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RESEARCH PAPER

Synthesis and characterization of palladium(II) and platinum(II) mixed ligand complexes of the type $[M(bpozs)_2(dppf)]Cl_2$, bpozs=2-(benzylthio)-5-phenyl-1,3,4-oxadiazole

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ABSTRACT:

Treatment of $[PdCl_2(dppf)].CH_2Cl_2$ or $[PtCl_2(dppf)]$ with two moles of 2-(benzylthio)-5-phenyl-1,3,4-oxadiazole (bpozs) in (ethanol: chloroform) solvents for (2 h) afford $[M(bpozs)_2(dppf)]Cl_2$ in good yield. The synthesized ligand and complexes were identified using FT-IR, ^{1}H-NMR, ^{13}C-NMR, ^{13}P-NMR, CHNS analysis, Uv-Visible spectra and conductivity measurements. The physiochemical data indicated that the prepared complexes have square planar geometries and the metal ions coordinate with the ligands through two phosphorous atoms of (dppf) and nitrogen atom of (bpozs).

KEY WORDS: palladium(II), platinum(II), diphosphine, oxadiazole.

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INTRODUCTION:

In recent years, the interest of preparing coordination compounds with desired properties -functional compounds have been increased. One of the promising ligands for creating multifunctional complexes are compounds containing a 1,3,4-oxadiazole fragment (Pirimova *et al.*, 2020). Oxadiazoles are interesting class of five-membered heterocyclic compounds containing two atoms of nitrogen and one atom of oxygen (Paulo *et al.*, 2018). They exist in four different regioisomeric forms, namely 1,2,3-, 1,2,4-, 1,2,5-, and 1,3,4-oxadiazoles. In the past 40 years, oxadiazole-based ligands stimulated the curiosity of many researchers, creating a vast literature that spans from synthesis to different applications. In fact, oxadiazoles exhibit a broad range of uses: in medicinal chemistry they were employed as drug candidates for several diseases, in organic synthesis as useful intermediates, and in material science as building blocks for new polymers (Giovanni and Alessio, 2019) (Zainab and Hameedi, 2020).Transition metal complexes of 1,3,4-oxadiazole derivatives are well known for their biological importance as well as their anticarcinogenic, antibacterial and antifungal properties (Adil, 2017).

A series of 1,1'-bis(diphenylphosphino)ferrocene possessing platinum(II) complexes depicting a novel series of tentative anticancer drug candidates. Compounds of this sort have already been displayed to illustrate both antineoplastic and antimicrobial activity (Puxty *et al.*, 2005). Metal complexes of bidentate phosphines have conceived considerable attention due to their possible use as antitumor agents (Al-Jibori *et al.*, 2013). Herein, we report the synthesis and identification of new mixed ligand Pd(II) and Pt(II) complexes including both 2-(benzylthio)-5-phenyl-1,3,4-oxadiazole (bpozs) and 1,1'-

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bis(diphenylphosphino)ferrocene (dppf) as ligands.

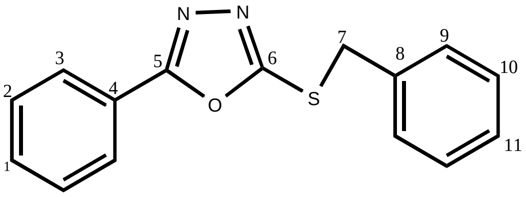
2. Materials and Methods

The ($PdCl_2$, $PtCl_2$, dppf, 2-Mercapto-5-phenyl-1,3,4-oxadiazole, materials were commercially available from Yahoo Chem. China. The $[PdCl_2(dppf)].CH_2Cl_2$ and $[PtCl_2(dppf)]$ complexes were prepared according to the literature (Rezan *et. al.,* 2017). IR spectra were recorded on IR-Affinity-1, Shimadzu CORP-A21375003225 spectrophotometer in the range of 400-4000 cm^{-1} using KBr discs. (^1H, ^{13}C and ^{31}P)-NMR spectra were recorded on a Bruker 400 MHZ Ultra-shied. Electronic spectra were recorded on a UV-Vis. spectrometer, AE-UV1609 (UK) CO., LTD. The conductivity was measured on a conductivity meter type Senz µSiemen conductivity tester. Elemental analysis was carried out on Euro EA 3000 Elemental Analyzer.

2.2. Synthesis of (bpozs) ligand

A solution of (4 mmole, 0.712 g) of 2-Mercapto-5-phenyl-1,3,4-oxadiazole (phozsH) in (10 ml) ethanol, was added to ethanol solution (10 ml) of NaOH (4 mmole, 0.16 g). The resultant mixture was refluxed for (15 min.) and (4 mmole, 0.474 ml) of benzyl bromide was added and refluxed for (4 h.). A white solid of (bpozs) was obtained after cooling of the solution in an ice bath. The formed precipitate was filtered off, dried and recrystallized from ethanol.

Yield = 0.85g, 60.93 %; mp: 95-96 °C; Color: White. $C_{15}H_{12}N_2OS$. IR (ν_{max}/cm^{-1}): ν(C-H) aromatic 3051, 2993; ν(C-H) aliphatic 2914, 2848; ν(C=N) 1608; ν (N-N) 1465; ν (C-O-C) 1080; ν (C-S) 704. ^1H-NMR (296K, ppm, d^6-DMSO): d, 8.05 ppm, ^3J(HH)=8 Hz. (2H, phenyl protons); m, 7.67-7.61 ppm (3H, phenyl protons); d, 7.56 ppm, ^3J(HH)= 8 Hz. (2H, phenyl protons); m, 7.44-7.35 ppm (3H, phenyl protons); s, 4.63 ppm (2H, CH_2 group), ^{13}C-NMR (DMSO, δ, 400 MHz): 158 and 156 $C_{5,6}$ (oxadiazole); 128.1 C_8; 123.8 C_2; 121.03 $C_{1,10}$; 120.8 C_9; 120.44 C_3; 119.75 C_{11}; 118.32 C_4; 39.88 $C_7(CH_2)$.



2-(benzylthio)-5-phenyl-1,3,4-oxadiazole (bpozs)

2.3. Synthesis of complexes

2.3.1. Preparation of $[Pd(bpozs)_2(dppf)]Cl_2$ (1)

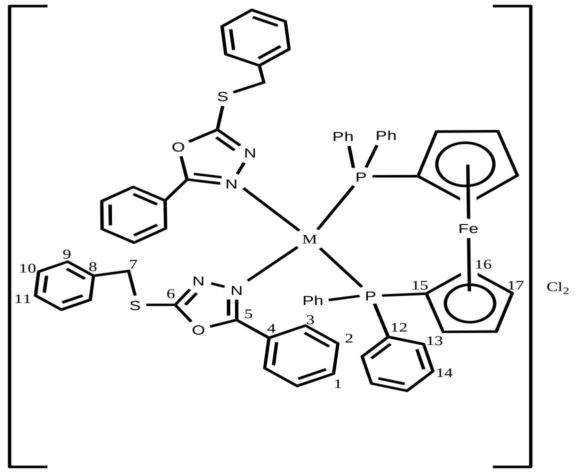
A warm solution of (bpozs) ligand (0.235 mmole, 0.066 g) in ethanol (10 ml) was added to a solution of $[PdCl_2(dppf)].CH_2Cl_2$ (0.117 mmole, 0.095 g) in $CHCl_3$ (20 ml). The mixture was refluxed for (2 h.) then filtered off. A brown precipitate was afforded after evaporation of the filtrate to dryness at room temperature.

