SYNTHESIS AND CHARACTERIZATION OF NEW COMPLEX SALTS OF SOME TRANSITION METALS WITH CARBAZOLIUM AND PHOSPHONIUM SALTS

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Abstract. *This study manufactured and described new organic salts containing (benzylcarabazoliumchloride) and (benzyltriphenylphosphonium) ligands, which were prepared and synthesized and then interacted with some transition metals (Cr(III), Co(II), Ni(II), Cu(II), and Zn(II)). Obtained complexes were characterized by element analysis, FTIR (Fourier change infrared) and UV-vis spectral investigation, magnetic measurement, conduction measurement, and* ^{1}*H-NMR for the ligands. The stoichiometry of the complexes was determined based on input to be (1:2) and (1:3) for metal-to-ligand ratio. The conductivity results suggest that these complexes are electrolytes. Furthermore, the electronic spectra and magnetic measurements provide insights into the nature of these composites.* $[bCar]_2[MCl_4]$ *and* $[bTpp]_2[MCl_4]$ *have a tetrahedral environment, while* $[bCar]_3[CrCl_6]$ *and* $[bTpp]_3[CrCl_6]$ *have an octahedral environment around the metal ions.*

Keywords: *organic salts, transition metal, octahedral, tetrahedral, metal complexes*
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1. Introduction

Carbazole is one of the three-membered aromatic organic compounds. It consists of two benzene rings fused to a pentagonal ring containing a heterogeneous nitrogen atom._The structure of carbazole is essentially based on the structure of the known indole, but in it a second benzene ring is incorporated. It has been found that carbazole is available in the anthracene portion of coal tar and is also called dibenzopyrrole and benzoindole [1].

Carbazole and its derivatives represent great importance in aromatic heterocyclic compounds. Such compounds contain electronic and charge transfer properties, which facilitates the process of introducing different functional groups into the carbazole ring [2].

Many researchers were able to find various methods for preparing carbazole and its derivatives, including the reduction method using palladium catalyst. Rago et al. developed a method for preparing N-acetyl carbazole using (N-acetyl-2-aminobiphenyl) as a primary raw material through the reduction method and using palladium catalysts [3].

Ionic liquids are distinct as salts that have a melting matter lower than boiling point of water [4]. Their low melting point can be explained by the weak attraction between negative ions and positive ions. The strength of this attraction depends on the composition and nature of the negative and positive ions, with little similarity in the composition of the ions. As it can reduce the force of attraction between them, ionic liquids are also called molten salts [5].

Ionic liquids were called green solvents because of their non-volatility, as they can be purified and recovered after use under experimental conditions of rarefied pressure and high temperature [6]. On this basis, they were classified as environmentally friendly solvents when compared to non-organic solvents and volatile substances, which often end up at the end of the reaction as waste in the atmosphere or groundwater [7, 8]. Through studies, it has been observed that solvents consume about 60% of the total energy used in the production of pharmaceutical preparations, in addition to their role in the emission of 50% of greenhouse gases after treatment [9]. Therefore, the choice of solvents should be carefully considered to improve reaction conditions and according to the principles of green chemistry [10].

Over the past years, researchers have increased their interest in complexes of transition element halides and metal tetrahalides with different types of positive ions. This importance is evident in theory, structure, magnetic and spectroscopic properties, and also interest in ionic complexes that have uses as catalysts or separation agents in some industrial uses [11, 12].

Nabeel et al. synthesized salts with compositions $[RA]_2[MCl_4]$ [R=carbazole, indole, or pyrrole; A=H or $PhCH_2$; M=Pd(II) or Pt(II)] and measured their molar conductance. Studied the IR and UV/Vis spectra along with the magnetic properties of these compounds. The UV/Vis spectral analysis and magnetic measurements indicated that the anion composite exhibits a plain conformation [13].

