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Spectroscopic and Thermal Studies of Some Palladium(II) Complexes with 2-amino-4-(4-substituted phenyl)thiazole Derivatives

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ABSTRACT

Six new complexes of palladium(II) with a general formula $[Pd(L)_2Cl_2]$, where L = 2-amino-4-(4-substituted phenyl)thiazole. The palladium complexes were prepared by the reaction of 2-amino-4-(4-substituted phenyl)thiazole ligands with Bis(benzonitrile)palladium(II) dichloride in chloroform solvent at molar ratio Pd:L=1:2. The resulting complexes were characterized by the magnetic susceptibility, conductivity measurements, infrared, ¹H NMR and the thermo gravimetric analysis. Elemental analyses, spectroscopic and another physical studies of the prepared palladium (II) complexes allowed structures to be proposed. The thermal properties of the prepared complexes indicated the all-decomposition steps and gave an insight about the stability of palladium(II) complexes. The physical analysis indicated that prepared ligands behaved as mono dental, bounding Pd(II) through the nitrogen atoms from the thiazole ring.

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1. Introduction

Palladium complexes with heterocyclic ligands were the subject on intensive studies due to their biological activity (Akpunar, et al., 2021; Mansour, et al., 2021). Many researches have been achieved on the palladium complexes with heterocyclic compounds containing nitrogen as a donor atom such as azines, pyridines and their derivatives. The resulting complexes have been stimulated by their magnetic coupling interactions, magnetic coupling interactions, catalytic, photocatalytic processes and electrochemical properties (Kantchev, et al., 2007; Gaw, et al., 2002).

The heterocyclic ligands that have nitrogen and / or sulphur atoms are of extensive use as building unit in coordination chemistry; where the sulphur being can give more biological activity for the ligands. These types of

compounds are recognized as potent biological molecules with a wide range of activity, and textile dyes too (Das, et al., 2016; Singh, et al., 2002). 2-aminothiazole and its derivatives have been used as building blocks in the synthesis of numerous compounds which have biocides, dyes, fungicides, accelerators for chemical reaction, sulfur drugs. Furthermore, a huge number of 2-aminothiazoles derivatives have been used in pharmaceutical field and are similarly used in the preparation of numerous types of dyes which have been used as synthetic fibers, alongside their action as corrosion inhibitors for protection of mild steel (Mašković, et al., 2018; Khaled & Amin, 2009).

Coordination chemistry of Pd(II), Pt(II) and Ru(II) have found a lot using as antitumor reagents (Alam & Huq, 2016; Alam, et al., 2020). Cis-platin derivatives of some benzimidazole molecules in addition to their corresponding palladium compounds have been characterized by different methods like X-ray structure analysis.

Many palladium complexes have got lots of attention due to the similarity between the coordination chemistry of palladium and platinum. Though the early studies indicated unpromising results obvious with larger toxicity and lower anti-cancer activity for Pd complexes than cis-platin, Lately, higher antitumor activity in Pd complexes together with low toxicity has been done by introduce of bulk substituted organic ligands (Alam & Huq, 2016; Alam, et al., 2020).

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Our interest in the coordination chemistry of heterocyclic ligands containing nitrogen atoms such as 2-hydroxybenzylidene-4-(4-substituted-phenyl)-2-aminothiazole (Aldelfy, et al., 2019) has encouraged us to investigate the reactions of Pd(II) with some selected 2-amino-4-(4-substituted phenyl)thiazole ligands (Scheme 1).

2. Materials and Methods

PdCl₂, Acetophenone, 4-Chloro-acetophenone, 4-Methyl-acetophenone, 4-Methoxy-acetophenone, 4-Bromo-acetophenone, 4-Nitro-acetophenone and thiourea were purchased from Fluka.

2.1. Instruments

The measurements of IR (KBr pellets) were achieved on a Shimadzu 8400S FT-IR spectrophotometer in the range of (4000-500) cm⁻¹. ¹H NMR measurements were carried out on a Bruker MHZ500. Samples were dissolved in deuterated DMSO-d₆ solvent with TMS as internal reference. Thermogravimetric analyses (TG and DTG) have been carried out under inner atmosphere of nitrogen gas with a heating rate of 10 °C/min. by using a SDT Q600 V20.9 Build 20 thermal analyzer. The prepared Palladium complexes were characterized by using elemental analysis (Perkin-Elmer 2400 CHN instrument).

