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Synthesis, spectral characterization, thermal and photoluminescence properties of Zn(II) and Cd(II)-azido/thiocyanato complexes with thiazolylazo dye and 1,2-bis(diphenylphoshino)ethane

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ABSTRACT

A series of complexes of the type [M(**L**)(dppe)X_2]; where M = Zn(II) or Cd(II); **L** = 4-(2′-thiazolylazo)chlorobenzene (**L_1**), 4-(2′-thiazolylazo)bromobenzene (**L_2**) and 4-(2′-thiazolylazo) iodobenzene (**L_3**); dppe = 1,2-bis(diphenylphosphino)ethane; X = N_3^- or NCS^- have been prepared and characterized on the basis of their microanalysis, molar conductance, thermal, IR, UV–vis and ^1H NMR spectral studies. IR spectra show that the ligand **L** is coordinated to the metal atom in bidentate manner *via* azo nitrogen and thiazole nitrogen. An octahedral structure is proposed for all the complexes. The thermal behavior of the complexes revealed that the thiocyanato complexes are thermally more stable than the azido complexes. All the complexes exhibit blue-green emission with high quantum yield as the result of the fluorescence from the intraligand emission excited state.

1. Introduction

Considerable research efforts have been given to the preparation of transition metal complexes of hybrid ligands because they can provide new materials with useful properties such as magnetic exchange [1–4], electrical conductivity [5,6], photoluminescence [7–9], non-linear optical property [10–12] and antimicrobial activity [13,14]. Among various ligands azo group containing ligands have received much attention in recent years. Due to presence of azo (–N=N–) group, these compound possesses several distinctive properties such as aggregation, optical data storage and tautomerisation [15,16]. Thiazolylazo compounds particularly are important because they can form different types of coordination compounds with transition metals due to the several electron rich donor centers with unusual structural and chemical properties. Because of the importance of thiazolylazo derivatives and its ability to act as polyfunctional ligand many studies on its metal complexes have been carried out [17–21]. 1,2-Bis(diphenylphosphino)ethane (dppe) is one of the most versatile phosphine ligand, capable of binding to the metal atoms in variety of ways: monodentate, chelating or bridging. They possess high covalency as well as ligand field effect to enforce a drastic change in magnetic and other behaviour of the resulting compounds [22]. The pseudohalides such as N_3^- and NCS^- also have distinct effects during the formation of complexes, not only acting as the charge equilibrium, but also influencing the final structures of the complexes. They can bind metal ions depending on the nature and the stereochemical environment surrounding the metal ion as well as the steric hindrance introduce by the other coordinating ligands [23–26].

In this paper, we report the synthesis and characterization of Zn(II) and Cd(II) complexes derived from some thiazolylazo ligands (**L_{1-3}**) with 1,2-bis(diphenylphosphino)ethane and N_3^- or NCS^- as coligands. The complexes were characterized particularly by elemental analysis, molar conductance, spectral (IR, UV–vis and ^1H NMR) and thermal studies. The photoluminescence property of the complexes has also been reported.

2. Experimental

2.1. Materials and methods

All the chemicals were used as received and solvents were purified according to the literature methods [27]. $Zn(NO_3)_2{\cdot}6H_2O$ (E-Merk India), $Cd(NO_3)_2{\cdot}4H_2O$ (E-Merck India), NaN_3 (Aldrich, USA), NH_4NCS (Aldrich, USA), 1,2-bis(diphenylphosphino)ethane (Aldrich) were purchased from the respective concerns and were used as received. Microanalysis (C, H, N and S) was performed on a Thermo Finnigan FLASH EA-112 CHNS analyzer. Electronic spectra were recorded on a Shimadzu 3600 UV-Visible-NIR spectrophotometer. ^1H NMR spectra of the samples were recorded on Bruker 300 MHz FT-NMR in DMSO-d_6. Molar conductance (Λ_M) was measured on the ELICO (CM-185) conductivity bridge using *ca.* 10^{-3} M

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solution in DMF. Infrared spectra were recorded on Perkin-Elmer FT-IR spectrometer as KBr pellets in the 4000–400 cm^{-1} range. Thermal analysis of the complexes was carried out on a Perkin-Elmer thermal analyzer in nitrogen atmosphere at a heating rate of 10 °C/min. The luminescence properties were measured using a JASCO F.P. 750 fluorescence spectrophotometer at room temperature (298 K) in DMF solution with 1 cm^3 path length quartz cell.