Yield = 0.10 g, 66.2 %, mp: 91-92 °C; Color: brown. Anal. Calc. for $C_{64}H_{52}FeCl_2\,N_4PdO_2P_2S_2$: C, 60.6; H, 4.10; N, 4.41; S, 5.05. Found: C, 59.98; H, 4.35; N, 3.62; S, 5.91 %. IR (ν_{max}/cm^{-1}): ν(C-H) aromatic 3051; ν(C-H) aliphatic 2924; ν(C=N) 1608; ν(N-N) 1465; ν(P-Ph) 1435; ν(C-O-C) 1091; ν(C-S) 692; ν(Pd-N) 528. ^1H-NMR (295K, ppm, $CDCl_3$): s, 7.93 ppm, m, 7.36 ppm (30H, phenyl protons); s, 4.47 ppm (2H, CH_2); s, 4.4, 4.34 (8H, Cp), ^{13}C-NMR ($CDCl_3$, δ, 400 MHz): 165 and 163 $C_{6,5}$(oxadiazole); 136.05 C_{12}; 135.6 C_{13}; 132.10 C_8; 131.8 C_2; 129.49 $C_{1,10,14}$; 129.27 C_9; 128.57 C_3; 127.13 C_{11}; 124.02 C_4; 75, 73, 71 C_{15-17}(Cp); 37.33 $C_7(CH_2)$, ^{31}P-{^1H}-NMR; δP= 30.68 ppm.

2.3.2. Preparation of $[Pt(bpozs)_2(dppf)]Cl_2$ (2)

A warm solution of (bpozs) ligand (0.235 mmole, 0.066 g) in ethanol (10 ml) was added to a solution of $[PtCl_2(dppf)]$ (0.117 mmole, 0.095 g) in $CHCl_3$ (20 ml). The mixture was refluxed for (2 h.) then filtered off. A yellow solid of the complex was appeared after evaporation of the filtrate to dryness at room temperature.

Yield = 0.12 g, 74.5 %, Decomposes at: 253°C; Color: Yellow. Anal. Calc. for $C_{64}H_{52}FeCl_2 N_4PtO_2P_2S_2$: C, 56.64; H, 3.83; N, 4.13; S, 4.72. Found: C, 56.51; H, 4.55; N, 4.26; S, 4.00 %. IR (ν $_{max}$/cm^{-1}): ν(C-H) aromatic 3078; ν(C-H) aromatic 3078; ν(C-H) aliphatic 2966; ν(C=N) 1653; ν(N-N) 1465; ν(P-Ph) 1435; ν(C-O-C) 1097; ν(C-S) 690; ν(Pt-N) 518. ^{1}H NMR (295K, ppm, $CDCl_3$): s, 7.86, m, 7.37 ppm (30H, phenyl protons); s, 4.36, 4.18 ppm (8H, Cp); s, 4.53 ppm (2H, CH_2), ^{13}C NMR ($CDCl_3$, δ, 400 MHz): 164 and 162 $C_{6,5}$(oxadiazole); 136 C_{12}; 135.31 C_{13}; 132 C_8; 131.58 C_2; 129.41 $C_{1,10,14}$; 129.19 C_9; 128.28 C_3; 127.04 C_{11}; 124.6 C_4; 75, 74, 72 C_{15-17}(Cp); 38 $C_7(CH_2)$, ^{31}P-{^{1}H}-NMR; δP= 9.79 ppm; J(Pt-P) = 3168 Hz.



3. Results and discussion

3.1. FT-IR, ^{1}H-NMR and ^{13}C-NMR Spectrum of (bpozs) ligand

In the IR spectrum of bpozs, the (SH) stretching band of phozsH compound at (2567 cm^{-1}) Fig. (1) was vanished and a new weak band at (2914 and 2848) cm^{-1} was appeared that corresponded to aliphatic (CH_2) group, this evidence for coordination of oxadiazole sulfur atom with benzyl group (Joshi *et al.*, 2015). The spectra exhibited bands at (1608, 1465, 1080 and 704) cm^{-1}, were respectively attributed to ν(C=N, N-N, C-O-C and C-S) of oxadiazole group Fig. (2) (Kumar *et al.*, 2014)(Al-Azzawi and Hamd, 2013)(Rangappa Santosh *et al.,* 2019).

The ^{1}H-NMR spectrum displayed a singlet band at (4.63) ppm, corresponded to the (CH_2) proton and disappearance of the thiol proton at (3) ppm was indicated for the formation of bpozs ligand. The phenyl protons of bpozs ligand appeared as two doublets at (8.05) ppm; (^3J(HH)= 8 Hz.) and δ(7.56) ppm; (^3J(HH)= 8 Hz.), with two unresolved mutliplets within δ(7.67-7.61 and 7.44-7.35) ppm. Fig. (5) (Joshi *et al.*, 2015)(Kumar *et al.,* 2014)(Bhava *et al.,* 2013).

The ^{13}C-NMRspectrum showed peaks at δ(158 and 156) ppm, were attributed to C_6 and C_5 of oxadiazole ring, respectively (Almajan *et al.*, 2008). The aromatic carbon atoms (C_{1-4} and C_{8-11}) of bpozs ligand were observed at δ(121.03,123.8,120.44,118.32 and 128.1,120.8,121.03,119.75) ppm respectively, furthermore, a new signal was detected at δ(39.88) ppm, attributed to carbon atom of (CH_2) group Fig. (6) (Tank and Acharya, 2013) (Aras and Hassan, 2018).

3.2. FT-IR, ^{1}H-NMR, ^{13}C-NMR and ^{31}P-{^{1}H}-NMR spectra of the synthesized complexes

The infrared spectra of Pd(II) and Pt(II) complexes Fig. (3, 4), displayed a weak intensity bands at (2924 and 2966) cm^{-1}, were respectively allocated to ν(CH_2) of benzyl group. Moreover, the ν(P-Ph) of dppf and ν(C=N), ν(N-N) and ν(C-S) stretching bands of bpozs ligand in (1 and 2) complexes occurred at (1435), (1608, 1653), (1465) and (692, 690) cm^{-1}, correspondingly (Jensen and Nielsen, 1963)(Zainab and Hameedi, 2020). Furthermore, a new weak intensity band was appeared at (528 and 518) cm^{-1}, were respectively attributed to the N-bound coordination of bpozs ligand to Pd and Pt metal (Al-Jibori *et al.,* 2002)(Al-Jibori et al., 2015).

The ^1H-NMR, ^{13}C-NMR and ^{31}P-{^1H}-NMR spectra for both (1 and 2) complexes were measured in $CDCl_3$ solvent.

^1H NMR spectra were as expected, both (1 and 2) complexes displaying singlet and multiplets at δ(7.93,7.36 and 7.86,7.37) ppm were respectively assigned to the phenyl protons and the two signals at δ(4.4, 4.34 and 4.36,4.18) ppm, were due to protons of cyclopentadienyl ligand, correspondingly (Al-Jibori *et al.,* 2012)(Riyadh, 2016). The aliphatic (CH_2) proton of bpozs in 1 and 2 complexes occurred as a singlet band at δ(4.47 and 4.53) ppm, respectively . In the spectrum of complex 2 there is no ^3J(Pt-H), which means that Pt is coordinated to bpozs through more basic N- atom not the S-atom (Lobana *et al.,* 2000)(Ivana and Marija, 2017)(Al-Jibori *et al.,* 2002).