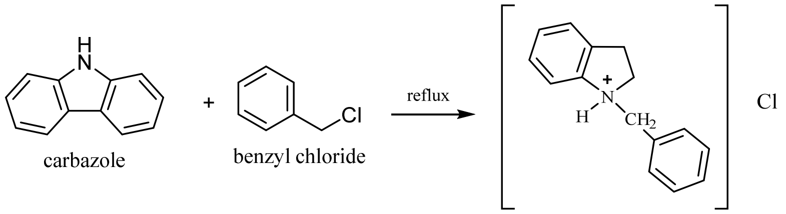
2. Experimental part

Material and Methods. The initial materials used were top quality, sourced from Fluka and BDH without any modifications. An elemental analyzer model 2400 Perkin Elmer was utilized to examine both the ligands and their compounds. The metal content was determined using a Shimadzu AA670 spectrophotometer. Infrared spectra, within the 400–4000 cm^{-1} range, were captured using an FT IR Brucker Tensor 27Co spectrophotometer. The electrical spectrum at room temperature was recorded with a Shimadzu UV Vis photographing UV1600 spectrophotometer. These analyses were conducted on concentrations in DMSO of 103 M. Measurements on the solid state at 25°C were carried out utilizing the Faraday method and Bruker BM6 apparatus. The solubility of $K_2[PtCl_4]$, $PdCl_2$ is acetone. Conductivities were measured with a conductivity meter model PCM3 Jenway for analysis.

Preparing of Organic salts

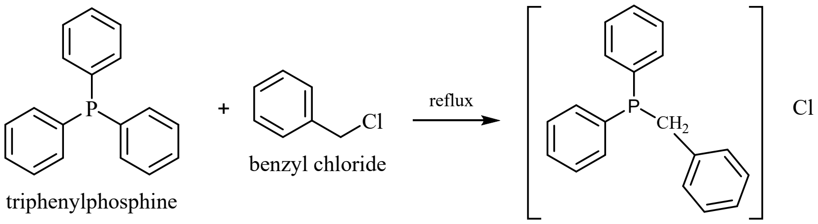
1) benzyl carbazolium chloride (bCar)Cl.

A carbazole (1.67g, 0.01 mole) in dioxin (20 ml) was added to a solution of benzyl chloride (1.26 g, 0.01 mole) in dioxin (20 ml). Addition was continued for 1 hour. The reaction combination was refluxed for 4 h and then left to stand at room temperature. The ashen solid thus formed was filtered off, washed twice with dioxin (10 ml), and then dried in a vacuum.



(bCar)Cl

Scheme 1. Preparation of the ligand (bCar)Cl



(bTpp)Cl

Scheme 2. Preparation of the ligand (bTpp)Cl

2) Benzyl triphenyl phosphonium (bTpp)Cl. A solution of triphenylphosphine (2.62 g, 0.01 mole) in ethanol (20 ml) was added to a solution of benzyl chloride (1.26 g, 0.01

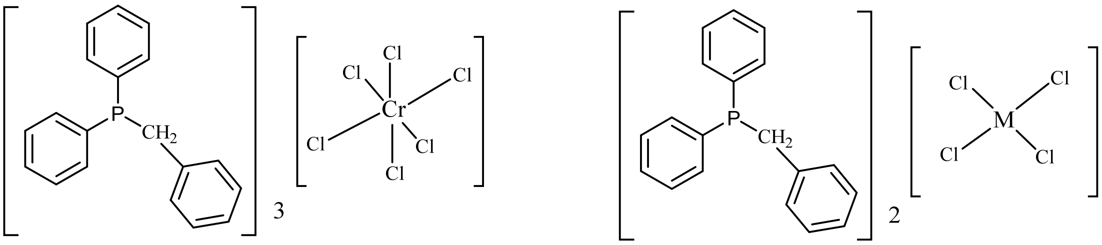
mole) in dioxin (20 ml). Addition was continued for 30 min. The reaction mixture was flowing for 2 h and then left to stand at room temperature. The white solid thus formed was filtered off, washed twice with dioxin (10 ml), and then dried in a vacuum.

Preparation of complex salts $[L]_3[CrCl_6]$ (L = (bCar)Cl, (bTpp)Cl). A stirred solution of the ligand (L) ((bCar)Cl = 0.89 g, 0.003 mole; (bTpp)Cl = 0.11g, 0.003 mole). A mixture of ethanol (10 ml) and chromium chloride (0.266 g, 0.001 mole) in ethanol (10 ml) was heated under reflux for three hours. The resulting combination was washed intermittently with ethanol and diethyl before being left to settle for 24 hr. at room heat. Subsequently it was dried in a vacuum for a few hours.

