu(BAPE)₂(OPTB)₂ [1]      | 7.35 - 7.85 (m, 20H, Ar. PABE), 6.97 - 7.32 (m, 8H, Ar. OPTB), 3.65 (s, 8H, CH₂-N), 1.91 (s, 12H, CH₃). |
| Fe(BAPE)₂(OPTB)₂ [2]      | 7.31 - 7.89 (m, 20H, Ar. PABE), 6.79 - 7.21 (m, 8H, Ar. OPTB), 3.71 (s, 8H, CH₂-N), 1.91 (s, 12H, CH₃). |
| Zn(BAPE)₂(OPTB)₂ [3]      | 7.34 - 7.81 (m, 20H, Ar. PABE), 6.91 - 7.12 (m, 8H, Ar. OPTB), 3.63 (s, 8H, CH₂-N), 1.90 (s, 12H, CH₃). |
| Ni(BAPE)₂(OPTB)₂ [4]      | 7.31 - 7.83 (m, 20H, Ar. PABE), 6.91 - 7.22 (m, 8H, Ar. OPTB), 3.72 (s, 8H, CH₂-N), 1.95 (s, 12H, CH₃). |
| Co(BAPE)₂(OPTB)₂ [5]      | 7.39 - 7.88 (m, 20H, Ar. PABE), 6.93 - 7.25 (m, 8H, Ar. OPTB), 3.87 (s, 8H, CH₂-N), 1.90 (s, 12H, CH₃). |

1H NMR spectra

1H NMR ghostly information in deturated DMSO arrangement of the integrated mixes are given in Table 1. The 1H NMR spectra of the Schiff base in DMSO displays multisignals inside the 7.31 – 7.92 ppm reach are appointed to the sweet-smelling protons of (BAPE) ligand rings and multisignals inside the 6.79 – 7.32 ppm extent are doled out to the fragrant protons of (OPTB) ligand rings. The spectra of the edifices are inspected in examination with those of the guardian Schiff base. The 1H NMR spectra of the Schiff base integrated from acetophenone and ethylene diamine uncovered a sign at 5.31 ppm (singlet) because of the proton OH in (OPTB) ligand and The proton OH has vanished in 1H NMR spectra of edifices, showing that the OH proton is evacuated by chelation with the metal ion.⁴³

The singlet signals at 3.63 – 3.87 ppm recommended the attribution of the proton of the CH₂-N bunches in (BAPE) ligand while in free ligand has been showed up in 5.33 ppm.

In conclusion, another complexes has been set up by the reaction between schiff base (BAPE) as ligand bidntate and organotellurium as monodentate ligand with metal halide. Explanatory IR, 1HNMR and elemental examination (CHN) revealed a 1:2:2 mile ratio in which one of Schiff base, organotellurium and metal halide. In another study will be utilized this edifices as a part of an extensive variety of natural exercises, including anti-infection, hostile to proliferative, calming, and antipyretic properties.

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