The ^{13}C-NMR spectra of complex 1 and 2 Fig. (7, 8), exhibit signals at δ(165,163 and 164,162) ppm, were assigned to the two oxadiazole carbon atoms, respectively. The phenyl carbon atoms of bpozs and dppf ligands appeared at δ(136.05 C_{12}; 135.6 C_{13}; 132.10 C_8; 131.8 C_2; 129.49 $C_{1,10,14}$; 129.27 C_9; 128.57 C_3; 127.13 C_{11}; 124.02 C_4) in complex 1 and at δ(136 C_{12}; 135.31 C_{13}; 132 C_8; 131.58 C_2; 129.41 $C_{1,10,14}$; 129.19 C_9; 128.28 C_3; 127.04 C_{11}; 124.6 C_4) in complex 2 (Almajan *et al.,* 2008). The carbon atoms of (Cp) groups (C_{15-17}) of phosphine ligand in 1 and 2 complexes occurred at δ(75, 73, 71 and 75, 74, 72) ppm and existence of a peak at δ(37.33 and 38) ppm, corresponded to the (CH_2) carbon atom (C_7) of bpozs ligand, respectively (Al-Jibori *et al.,* 2012)(Nur *et al.*, 2018)(Ivana and Marija, 2017).

The ^{31}P-{^1H}-NMR spectrum of complex (1 and 2) Fig. (9, 10), show a singlet band with platinum satellites, indicates that the electronic environment of both phosphorous atoms of dppf ligand are the same; both phosphorous are equivalent and trans to the same atom.

The spectrum of $[Pt(bpozs)_2(dppf)]Cl_2$ complex showed a singlet at δ(9.79) ppm with associated platinum satellites, *J*(Pt-P) = 3168 Hz. and the $[Pd(bpozs)_2(dppf)]Cl_2$ complex exhibited a singlet band at δ(30.68) ppm, theses suggests that dppf behaves as a bidentate chelating ligand that linked to metal ions through both phosphorous atoms and both P-atoms trans to N-atom of bpozs ligand (Al-Jibori *et al.,* 2012) (Al-Jibori *et al.,* 2007).

3.3. Elemental analysis for the prepared complexes

The elemental analysis (CHNS) data for the synthesized complexes are coherent with the recommended stoichiometries Table (1).

Electronic Spectra of the Prepared Complexes 3.4.

The electronic spectrum of (bpozs) ligand Fig. (11) was measured in methanol and their synthesized mixed ligand complexes in DMSO solvent Fig. (12, 13). The spectrum of bpozs ligand illustrated two absorption bands in the Uv. region at (41666 and 37037) cm^{-1}, these transitions were respectively belonged to π- π* and n-π* transitions. The electronic spectrum of Pd(II) and Pt(II) complexes exhibited charge transfer band at (38461) cm^{-1} with two d-d bands in the uv. and visible regions at (32258, 21276) and (30303, 23255) cm^{-1}, were associated to $^1A_{1g} \rightarrow {}^1E_g$ and $^1A_{1g} \rightarrow {}^1B_{1g}$ transitions correspondingly Table (2), these electronic transitions indicated that the complexes are formed as a square planer geometry (Sutton, 1968).

3.5. Molar conductivity for the prepared complexes

The molar conductivity of bpozs ligand was surveyed for (10^{-3} M) solution in methanol and the prepared complexes in DMSO solvent at (25 °C). The conductivity measurements for both complexes are high, it was deduced that the prepared complexes are electrolyte that formed in the ratio of (1:2) as illustrated in Table 3.

Conclusion

We have illustrated herein that palladium(II) and platinum(II) complexes of the type $[M(bpozs)_2dppf]Cl_2$ are synthesized from the addition of two moles of (bpozs) ligand to the prepared $[MCl_2(dppf)]$ complexes, where M=Pd(II) and Pt(II). Characterization was relatively straight-forward on the basis of spectroscopic and analytical data. According to the measurements data, we deduced that the prepared complexes have a square planer structure in which bpozs ligand bonded in a monodentate fashion through nitrogen atom, while the dppf ligand bind to metals centre through both P-atoms. Moreover, based on the molar conductivity data, it has been proposed that the synthesized complexes are ionic in the ratio of (1:2).

Acknowledgements

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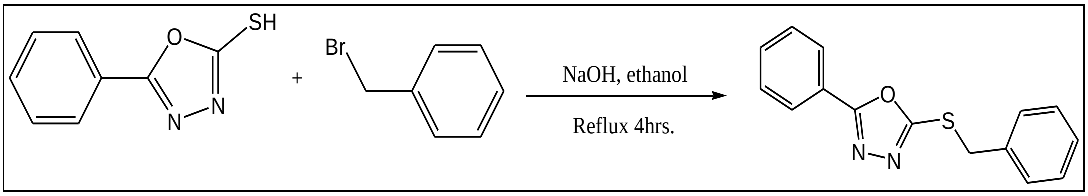
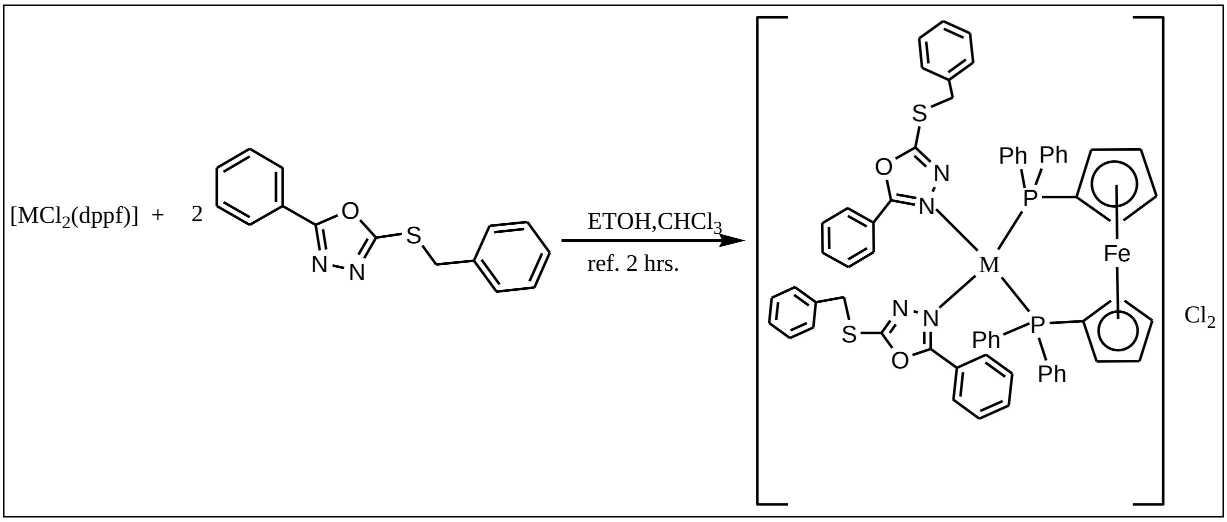