2.2. *Synthesis of thiazolylazo ligands ($\mathbf{L_{1-3}}$)*

The thiazolylazo ligands 4(2′-thiazolylazo)chlorobenzene ($\mathbf{L_1}$), 4(2′-thiazolylazo)bromobenzene ($\mathbf{L_2}$) and 4(2′-thiazolylazo)iodobenzene ($\mathbf{L_3}$) were prepared by diazotization of 2-aminothiazole by reported procedure [28] and were characterized by elemental analysis, IR and UV–vis spectra.

2.3. *Preparation of azido complexes (**1a**–**6a**)*

To a methanolic solution of appropriate metal salt (1 mmol, 0.297 g $Zn(NO_3)_2{\cdot}6H_2O$) or 0.308 g $Cd(NO_3)_2{\cdot}4H_2O$), a methanolic solution of **L** (1 mmol, 0.223 g, $\mathbf{L_1}$ or 0.268 g, $\mathbf{L_2}$ or 0.315 g, $\mathbf{L_3}$) was added while stirring. To this, a CH_2Cl_2 solution (5 ml) of 1,2-bis(diphenylphosphino)ethane (1 mmol, 0.398 g) was added, followed by the addition of NaN_3 (2 mmol, 0.130 g) in warm methanol. The resultant mixture was stirred for 3 h at room temperature. The solid product obtained was filtered, washed with ethanol:water (1:1) mixture and dried under vacuum over $CaCl_2$.

2.4. *Preparation of thiocyanato complexes (**1b**–**6b**)*

To a methanolic solution of appropriate metal salt (1 mmol, 0.297 g $Zn(NO_3)_2{\cdot}6H_2O$) or 0.308 g $Cd(NO_3)_2{\cdot}4H_2O$), a methanolic solution of **L** (1 mmol, 0.223 g, $\mathbf{L_1}$ or 0.268 g, $\mathbf{L_2}$ or 0.315 g, $\mathbf{L_3}$) was added while stirring. To this, a CH_2Cl_2 solution (5 ml) of 1,2-bis-(diphenylphosphino)ethane (1 mmol, 0.398 g) was added, followed by the addition of NH_4NCS (2 mmol, 0.152 g) in warm methanol. The resultant mixture was stirred for 3 h at room temperature. The solid product obtained was filtered, washed with ethanol: water (1:1) mixture and dried under vacuum over $CaCl_2$.

3. Results and discussion

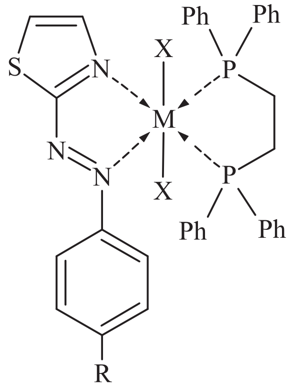
The reaction of thiazolylazo ligands $\mathbf{L_{1-3}}$ with Zn(II) and Cd(II) salts in presence of dppe and NaN_3 or NH_4NCS in 1:1:1:2 molar ratio yields mononuclear complexes of the type $[M(\mathbf{L})(dppe)(N_3)_2]$ (**1a**–**6a**) and $[M(\mathbf{L})(dppe)(NCS)_2]$ (**1b**–**6b**); where M = Zn(II), Cd(II); **L** = 4-(2′-thiazolylazo)chlorobenzene ($\mathbf{L_1}$), 4-(2′-thiazolylazo)bromobenzene ($\mathbf{L_2}$), 4-(2′-thiazolylazo)iodobenzene ($\mathbf{L_3}$); dppe = 1,2-bis(diphenylphosphino)ethane (Fig. 1). The analytical and physical properties of the complexes are given in Table 1. The air stable, moisture insensitive complexes are insoluble in common organic solvents such as dichloromethane, acetonitrile, chloroform except DMF and DMSO. The results of elemental analysis support the composition of the complexes. The molar conductances of the complexes in 10^{-3} M solution in DMF are in the range of 15.93–27.09 $\Omega^{-1}\,cm^2\,mol^{-1}$ indicating that, although some dissociation of these complexes seems to occurs in the solvent, the conductance value support the non-electrolytic nature of the complexes [29].