Preparation of complex salts $[L]_2[MCl_4]$ (L=(bCar)Cl, (bTpp)Cl, M= Co(II), Ni(II), Cu(II), Zn (II)). 0.23 g (0.001 mole) of $CoCl_2 \cdot 4H_2O$ was dissolved in 10 ml of ethanol, then added to a solution of ligand (L) ((bCar)Cl = 0.58 g, 0.002 mole; (bTpp)Cl = 0.77g, 0.002 mole) in 10 ml of ethanol; the mixture was stirred under reflux for 2 hr. The precipitate that formed was filtered off, washed with diethyl ether, and dried in vacuum for several hours. The same procedure was used to prepare other complexes by using the stoichiometric quantities of other metal salts as shown in Table 1. The structures of synthesized complexes are presented in Fig. 1.

Table 1. The weight of (0.001 mole) metal salts used

Metal salts	Weight
$NiCl_2 \cdot 6H_2O$	0.237
$CuCl_2 \cdot 2H_2O$	0.170
$ZnCl_2 \cdot 2H_2O$	0.172



$[bTpp]_3[CrCl_6]$

$[bTpp]_2[MCl_4]$

M= Co(II), Ni(II), Cu(II), Zn (II)

Fig. 1. The structure of synthesized complexes

3. Results and discussion

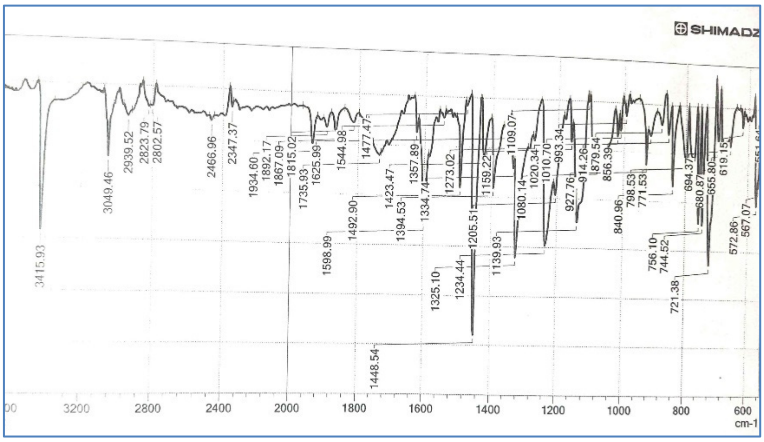
The compositions of complexes were identified based on the analysis presented in Table 2. The molar conductance data in DMSO suggested that these complexes exhibited conducting properties. The chlorine content was determined using the precipitation method (Volhard). The obtained percentages matched the expectations, confirming the accuracy of the chemical formulas listed in Table 1. The elements C, H, N, and S present in both the ligand and the formulated complexes were tested at a research laboratory located at Ordu University in Ordu, Turkey.

Infrared spectra of ligands and complexes (Fig. 2). The ligand ((bCar)Cl): the infrared spectra show stretching absorption bands at 3415 cm^{-1} linked to the (N+H) group [15]. It was also observed that an absorption band appeared at (1625 cm^{-1}) due to stretching of the group (C=N), and this band was shifted towards a higher or lower frequency when the metal was bound to the ligand [16]. While absorption bands appeared at (3049cm^{-1}) and (2823cm^{-1}) belonging to the aromatic and aliphatic (C-H) bands, respectively, it was noted that these bands were shifted towards higher or lower frequencies when the metal was bound to the ligand, indicating that the reaction had occurred [17].

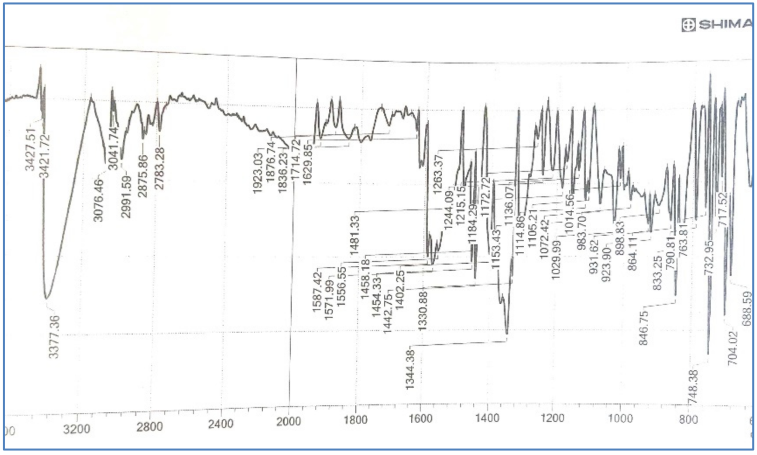
While the ligand ((bTpp)Cl) shows an absorption band at (3076 cm^{-1}) belonging to the aromatic (C-H) [18], an absorption band also appears at (2991cm^{-1}) belonging to the aliphatic (C-H) [19]. It was observed that when the metal is bound to the ligand, the absorption bands shift either up or down, and this indicates the formation of complexes of prepared organic salts [20].