Scheme 1. Synthesis of (bpozs) ligand



Scheme 2. Synthesis of $[M(bpozs)_2(dppf)]Cl_2$, where M= Pd(II) or Pt(II)

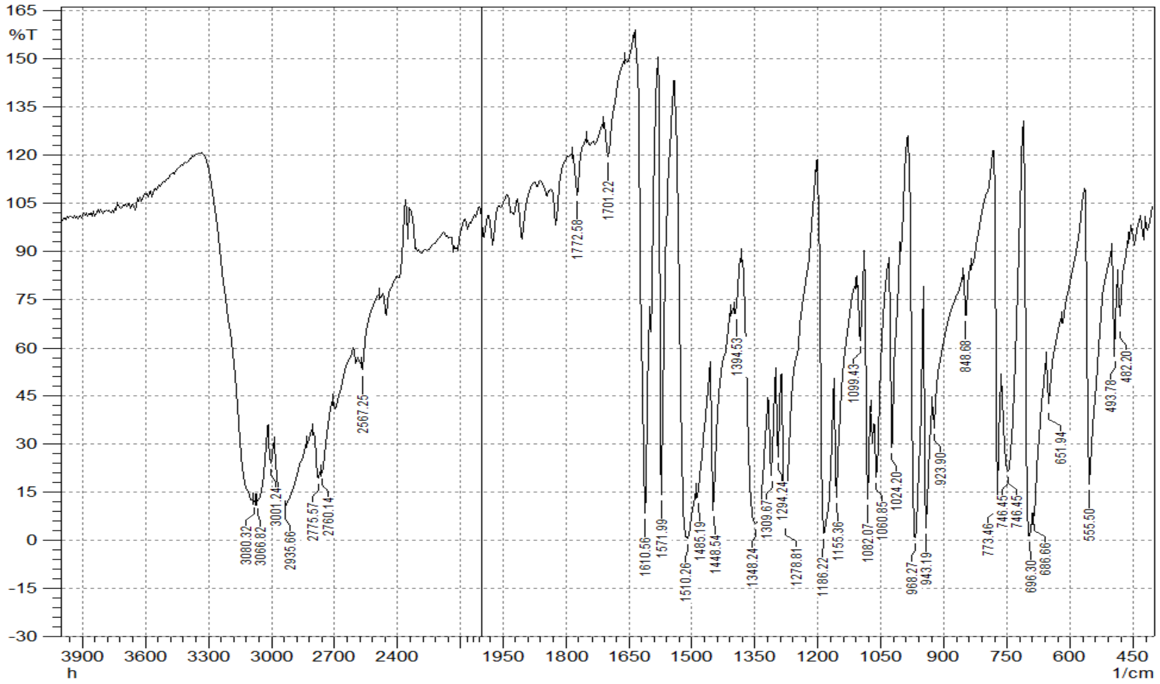


Figure 1: IR spectrum of phozsH ligand

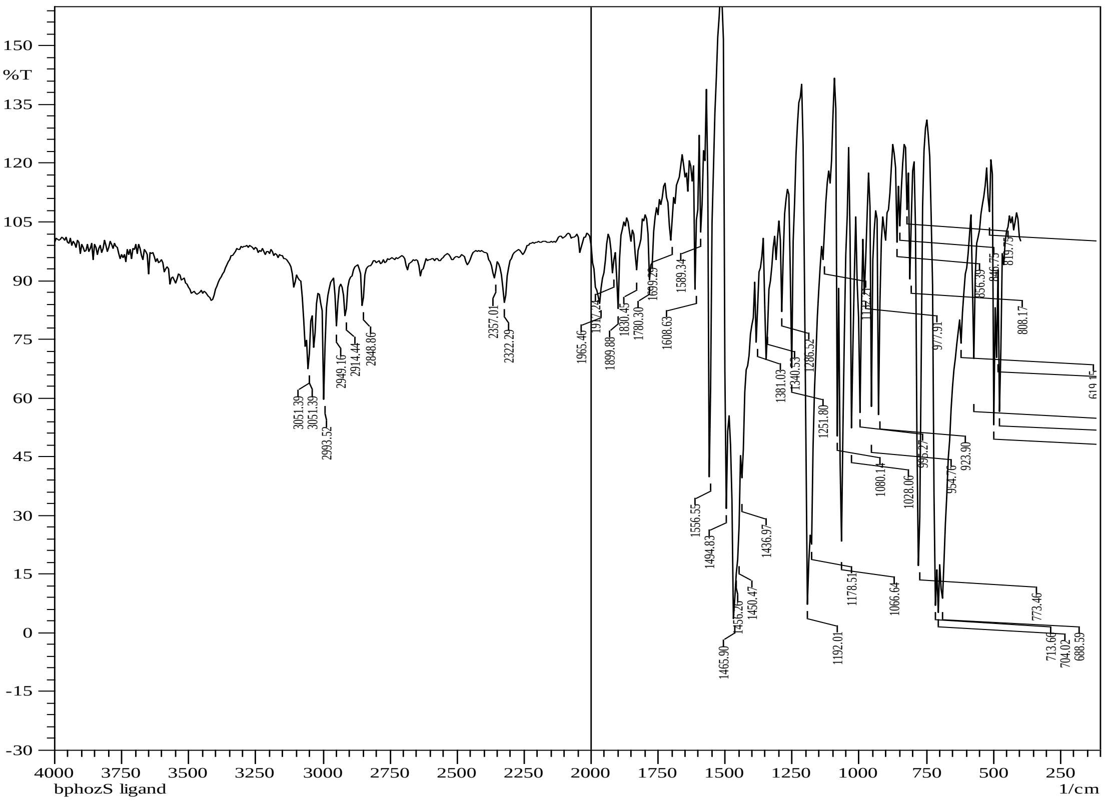