2.1.1. Preparation of 2-Amino 4-(4-substitutedphenyl) thiazole (general procedure)

2-Amino 4-(4-substitutedphenyl)thiazole compounds were prepared according to literature (Dodson & King, 1945), Iodine (15 mmol) dissolved in 10 ml ethanol was added to a mixture of thiourea (30 mmol) and acetophenone derivatives (15 mmol) dissolved in 20 ml ethanol, the resulting mixture was heated to reflux overnight. After that, it was diluted with water and then heated till the biggest amount of the solid has been dissolved. Then the resulting solution was cooled, filtered and it alkaline by using concentrated solution of NH₄OH. The resulting precipitated of 2-amino-4-phenylthiazole was separated and recrystallized from ethanol to constant melting point.

2.1.2. Preparation of Bis (benzonitrile) palladium (II) Dichloride

This complex PdCl₂(PhCN)₂, was prepared as described in the literature (Olmstead, et al., 2000). 0.4 gram of palladium(II) dichloride was dissolved in 2 mL of benzonitrile

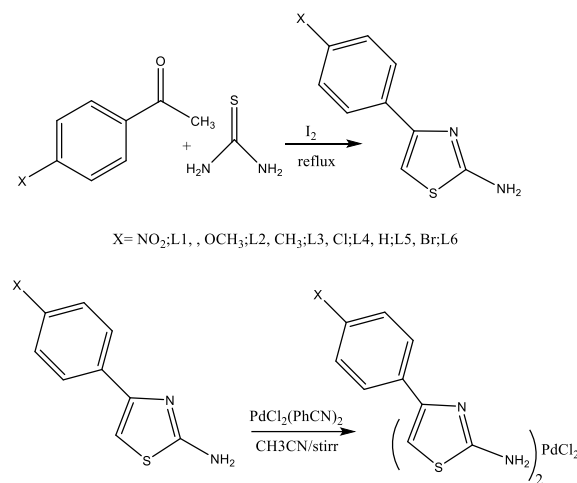
solvent. The resulting solution (dark orange) has been transferred after that to a 10 mL beaker and then heated for an hour at 100 °C. The resulting solution was partly evaporated and then cooled to give dark red crystals. The resulting crystals were collected by filtration and washed with ether.

2.1.3. Preparation of complexes of palladium with 2-Amino 4-(4-substitutedphenyl) thiazole

2-Amino 4-(4-substitutedphenyl) thiazole (2 mmol) dissolved in 25 ml of chloroform was added gradually with continuous stirring to solution of PdCl₂(PhCN)₂ (1 mmol) dissolved in 20 ml of chloroform. The resulting solution was left under reflux for four hours. The resulting mixture was cooled and the precipitate was collected by filtration and washed with ethanol.

3. Results & Discussion

Six thiazole ligands have been prepared by the reaction of thiourea and acetophenone derivatives, then the resulting ligands of 2-Amino 4-(4-substitutedphenyl)thiazole have been used to synthesized new palladium complexes by the reaction of these ligands with Bis(benzonitrile)palladium(II) Dichloride in chloroform solvent; see Scheme 1.



Scheme 1. The synthetic route to complexes of palladium with 2-Amino 4-(4-substitutedphenyl) thiazole derivatives.

Table 1

Elemental analysis, color, yield. Magnetic susceptibility and conductivity data for the palladium complexes

Complex	Color	Yield (%)	Calcul (Found)			m.p	μ_{eff} (B.M)	$\Delta m = \text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$
			C	H	N			
[Pd(L ₁) ₂ Cl ₂]	Dark yellow	51	34.80 (34.34)	2.26 (2.26)	13.57 (13.59)	296- 298	0.76	0.270
[Pd(L ₂) ₂ Cl ₂]	Brown	48	40.74 (40.98)	3.39 (3.24)	9.50 (9.56)	270- 272	0.82	0.490
[Pd(L ₃) ₂ Cl ₂]	Brown	50	43.64 (43.68)	3.60 (3.60)	10.44 (10.92)	Dec.> 300	0.79	0.168
[Pd(L ₄) ₂ Cl ₂]	Brown	45	36.16 (36.22)	2.33 (2.27)	9.34 (9.27)	Dec. 200	0.83	3.927
[Pd(L ₅) ₂ Cl ₂]	Brown	54	40.83 (40.97)	3.02 (3.25)	10.58 (10.59)	Dec.> 300	0.78	0.144
[Pd(L ₆) ₂ Cl ₂]	Brown	52	31.44 (31.65)	2.77 (2.90)	8.15 (8.43)	Dec.> 300	0.77	0.060

The physical and elemental analysis data of the prepared complexes are shown in Table 1. As shown in this table, there were a good agreement between theoretical and calculated results. Another data like color, yield and melting points of the prepared complexes are listed also in Table 1.