Table 2. Electronic and Infrared spectral data of ligands and complexes

No.	Structure	N^+-H	ν(C=N)	ν(C-H) aromatic	ν(C-H) aliphatic
	(bCar)Cl	3415	1625	3049	2823
	(bTpp)Cl	---	---	3076	2991
1	$[bCar]_3[CrCl_6]$	3659	1613	3190	2841
2	$[bCar]_2[CoCl_4]$	3276	1621	3022	2798
3	$[bCar]_2[NiCl_4]$	3520	1634	3182	2737
4	$[bCar]_2[CuCl_4]$	3415	1624	3031	2374
5	$[bCar]_2[ZnCl_4]$	3454	1624	3067	2834
6	$[bTpp]_3[CrCl_6]$	---	---	3187	2891
7	$[bTpp]_2[CoCl_4]$	---	---	3064	2889
8	$[bTpp]_2[NiCl_4]$	---	---	3186	2856
9	$[bTpp]_2[CuCl_4]$	---	---	3017	2968
10	$[bTpp]_2[ZnCl_4]$	---	---	3059	2941



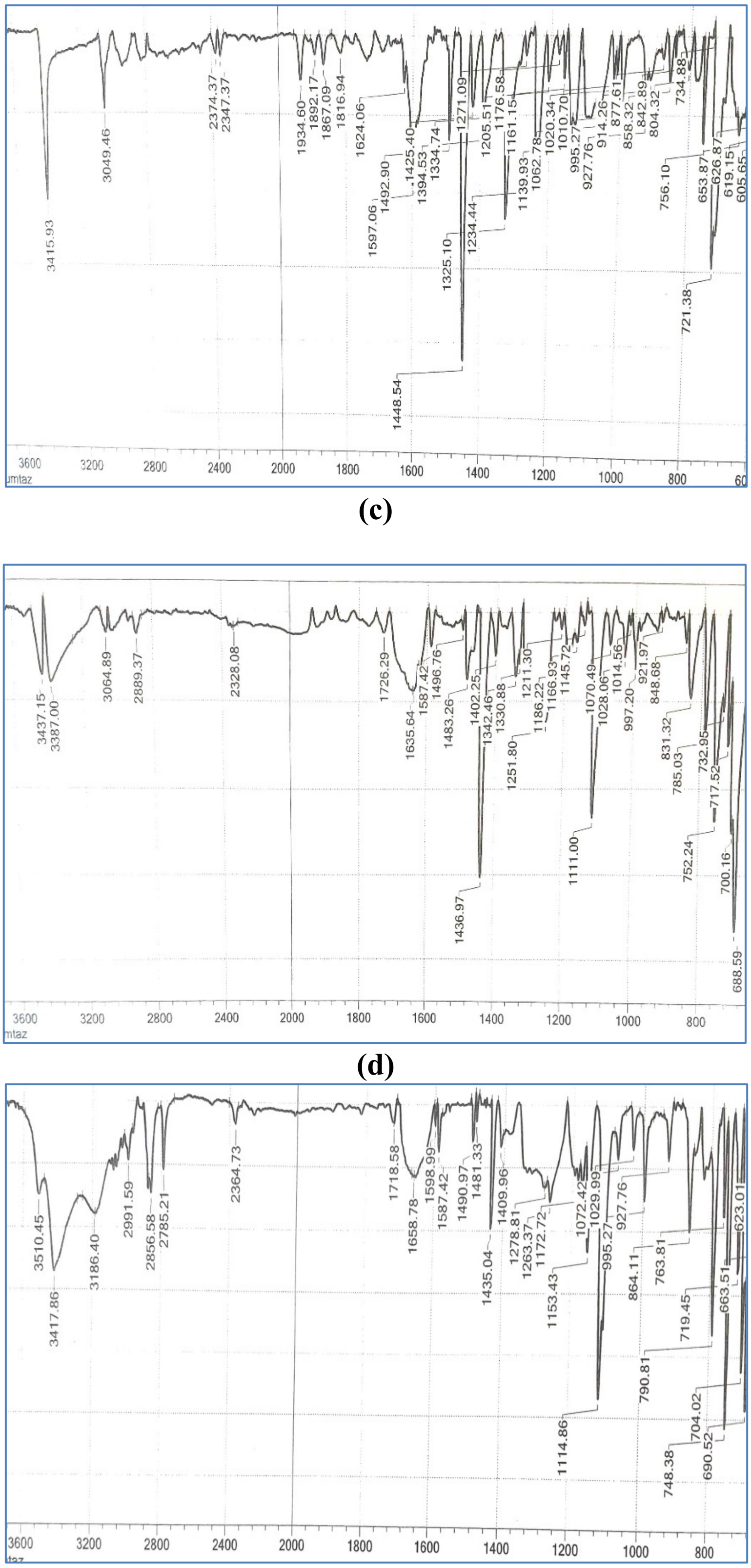
(a)