Figure 2: IR spectrum of bpozs ligand

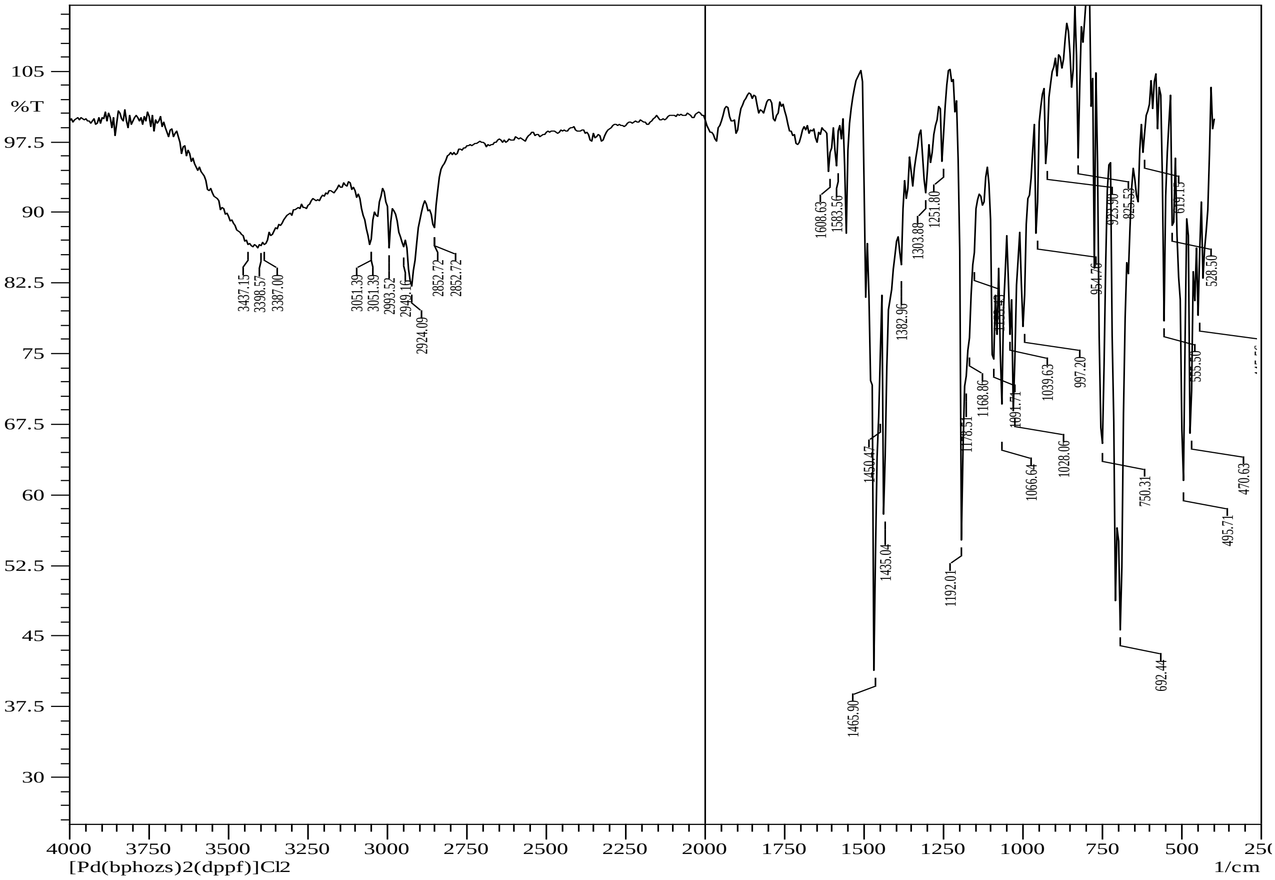


Figure 3: IR spectrum of complex 1

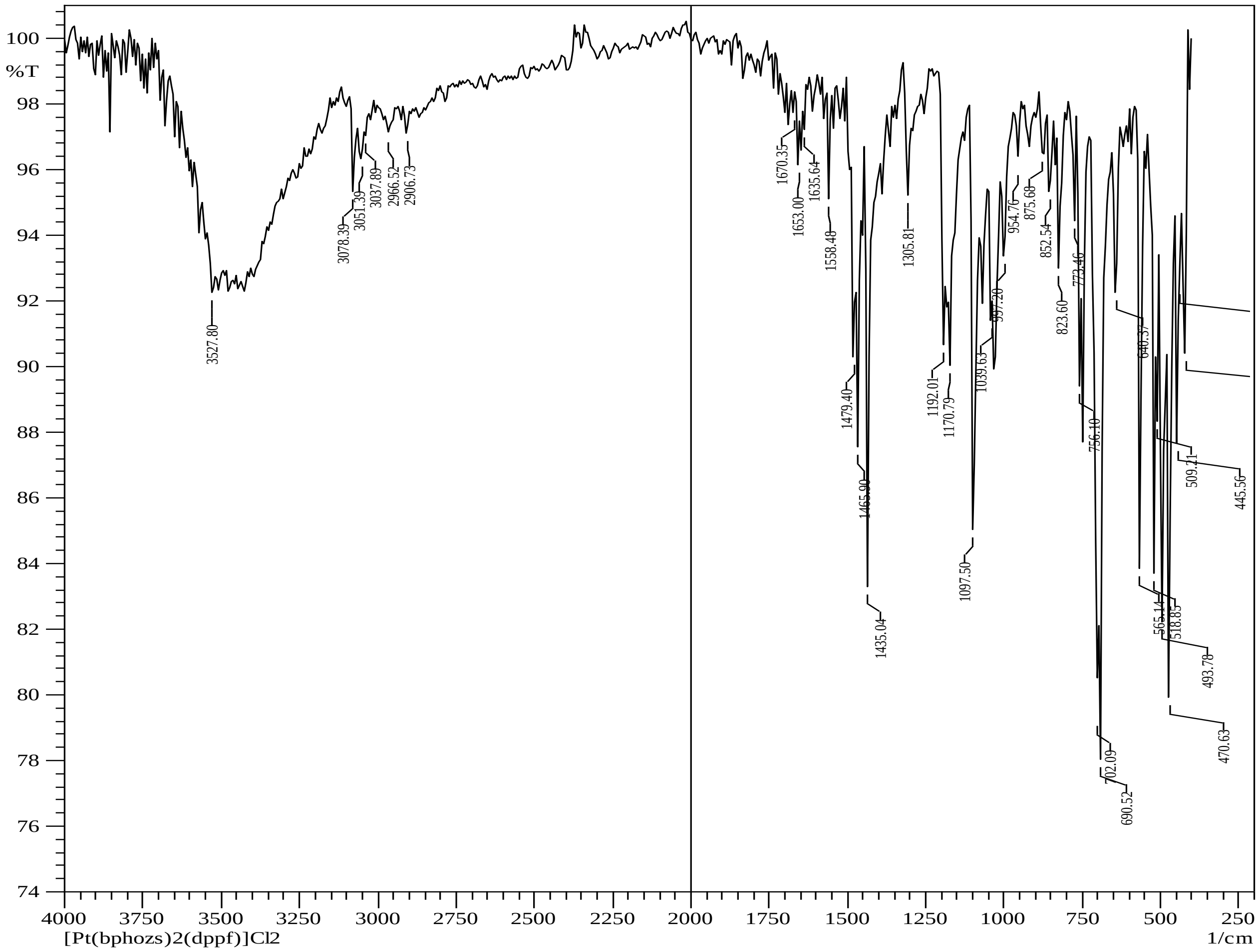


Figure 4: IR spectrum of complex 2

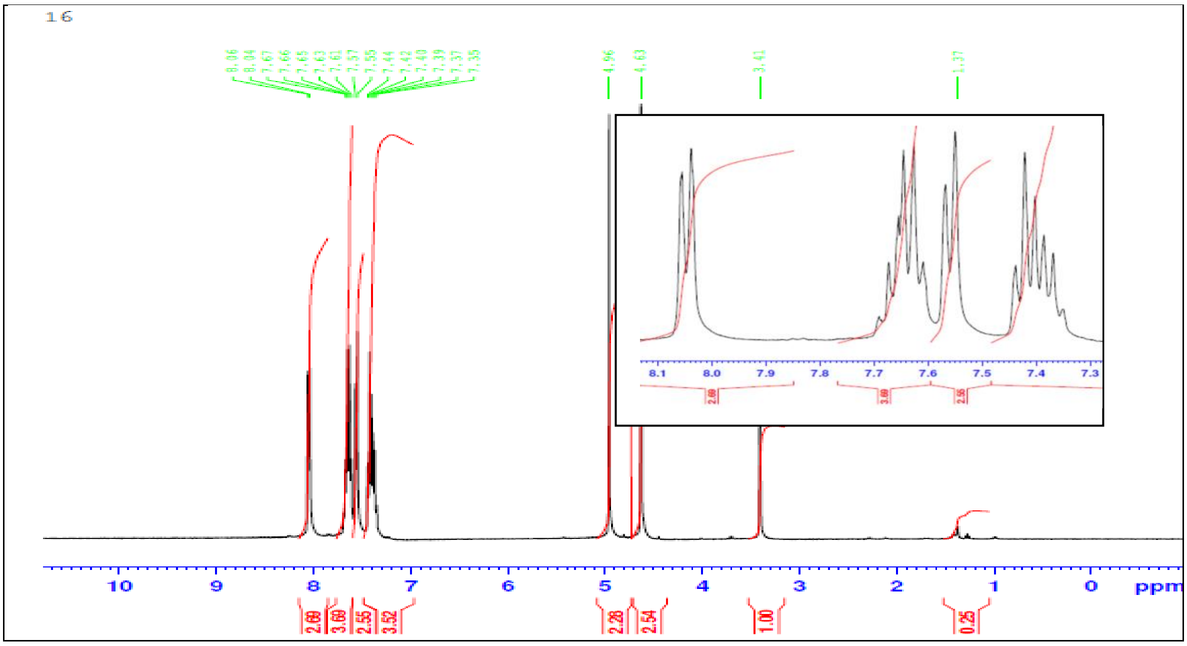