The magnetic susceptibility of the prepared palladium complexes was calculated at room temperature 298K, where the complexes showed diamagnetic properties. The observed magnetic moments of palladium complexes were less than one confirming the diamagnetic properties of the square planar complexes and hybridization dsp^2 , see Table 1. To investigate the behavior of palladium complexes in solution, their conductivity was studied in DMSO solvent. The conductivity of the studied complexes indicate that are non-electrolyte, where the molar conductivity values range between (3.93-0.060 $\text{ohm}^{-1}\text{cm}^2\text{mol}^{-1}$) at a concentration of ($1 \times 10^{-3}\text{M}$). Thus, the prepared palladium complexes have not a counter ion outside the coordination sphere, see Table 1.

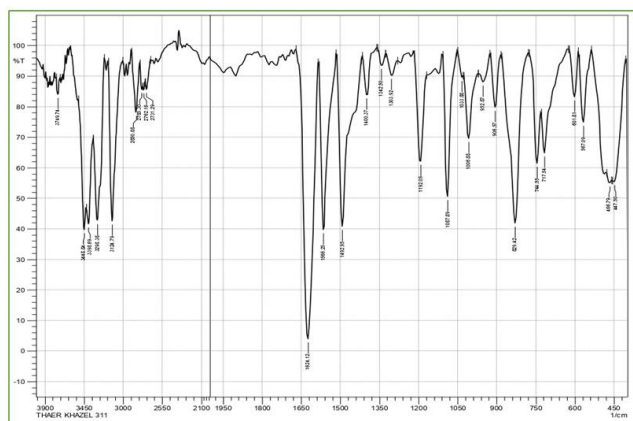


Fig. 1. IR spectrum of 2-Amino 4-(4-Chloro-phenyl)thiazole ligand

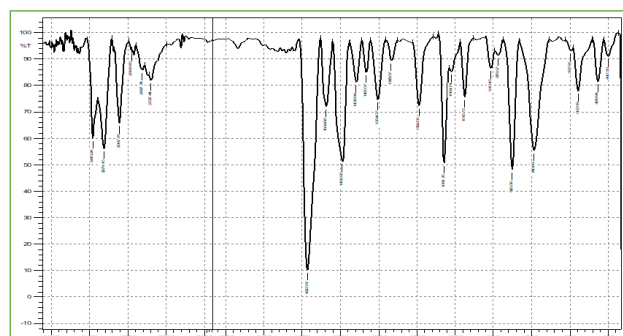


Fig. 2. IR spectrum of palladium complexes of 2-Amino 4-(4-Chloro-phenyl)thiazole

The infra-red spectra of the prepared complexes are presented in Table 2, see also Figure 1 and Figure 2. The IR spectra showed the characteristic bands of the ligands and complexes due to functional groups like NH, C=N, C=C and C-S with the suitable shifts, see Table 2. It can be seen clearly from the palladium complexes spectra that the bands characterizing the stretching vibration of NH_2 group did not shift towards low frequencies. This means that there is not bound between NH_2 group and Pd metal. On the other hand, the palladium complexes spectra indicated the stretching vibrations of the C=N group between 1600-1647

cm^{-1} , these bands were shifted towards lower frequencies with 39-20 cm^{-1} , in comparison with the free ligands, because of complex formation between the nitrogen atom of thiazole ring and the palladium metal. The stretching vibrations of C=N bond in free ligands indicated higher frequencies due to the redistribution of the electronic density as a result of the participation of the C=N group. Furthermore, new band in the IR spectra have been observed in the spectra of palladium complexes which can be assigned to $\nu(\text{Pd-N})$ in the 474-582 cm^{-1} range.