3.1. *IR spectra*

The IR spectra of ligands and their complexes are found to be quite complex as they are in general exhibit large number of bands of varying intensities. However, a strong band observed

Table 1
Analytical and physical data for the Zn(II) and Cd(II) complexes.

Complex	M.F. colour	Yield (%)	M.p./d.p. (°C)	Analytical data % found (Calc.)				Λ_M ($\Omega^{-1}\,cm^2\,mol^{-1}$)
				M	C	H	N	
$[Zn(\mathbf{L_1})(dppe)(N_3)_2]$ (**1a**)	$C_{35}H_{30}N_9SP_2ClZn$ (brown)	63	247	8.38 (8.48)	54.58 (54.48)	3.81 (3.92)	16.46 (16.34)	16.73
$[Zn(\mathbf{L_2})(dppe)(N_3)_2]$ (**2a**)	$C_{35}H_{30}N_9SP_2BrZn$ (brown)	62	253	8.14 (8.02)	51.36 (51.52)	3.58 (3.71)	15.67 (15.45)	22.31
$[Zn(\mathbf{L_3})(dppe)(N_3)_2]$ (**3a**)	$C_{35}H_{30}N_9SP_2Izn$ (gray)	65	284	7.44 (7.57)	48.54 (48.71)	3.39 (3.50)	14.75 (14.61)	18.32
$[Zn(\mathbf{L_1})(dppe)(NCS)_2]$ (**1b**)	$C_{37}H_{30}N_5S_3P_2ClZn$ (dark brown)	62	272	8.03 (8.13)	55.38 (55.30)	3.68 (3.76)	8.97 (8.71)	19.92
$[Zn(\mathbf{L_2})(dppe)(NCS)_2]$ (**2b**)	$C_{37}H_{30}N_5S_3P_2BrZn$ (dark brown)	64	268	7.54 (7.71)	52.54 (52.40)	3.69 (3.57)	8.22 (8.26)	21.51
$[Zn(\mathbf{L_3})(dppe)(NCS)_2]$ (**3b**)	$C_{37}H_{30}N_5S_3P_2Izn$ (brown)	62	264	7.22 (7.31)	49.78 (49.65)	3.27 (3.37)	7.72 (7.82)	15.93
$[Cd(\mathbf{L_1})(dppe)(N_3)_2]$ (**4a**)	$C_{35}H_{30}N_9SP_2ClCd$ (brown)	67	262	13.58 (13.72)	51.21 (51.36)	3.81 (3.69)	15.64 (15.41)	27.09
$[Cd(\mathbf{L_2})(dppe)(N_3)_2]$ (**5a**)	$C_{35}H_{30}N_9SP_2BrCd$ (gray)	64	286	13.14 (13.03)	48.79 (48.71)	3.58 (3.50)	14.53 (14.61)	23.10
$[Cd(\mathbf{L_3})(dppe)(N_3)_2]$ (**6a**)	$C_{35}H_{30}N_9SP_2Icd$ (dark gray)	65	243	12.46 (12.34)	46.08 (46.19)	3.44 (3.32)	13.78 (13.85)	24.70
$[Cd(\mathbf{L_1})(dppe)(NCS)_2]$ (**4b**)	$C_{37}H_{30}N_5S_3P_2ClCd$ (gray)	63	268	13.32 (13.21)	52.13 (52.24)	3.44 (3.55)	8.39 (8.23)	22.31
$[Cd(\mathbf{L_2})(dppe)(NCS)_2]$ (**5b**)	$C_{37}H_{30}N_5S_3P_2BrCd$ (green)	65	274	12.42 (12.55)	49.51 (49.65)	3.24 (3.37)	7.95 (7.82)	17.72
$[Cd(\mathbf{L_3})(dppe)(NCS)_2]$ (**6b**)	$C_{37}H_{30}N_5S_3P_2Icd$ (gray)	62	256	11.67 (11.93)	46.68 (47.17)	3.12 3.21)	7.58 (7.43)	19.72



M = Zn(II), Cd(II); R = Cl, Br, I; X = N_3, NCS

Fig. 1. Proposed structure of the complexes.