Figure 5: ^{1}H-NMR spectrum of bpozs ligand

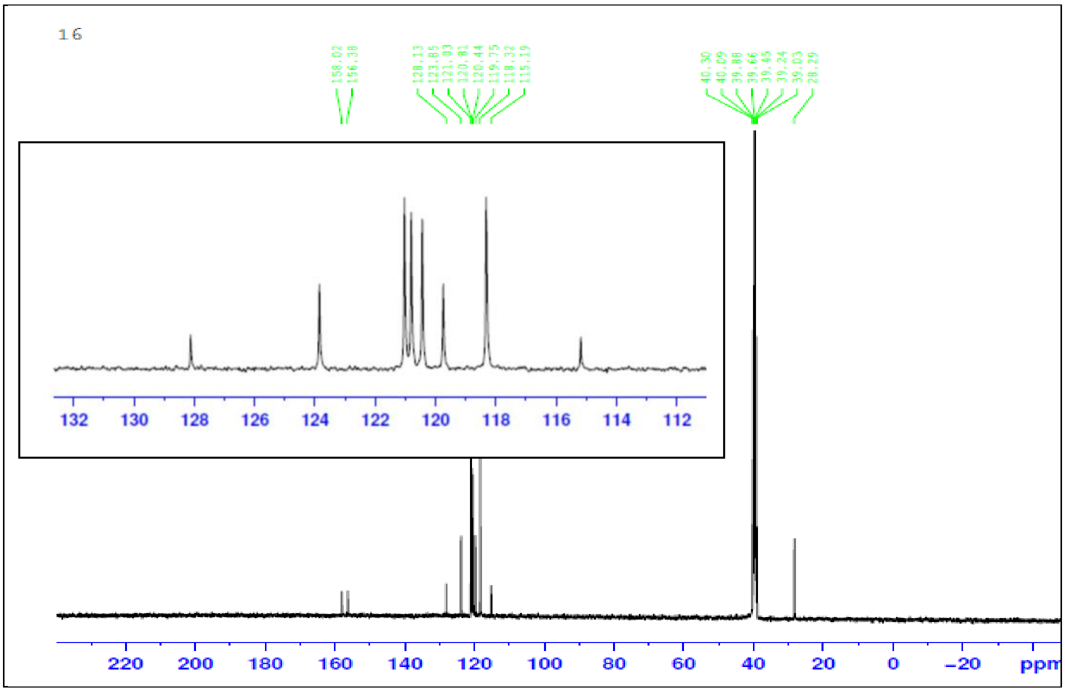


Figure 6: ^{13}C-NMR spectrum of bpozs ligand

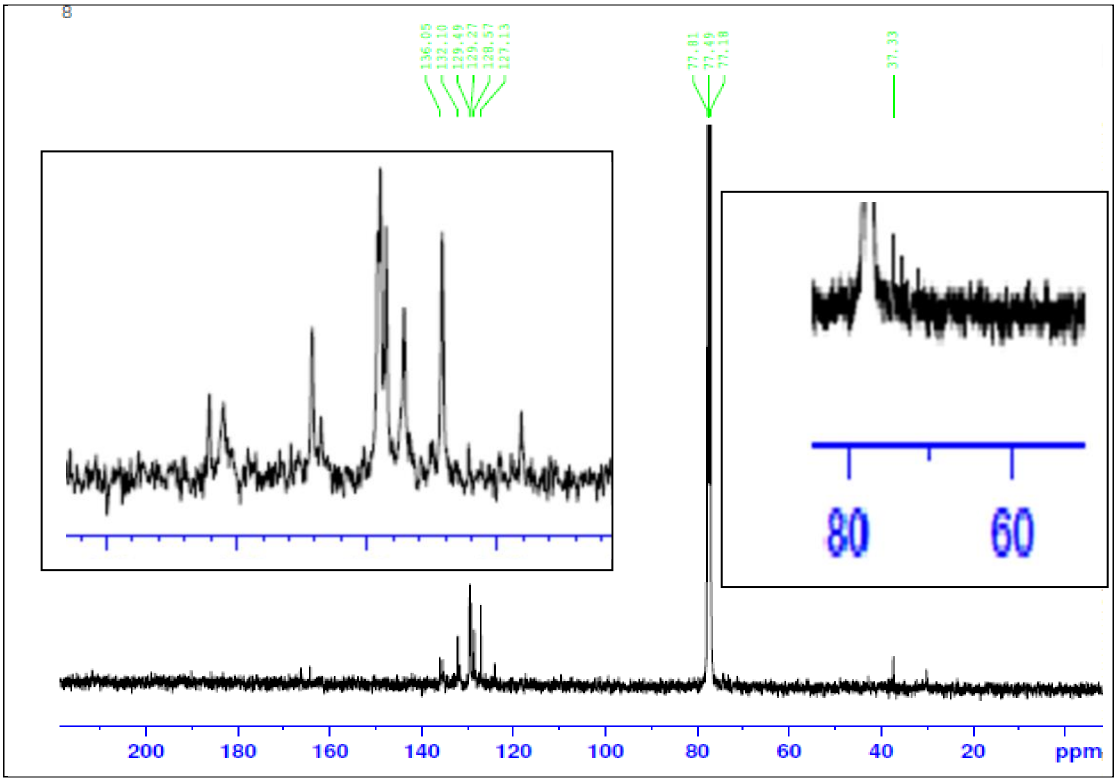


Figure 7: ^{13}C-NMR spectrum of complex 1

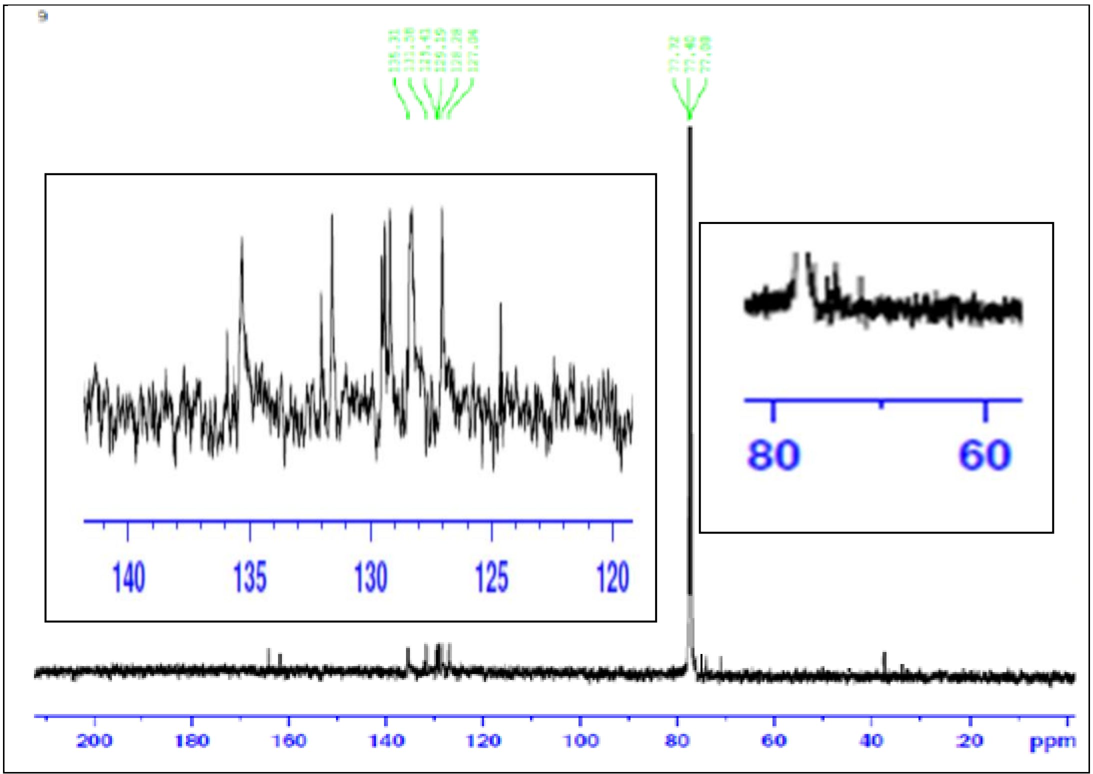