The ^1H NMR spectra of the prepared palladium complexes are listed in Table 2, see also Figure 3 and Figure 4. The ^1H NMR spectra of complexes indicate signals due to the different proton nuclei in thiazole ligands. Generally, as shown in this table, the signals of the protons were significantly shifted down field, thus confirming the coordination of palladium through the nitrogen atom of thiazole ring. The signals shift of protons of methyl and methoxy group did not indicate noticeable change.

The thermo gravimetric analysis (TGA) and differential thermo gravimetric decomposition (DTG) for palladium complexes have been achieved. The data of thermo gravimetric analyses of palladium complexes are shown in Table 3, see also Figure 4 and Figure 5. Thermogravimetric analyses (TG and DTG) have been carried out under inner atmosphere of nitrogen gas with a heating rate of 10 $^\circ\text{C}/\text{min}$.

The TGA curve of $[\text{Pd}(\text{L}_1)_2\text{Cl}_2]$ indicate three phases of decomposition steps for complex. The first step inside the temperature range 99-340 $^\circ\text{C}$ ($\text{DTG}_{\text{max}}=290$ $^\circ\text{C}$) is the moisture and a part of ligand ($\text{C}_8\text{H}_4\text{S}_2$) which reflected mass losses of 26.52 % (cal. 26.55%).

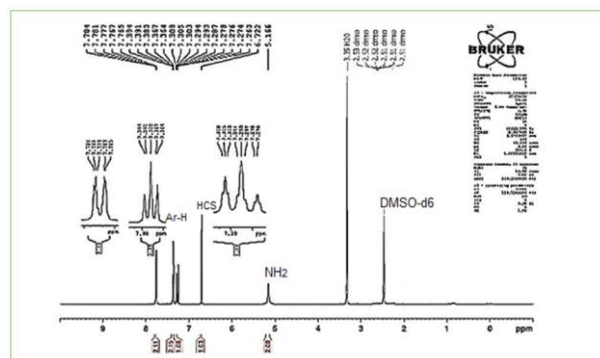


Fig. 3. ^1H NMR of spectrum of 2-Amino 4-(phenyl)thiazole ligand

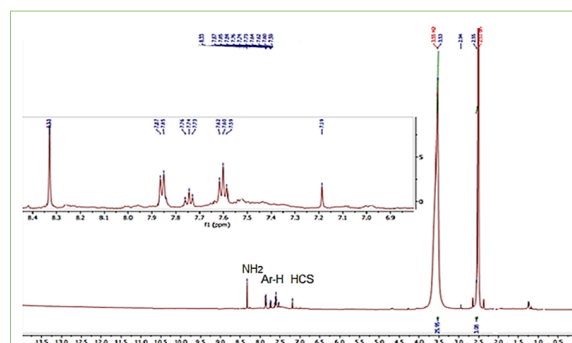


Fig. 4. ^1H NMR of spectrum of palladium complex of 2-Amino 4-(phenyl)thiazole

Table 2Important IR and ¹H NMR data for 2-Amino 4-(4-substitutedphenyl) thiazole derivatives ligands and their palladium complex