at 1618–1624 cm^{-1} in the spectra of all ligands ($\mathbf{L_{1-3}}$) is due to ν(C═N) of thiazole nitrogen. This band shifted to lower frequencies (1586–1595 cm^{-1}) in the complexes indicates involvement of thiazole nitrogen in coordination [30]. Another band appeared at the frequency range 1469–1480 cm^{-1} in the spectra of ligands assigned to –N═N– group shifted to lower frequency (1434–1440 cm^{-1}) in the complexes indicates involvement of azo nitrogen in coordination with metal ion [31]. This is also supported by the appearance of ν(M–N) band at 435–448 cm^{-1} in the complexes [32]. The band at 744 cm^{-1} in the spectra of free ligands $\mathbf{L_{1-3}}$ remains unaltered in the spectra of all the complexes, indicating non-involvement of thiazole sulfur in coordination. The IR spectra of all the Zn(II) and Cd(II) complexes exhibited the expected bands due to dppe ligand at 1483, 1384, 1172 and 738 cm^{-1}. The azido complexes **1a**–**6a** show strong bands at ~2113 and ~1343 cm^{-1}. These are assigned to ν_{as} and ν_s stretching vibrations of the coordinated azido group [33]. The thiocyanato complexes **1b**–**6b** exhibit a strong and sharp band at ~2096 cm^{-1}, a weak band at ~762 cm^{-1} and another weak band at ~488 cm^{-1}, which can be attributed to ν(CN), ν(CS) and ν(NCS), respectively. These values are typical for N-bonded thiocyanate complexes [34].

3.2. *Electronic and NMR spectra*

The electronic spectra for all the complexes were recorded in DMF (10^{-4} M) in the range 200–900 nm. All the complexes exhibit two bands in the region 278–292 and 330–342 nm. These transitions are presumably due to intra-ligand π–π^* and n–π^* transitions, respectively. Another intense absorption observed in the 437–462 nm region at longer wavelength in the spectra of Zn(II) and Cd(II) complexes which may be assigned to the characteristic n–π^* transition of the coordinated azo ligand.

The ^1H NMR spectra of all the complexes were recorded in DMSO-d_6. The ^1H NMR spectra of the complexes show that the resonances of phenyl protons of the coordinated dppe ligand overlap to some extent with those of phenyl hydrogen atoms of **L** in the complexes. However, a broad multiplet observed in the range δ 6.87–7.56 ppm for all the complexes were assigned to the phenyl group of dppe together with ring proton of azo ligand $\mathbf{L_{1-3}}$. The spectra of all the complexes exhibit a broad singlet at approximately δ 2.45 ppm is due to CH_2 protons of the dppe ligand [35].

3.3. *Thermal analysis*

In order to examine the thermal stability of the complexes, thermo gravimetric (TG) and differential thermal analysis (DTA) were carried out for complexes **1a**–**6a** and **1b**–**6b** between the room temperature and 1000 °C under nitrogen atmosphere. Typical TG–DTA curves of **1a** and **1b** are presented in Figs. 2 and 3.

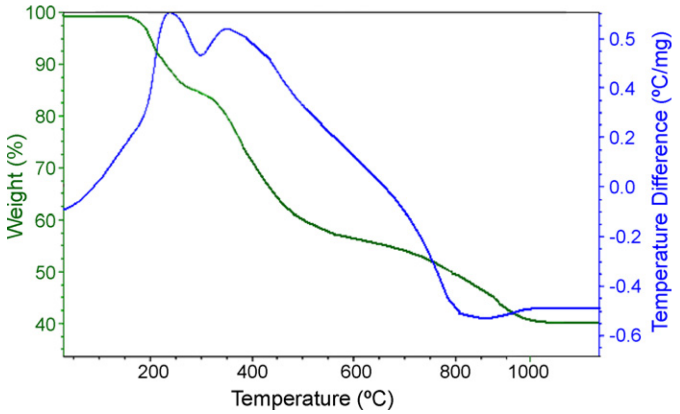


Fig. 2. TG–DTA curve of [Zn($\mathbf{L_1}$)(dppe)(N_3)$_2$] (**1a**).