(b)

The IR spectrum of the complex salts (1-5) show bands at (3276-3659 cm^{-1}) were related to return to a group (N^+H). Compared with the ligand, it was found that this value moved towards a frequency higher or lower than the ligand, indicating that the reaction had occurred

[21]. An absorption band with limits (3017-3187 cm^{-1}) also appeared, dating back to the stretching frequency of the string (C-H) aromatic bond, and this indicates that the reaction took place [22]. The ligands and their composite electrical spectral bands are listed in Table 2.



(c)

(d)

(e)

Fig. 2. IR-Spectra of (a) (bCar)Cl, (b) (bTpp)Cl, (c) [bCar]$_2$[CuCl$_4$], (d)[bTpp]$_2$[CoCl$_4$], and (e) [bTpp]$_2$[NiCl$_4$]

^{1}H-NMR Spectra (Fig. 3). The ligands ^{1}H-NMR proton nuclear magnetic reflection spectrum was analyzed using DMSO-d6. In the spectrum of the ligand ((bCar)Cl), there was a peak observed at δ = 11.26 ppm corresponding to the (NH) protons, and another peak at δ = 7.14-7.92 ppm related to the protons of two rings. Also showed band at δ = (8.10-8.12 ppm) back to the protons of the -(CH_2), while the ligand ((bTpp)Cl) showed bands a band at δ = (7.23-7.87ppm) back to the protons of four aromatic rings [23], and another band at δ = (8.02-8.03 ppm) back to the protons of the -(CH_2) [24].

Table 3. The ^{1}H-NMR data of the ligand

	ς(ppm)	Assignment
(bCar)Cl	11.26	NH(s,1H)
	8.10-8.12	CH_2(s,2H)
	7.14-7.92	2phenyl ring (m,10H)
(bTpp)Cl	8.02-8.03	CH_2 (s,2H)
	7.23-7.87	4phenyl ring (m,10H)