Comp.	IR data (cm ⁻¹)				¹ H NMR DATA (ppm)
	ν(NH)	ν(C=N)	ν(C=C)	ν(C-S)	
L1	3402,3309 (m)	1639	1593	1107,1037	7.2 (s,2H,NH ₂) 7.4 (s, ¹ H, ring thiazole) 8.05-8.20 (m,4H,Ar-H) (8.04 - 8.26) (m,8H,Ar-H) (8.38 - 8.40)(s,4H,NH ₂) 7.74-7.72 (s,2H, ring thiazole)
[Pd(L ₁) ₂ Cl ₂]	3422,3325	1600	1512	1111,1065	7.01 (s ,2H,NH ₂) 7.4 (s, ¹ H, ring thiazole) 6.91-7.74 (m,4H,Ar-H) 3.7 (s, 3H, OCH ₃) 7.60 - 6.96 (m,8H,Ar-H) 7.83 (s,4H,NH ₂) , 6.86 -6.90 (s,2H,ring thiazole) 3.8(s,6H,OCH ₃) 8.3 (s,2H,NH ₂)
L2	3437,3271 (m)	1624	1531	1111,1033	7.4 (s, ¹ H, ring thiazole) 7.6-7.9(m,4H,Ar-H) 2.3 (s,3H,CH ₃) 7.6 -7.85 (m,8H,Ar-H) 7.76 (s,4H,NH ₂)
[Pd(L ₂) ₂ Cl ₂]	3421,3298	1604	1512	1180,1022	7.25(s,2H,ring thiazole) 2.3(s,6H,CH ₃) 4.56 (s,2H,NH ₂) 7.55 (s, ¹ H,ring thiazole) 7.4 - 7.7 (m,4H,Ar-H) 7.49 -7.80 (m,8H,Ar-H) 7.86-7.88(s, 4H,NH ₂) 7.18 (s,2H, ring thiazole) 5.16(s,2H,NH ₂)
L3	3417,3302 (m)	1627	1519	1033,1130	6.72(s, ¹ H,ring thiazole) 7.26 -7.78(m,5H,Ar-H) 7.59 -7.87 (m,8H,Ar-H) 8.30 - 8.33 (s,4H,NH ₂) 7.08 (s, ² H, ring thiazole) 7.9(s,2H,NH ₂)
[Pd(L ₃) ₂ Cl ₂]	3417,3286	1647	1519	1184,1045	7.24 (s, ¹ H,ring thiazole) 7.63-7.75 (m,4H,Ar-H) (7.86 - 7.51) (m,8H,Ar-H) 7.88(S,4H,NH ₂) (7.18)(S, ² H,ring thiazole)
L4	3448,3298 (m)	1624	1566	1192,1087	
[Pd(L ₄) ₂ Cl ₂]	3410,3279	1644	1554	1192,1091	
L5	3437,3259 (m)	1600	1523	1072,1033	
[Pd(L ₅) ₂ Cl ₂]	3448,	1620	1519	1176,1030	
L6	3441,3352 (m)	16027	1566	1037,1107	
[Pd(L ₆) ₂ Cl ₂]	3421,3302	1608	1519	1176,1072	

The second decomposition step was within the 350-475 °C range (DTGmax=396 °C) agrees to the loss two Cl ion as two HCl molecules with percentage of 11.14% (cal. 11.44%). The last step of decomposition was within 475- 800 °C range, (DTGmax= 625 °C) is the loss of C₄H₅N₄ fragment of ligand with mass losses of 17.73% (cal.17.60%). There was a remaining residue of palladium complexes as carbon and metal oxide due to decomposition.

The TGA curve of [Pd(L₂)₂Cl₂] indicate two phases of decomposition steps for complex. The first step was at 30-310 °C range, (DTG max= 277 °C) with losing percentage of 18.92% (cal. 18.16%) due to losing of C₇H₇O fragment. The second step was at 310-800 °C range, (DTGmax= 475 °C) with losing percentage of 27.89% (cal. 27.84%) due to losing of the organic fragment of C₈H₄S₂.

The TGA curve of [Pd(L₃)₂Cl₂] indicate three phases of decomposition steps for complex. The first step was at 25-380 °C range, (DTGmax= 284 °C) with losing percentage of 28.00% (28.02%) due to the losing of C₈H₄N₄ fragment. On the other hand, the second step carried out at 380-580 °C range, (DTGmax= 457 °C) with losing percentage circa about 9.96 (cal. 9.86) due to losing of C₄H₇ fragment. The last third step was occurred at 580-800 °C range, (DTGmax= 710 °C) with losing percentage of 9.21% (cal.9.147) due to losing CH₇S fragment.