Figure 8: ^{13}C-NMR spectrum of complex 2

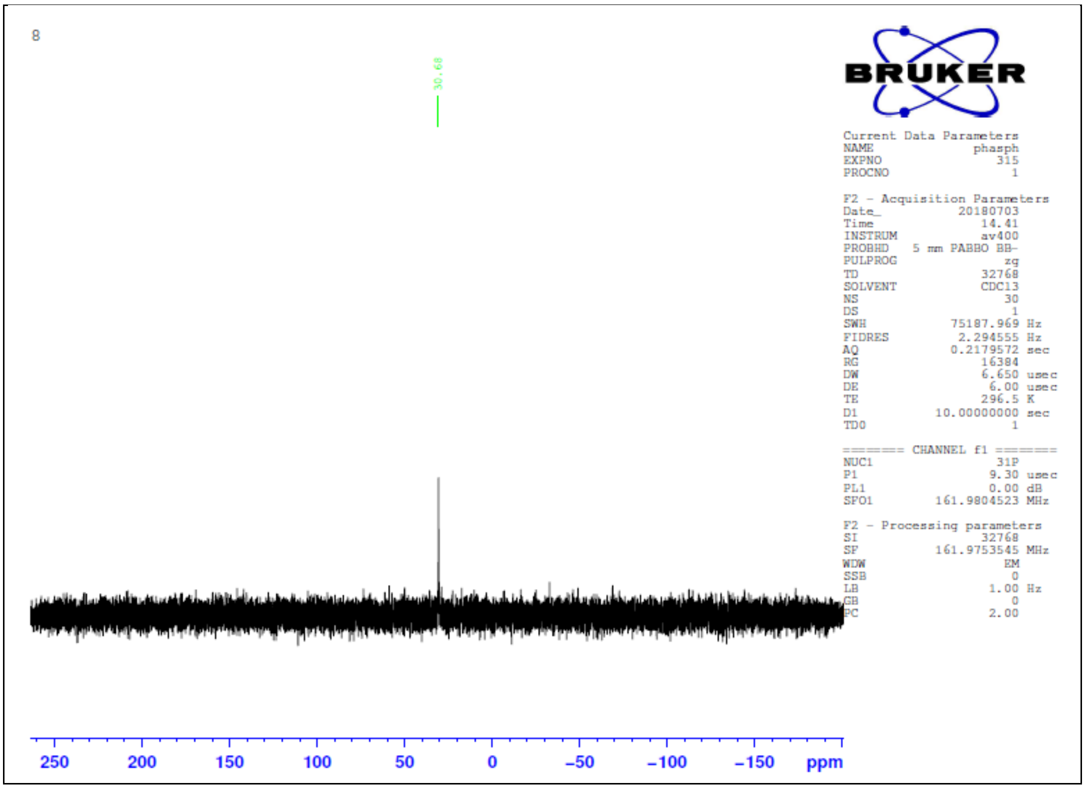


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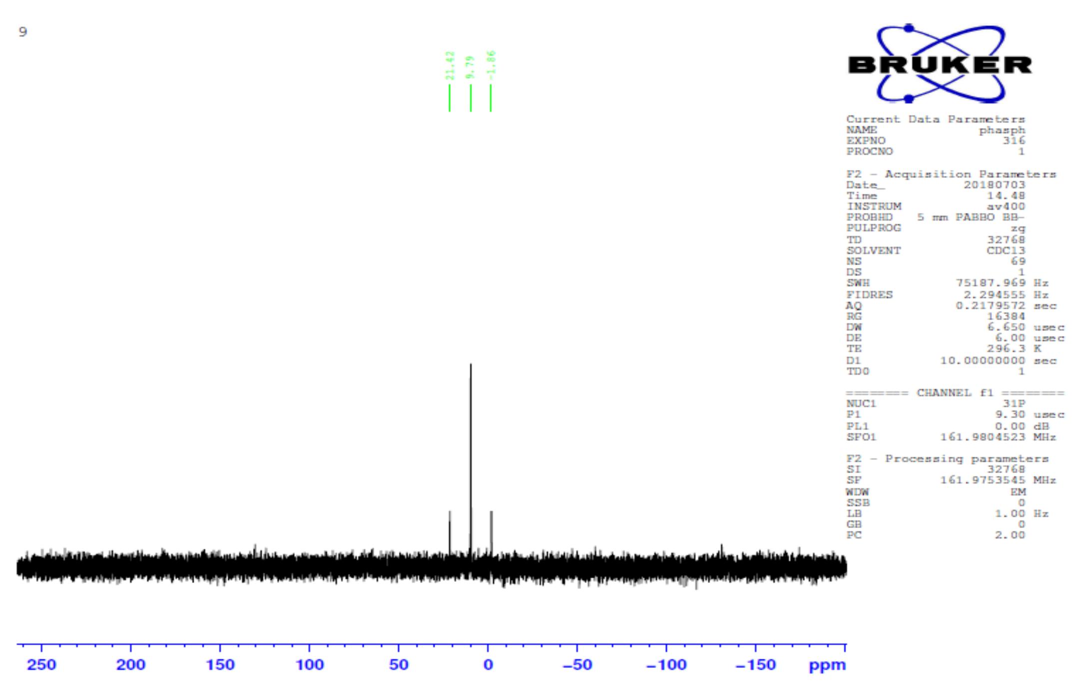
Figure 9: ^{31}P-{^{1}H}-NMR spectrum of complex 1