The thermal decomposition process of Zn(II) and Cd(II) azido complexes (**1a**–**6a**) involves three decomposition stages. The first stage for both Zn(II) and Cd(II) azido complexes takes place in the range 188–274 °C for **1a**–**3a** and 190–288 °C for **4a**–**6a** with exothermic DTA peaks in the range 243–256 °C (**1a**–**3a**) and 246–248 °C (**4a**–**6a**) corresponding to loss of two azide ions. The second stage occurs in the range 265–390 °C for **1a**–**3a** and 282–417 °C for **4a**–**6a** which may be attributed to the decomposition of the ligand $\mathbf{L_1}$, $\mathbf{L_2}$ or $\mathbf{L_3}$. The DTA curve gives exothermic peaks in the range 345–352 °C for **1a**–**3a** and 340–352 °C for **4a**–**6a**. The third decomposition stage takes place in the range 376–874 °C for **1a**–**3a** and 405–893 °C for **4a**–**6a** corresponding to the decomposition of dppe molecule leaving anhydrous ZnO and CdO as a residue at the end.

The Zn(II) and Cd(II) thiocyanato complexes **1b**–**6b** show very similar behavior to the azido complexes. However, the first decomposition stage occurs in the range 198–300 °C for **1b**–**3b** and 198–294 °C for **4b**–**6b** with accompanying exothermic DTA peaks in the range 340–346 °C (**1b**–**3b**) and 340–348 °C (**4b**–**6b**) which may be attributed to the loss of two thiocyanate ions. The second stage occurs in the range 286–418 °C for **1b**–**3b** and 284–420 °C for **4b**–**6b** assigned to the decomposition of the ligand $\mathbf{L_1}$, $\mathbf{L_2}$ or $\mathbf{L_3}$. The DTA curve gives exothermic peaks in the range 340–346 °C for **1b**–**3b** and 340–348 °C for **4b**–**6b**. The third stage takes place in the range 410–882 °C (**1b**–**3b**) and 409–890 °C (**4b**–**6b**) corresponding to the decomposition of dppe molecule leaving anhydrous ZnO and CdO, respectively.

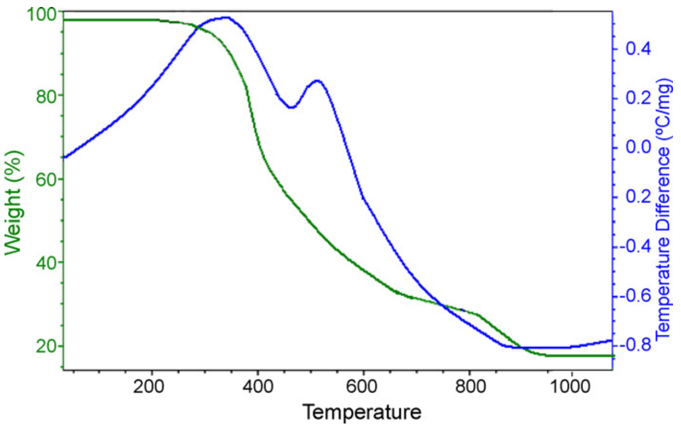


Fig. 3. TG–DTA curve of [Zn($\mathbf{L_1}$)(dppe)(NCS)$_2$] (**1b**).

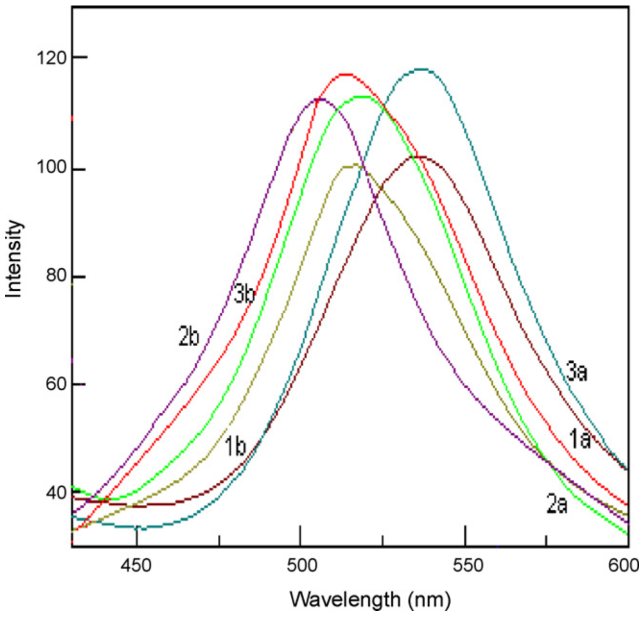


Fig. 4. Emission spectra of Zn(II) complexes (**1a**–**3a** and **1b**–**3b**).