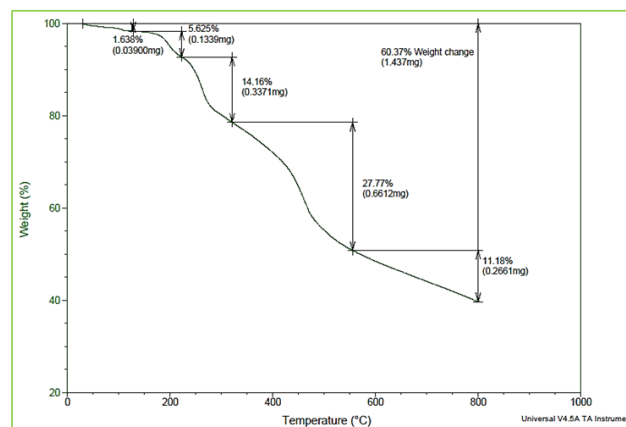


Fig. 5. TGA curve of palladium complexes of 2-Amino 4-(4-Chloro-phenyl)thiazole

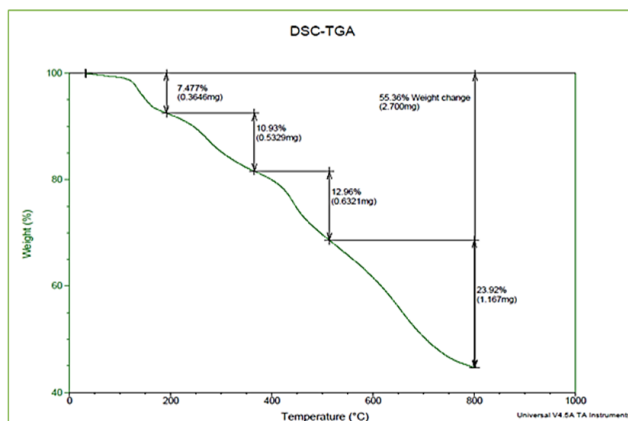


Fig. 6. TGA curve of palladium complex of 2-Amino 4-(phenyl) thiazole

The TGA curve of $[\text{Pd}(\text{L}_4)_2\text{Cl}_2]$ exhibited five phases of decomposition steps of complex. The first decomposition step occurred in the range 30-125 °C, (DTGmax=103 °C) with a net weight loss of 1.63% (cal. 1.66%), which corresponds to the elimination of five H_2 . The second step occurred in the 125-220 °C range, (DTGmax= 197 °C) with losing percentage of 5.62% (cal.6.01%) due to losing of two chloride ions as two HCl molecules. The third step carried out at 220-320 °C range, (DTGmax= 260 °C) with losing percentage of 14.16% (cal. 14.02%) due to losing of $\text{C}_4\text{H}_4\text{S}$ fragment. The fourth step carried out at 320-550 °C range, (DTGmax= 459 °C) with losing percentage of 27.77% (cal. 27.87%) due to losing of C_{10}HNS fragment. The final fifth step occurred at 550-800 °C range, (DTGmax= 600 °C) with losing percentage of 11.18 (cal.11.18%) due to losing $\text{C}_3\text{H}_3\text{N}_2$ fragment.

Table 3

Thermo analytical results (TG, DTG) of palladium complexes with with 2-Amino 4-(4-substitutedphenyl) thiazole derivatives

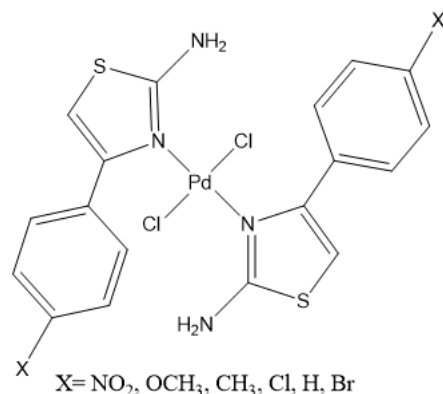
Assignment	%		DTG max °C	TG Range °C	Stage	Complex
	Cal.	Mass loss found				
$\text{C}_8\text{H}_4\text{S}_2$	26.5	26.52	290	100-340	I	$[\text{Pd}(\text{L}_1)_2\text{Cl}_2]$
2HCl	11.44	11.14	396	350-475	II	
$\text{C}_4\text{H}_5\text{N}_4$	17.6	17.73	625	475-800	III	
$\text{C}_4\text{H}_7\text{N}_4$	18.84	18.92	277	30-310	I	$[\text{Pd}(\text{L}_2)_2\text{Cl}_2]$
$\text{C}_8\text{H}_4\text{S}_2$	27.84	27.89	475	310-800	II	
$\text{C}_8\text{H}_4\text{N}_4$	28.02	28	284	25-380	I	$[\text{Pd}(\text{L}_3)_2\text{Cl}_2]$
C_4H_7	9.86	9.95	457	380-580	II	
CH_7S	9.147	9.21	710	580-800	III	
5H_2	1.66	1.63	103	30-125	I	$[\text{Pd}(\text{L}_4)_2\text{Cl}_2]$
HCl	6.01	5.62	197	125-220	II	
$\text{C}_4\text{H}_4\text{S}$	14.02	14.16	260	220-320	III	
C_{10}HNS	27.87	27.77	459	320-550	IV	$[\text{Pd}(\text{L}_5)_2\text{Cl}_2]$
$\text{C}_3\text{H}_3\text{N}_2$	11.18	11.18	600	550-800	V	
$2\text{H}_2\text{O}$	6.8	7.47	138	30-190	I	
$\text{C}_3\text{H}_9\text{N}$	11.15	10.93	271	190-360	II	$[\text{Pd}(\text{L}_6)_2\text{Cl}_2]$
2HCl	13.23	12.96	442	360-510	III	
$\text{C}_4\text{H}_3\text{N}_3\text{S}$	23.62	23.92	654	510-800	IV	
N_2	4.07	4.246	136	30-160	I	$[\text{Pd}(\text{L}_6)_2\text{Cl}_2]$
HCl	5.16	5.429	237	175-250	II	
C_7N_2	16.3	16.29	305	250-400	III	
HBr	11.49	11.3	454	410-510	IV	$[\text{Pd}(\text{L}_6)_2\text{Cl}_2]$
$\text{C}_4\text{H}_2\text{S}_2$	16.59	16.64	570	510-800	V	