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PC                  2.00
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Figure 10: ^{31}P-{^{1}H}-NMR spectrum of complex 2

Table 1: Colors, molecular weight, melting points and elemental analysis for the synthesized complexes

No.	Complexes	Color	M.Wt g/mol	M.P. (°C)	(Calculated) Found %			
					C	H	N	S
	bpozs	White	268.06	95-96	…	…	…	…
1	$[Pd(bpozs)_2(dppf)]Cl_2$	Brown	1267.2	91-92	(60.6) 59.98	(4.10) 4.35	(4.41) 3.62	(5.05) 5.91
2	$[Pt(bpozs)_2(dppf)]Cl_2$	Yellow	1355.88	d.p. 253	(56.64) 56.51	(3.83) 4.55	(4.13) 4.26	(4.72) 4.00

Table 2: Electronic spectral bands of the ligand and its metal complexes

Complexes	Absorption band		Assignment Transition
	cm^{-1}	nm	
bpozs	41666	240	$\pi \rightarrow \pi^*$
	37037	270	$n \rightarrow \pi^*$
1	38461	260	C.T.
	32258	310	$^1A_{1g} \rightarrow {}^1E_g$
	21276	470	$^1A_{1g} \rightarrow {}^1B_{1g}$
2	38461	260	C.T.
	30303	330	$^1A_{1g} \rightarrow {}^1E_g$
	23255	430	$^1A_{1g} \rightarrow {}^1B_{1g}$

Table 3: Molar conductivity (cm^2. ohm^{-1}. mol^{-1}) of (10^{-3} M) solution in DMSO for the synthesized complexes

No.	Complexes	Molar conductivity (cm^2. ohm^{-1}. mol^{-1})
	bpozs	26
1	[Pd(bpozs)$_2$(dppf)]Cl$_2$	72
2	[Pt(bpozs)$_2$(dppf)]Cl$_2$	68

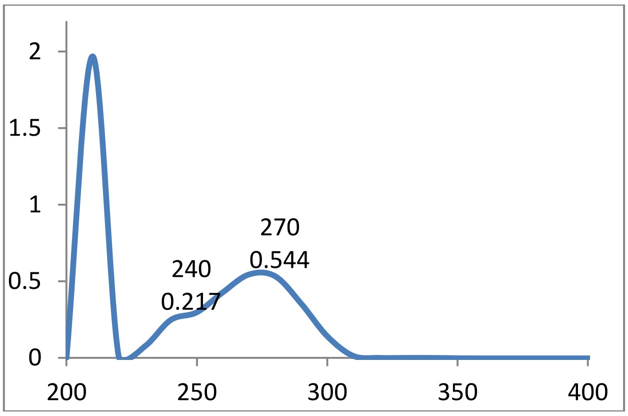
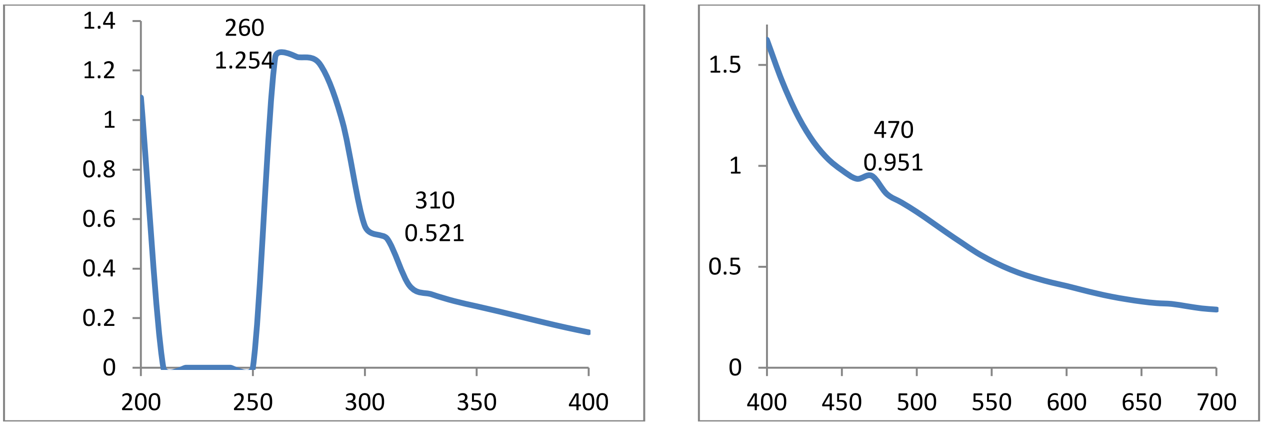
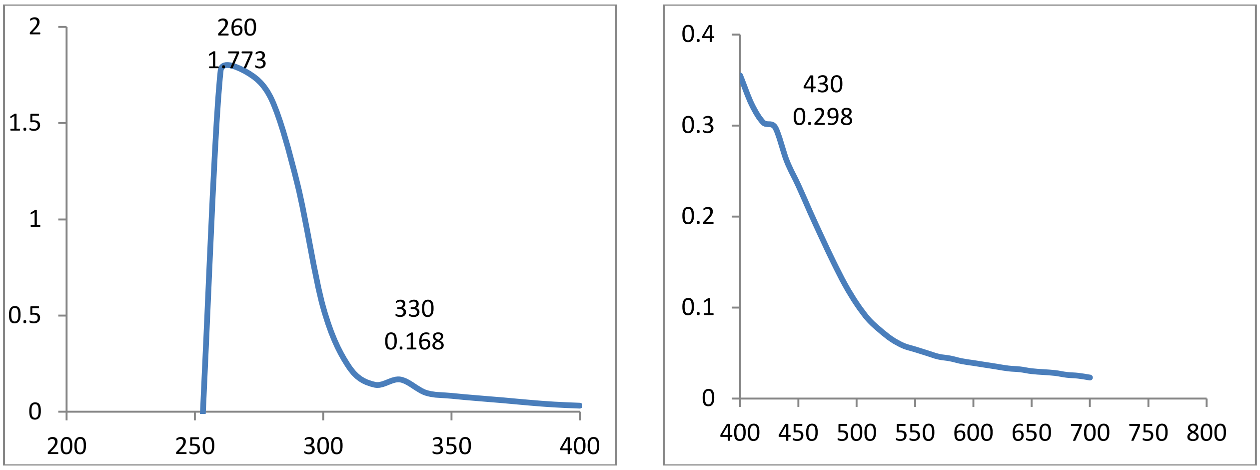


Figure 11: Uv.-Vis. spectrum of bpozs ligand



(a) **(b)**

Figure 12: Electronic spectrum of [Pd(bpozs)$_2$(dppf)]Cl$_2$ complex: (a) UV. (b) Vis. Region



(a) **(b)**

Figure 13: Electronic spectrum of $[Pt(bpozs)_2(dppf)]Cl_2$ complex: (a) UV. (b) Vis. Region

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