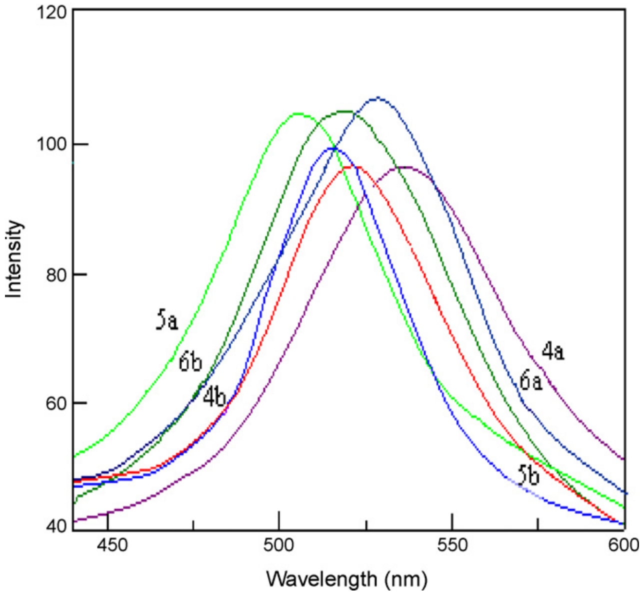


Fig. 5. Emission spectra of Cd(II) complexes (**4a**–**6a** and **4b**–**6b**).

3.4. Photoluminescence properties

The photoluminescence properties of ligands $\mathbf{L_{1-3}}$ and their Zn(II) and Cd(II) complexes were investigated at room temperature (298 K) in DMF solution. The ligands $\mathbf{L_{1-3}}$ show a photoluminescence with emission at 417–427 nm at 298 K upon excitation at 350–370 nm. The complexes show strong fluorescence with a high quantum yield in DMF solution. The Zn(II) complexes (**1a**–**3a** and **1b**–**3b**) exhibit fluorescence with λ_{max} at 507–538 nm (Fig. 4) excited upon 363–374 nm, however, Cd(II) complexes (**4a**–**6a** and **4b**–**6b**) show emission at 507–534 nm excited upon 356–368 nm (Fig. 5) with life time of 2.52–2.59 and 2.53–2.61 ns, respectively. No emission originating from metal-centered excited states are expected for the Zn(II) and Cd(II) complexes, since they are difficult to oxidize or reduce due to their d^{10} configuration. Thus the emission observed in the complexes is assigned to the ($\pi \rightarrow \pi^*$) intraligand fluorescence and these are red shifted by >140 nm. The intensity of emission in the Zn(II) and Cd(II) complexes is found to be higher than that of free ligands. The chelation of the ligands increases the rigidity and reduces the loss of energy by thermal vibrational decay [36,37]. Significant differences in the intensities of the emission of the complexes from that of the ligands may be considered as an evidence of the metal–ligand complexation. Introduction of substituents on the azo ligands ($\mathbf{L_{1-3}}$) has also an interesting effect on the intensity of emission spectra.

The fluorescence quantum yield (Φ) of the complexes was determined by using quinine sulfate as a reference with known Φ_R of 0.52 and appeared at 0.014–0.069. The area of emission spectrum was integrated using the software available in the instrument and quantum yield was calculated according to the following equation.

$$\Phi_S = \frac{A_S}{A_R} \times \frac{(\mathrm{Abs})_R}{(\mathrm{Abs})_S} \times \Phi_R$$

Here Φ_S and Φ_R are the fluorescence quantum yield of the sample and reference, respectively. A_S and A_R are the area under the fluorescence spectra of the sample and reference, respectively. $(\mathrm{Abs})_S$ and $(\mathrm{Abs})_R$ are the respective optical densities of the sample and the reference solution at the wavelength of excitation.

4. Conclusion

In the present study, the synthesis and spectroscopic characterization of Zn(II) and Cd(II) complexes of thiazolylazo dye with 1,2-bis(diphenylphosphino)ethane and N_3^- or NCS^- coligands have been carried out by microanalysis, IR, UV–vis, ^{1}H NMR, thermal analysis and fluorescence properties. IR spectra reveal that the ligand is coordinated to the metal atom in bidentate manner *via* azo nitrogen and thiazole nitrogen. Thiocyanato complexes are thermally more stable than the azido complexes. All the complexes exhibit blue-green emission with high quantum yield as the result of the fluorescence from the intraligand emission excited state.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.saa.2010.09.004.

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