The TGA curve of $[\text{Pd}(\text{L}_5)_2\text{Cl}_2]$ exhibited four phases of decomposition steps of complex. The first decomposition step occurred in the range 30-190 °C, (DTGmax=138 °C) with a net weight loss of 7.48% (cal. 6.80%), which corresponds to the losing of two H_2O molecules. The second step occurred in the 190-360 °C range, (DTGmax= 271 °C) with losing percentage of 10.93% (cal.11.15%) due to losing of $\text{C}_3\text{H}_9\text{N}$. The third step carried out at 360-510 °C range, (DTGmax= 442 °C) with losing percentage of 12.96% (cal. 13.23%) due to losing of two chloride ions as two HCl molecules. While, the fourth step carried out at 510-800 °C range, (DTGmax= 654 °C) with losing percentage of 23.92% (cal. 23.62%) due to losing of $\text{C}_4\text{H}_3\text{N}_3\text{S}$ fragment.

Finally, the TGA curve of $[\text{Pd}(\text{L}_6)_2\text{Cl}_2]$ complex showed fifth phases of decomposition steps of complex. The first decomposition step occurred in the range 30-160 °C, (DTGmax=136 °C) with a net weight loss of 4.25% (cal. 4.07%), which corresponds to the losing of nitrogen molecule. The second step occurred in the 175-250 °C range, (DTGmax= 237 °C) with losing percentage of 5.43% (cal.5.16%) due to losing of chloride ion as HCl molecule. The third step carried out at 250-400 °C range, (DTGmax= 305 °C) with losing percentage of 16.29% (cal. 16.30%) due

to losing of C_7N_2 fragment. The fourth step carried out at 410-510 °C range, (DTGmax= 454 °C) with losing percentage of 11.30% (cal. 11.49%) due to losing of bromide ion as HBr molecules. The final fifth step occurred at 510-800 °C range, (DTGmax= 570 °C) with losing percentage of 16.64% (cal.16.59%) due to losing $\text{C}_4\text{H}_2\text{S}_2$ fragment.

All in all, based on the previous analysis, the magnetic susceptibility, conductivity measurements, infrared, proton NMR and the thermo gravimetric analysis, the suggested geometry of the prepared palladium complexes is square planar as shown in Scheme 2.



Scheme. 2. The suggested structure of palladium complexes with 2-Amino-4-(4-substitutedphenyl) thiazole derivatives.

4. Conclusions

2-amino-4-(4-substituted phenyl)thiazole derivatives were prepared and the analog palladium(II) complexes were prepared by reaction of two mole of ligand and one mole of palladium dichloride. The resulting palladium(II) complexes were characterized by different methods like elemental analyses magnetic susceptibility, conductivity measurements, infrared, ^1H NMR and the thermo gravimetric analysis. The previous analysis indicated that prepared ligands behaved as monodentate, bounding Pd(II) through the nitrogen atoms from the thiazole ring. The thermal properties of the prepared complexes indicated the all-decomposition steps and gave an insight about the stability of palladium(II) complexes.

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Competing Interests

The authors have declared that no competing interests exist.

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