s=singlets, m=multiplets

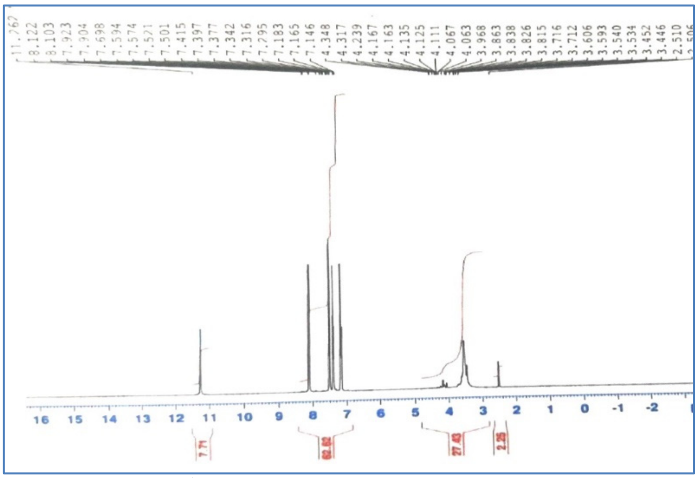


Fig. 3. ^{1}H-NMR for the ligand(bTpp)Cl

Magnetic measurement and the electronic spectra. The magnetic moments provide information about the magnetic properties of the complexes. Higher magnetic moments generally indicate the existence of unpaired electrons, suggesting paramagnetic behavior, whereas lower moments indicate diamagnetic behavior. For instance, the Cr(II) complexes (1, 6) have an octahedral geometry with a magnetic moment of (3.85, 3.79 B.M.), respectively [25]. Electronic spectra showing absorption bands in the region (20688, 32263 cm^{-1}) respectively return to (ν_3) and indicate the transition (^4A_2g--$^4T_1g(p)$); other absorption bands indicate the transition (^4A_2g--$^4T_1g(F)$) within the boundaries of (23781, 2376 cm^{-1}) respectively dating back to (ν_2)._As for the (ν_1) bands returning to the (^4A_2g--$^4T_2g(F)$) transition, they were within the limits of (15643, 14768 cm^{-1}) respectively, and these bands correspond to hexagonal octahedral chromium complexes [26].

The Co(II) complexes (2 and 7) have a tetrahedral geometry, with a magnetic moment of (4.03, 4.89 B.M.) respectively and electronic spectra showing a splitting band due to Jahn-Teller distortion [27].

The metallic ion of Ni (II) complexes (3 and 8) exhibits a magnetic moment of (3.96, 3.32 B.M.) severally. The electronic spectrum showed bands at (14690, 14588 cm^{-1}) and this band indicates a tetrahedral arrangement around the nickel [28].

The Cu(II) complexes (4 and 9) have a tetrahedral geometry, with a magnetic moment of (2.63, 2.06 B.M.) [29], and electronic spectra with a band at (19876, 24379 cm^{-1}) respectively, which indicates that the complexes are tetrahedral in shape [30].

Electronic spectra of complexes (5 and 10) of zinc (II) do not expose a d-d band, and these complexes have diamagnetic moments for d^{10} ions. These findings suggest that the formal arrangement of that chelating agent might be as follows [31]. Electronic spectra and magnetic instant data are listed in Table 4.

Table 4. Electron spectra and magnetic moments of complexes

No.	Complex	Transition		μ_{eff} (B.M)
1	$[bCar]_3[CrCl_6]$	20688 23781 15643	$^4A_{2g} \rightarrow {}^4T_{1g}(p)$ $^4A_{2g}$ $^4T_{1g}(F)$ $^4A_{2g}$ $^4T_{2g}(F)$	3.85
2	$[bCar]_2[CoCl_4]$	---	----	4.03
3	$[bCar]_2[NiCl_4]$	14690	---	3.96
4	$[bCar]_2[CuCl_4]$	19876	---	2.63
5	$[bCar]_2[ZnCl_4]$	---	---	Dia
6	$[bTpp]_3[CrCl_6]$	32263 2376 14768	$^4A_{2g}$ $^4T_{1g}(p)$ $^4A_{2g}$ $^4T_{1g}(F)$ $^4A_{2g}$ $^4T_{2g}(F)$	3.79
7	$[bTpp]_2[CoCl_4]$	---	----	4.89
8	$[bTpp]_2[NiCl_4]$	14588	---	3.32
9	$[bTpp]_2[CuCl_4]$	24379	----	2.06
10	$[bTpp]_2[ZnCl_4]$	----	----	Dia

Conductivity measurements. The results of the molar electrical conductivity measurements of the prepared complex salts have shown that they agree with the proposed compositional formulas. The conductivity values in the DMSO solvent for all the complex salts indicate that they are electrically conductive and that they fall within the range of electrolytic complexes with a ratio of 2:1 for all complex salts [32, 33, 34] except chromium complexes, which were electrolyte complexes with a ratio of 3:1 as shown in Table 5.

Table 5. Physical characteristicof prepared ligands and their complicated

No.	Compound	Color	m.p (°c)	Yield %	Analysis, (calc.)%					(Λ) $cm^2.ohm^{-1}.mol^{-1}$
					C	H	N	M	Cl	
	(bCar)Cl	Grey	210	89	77.68 (77.35)	5.45 (5.31)	4.77 (4.65)	---		---
	(bTpp)Cl	White	300	85	77.22 (77.01)	5.66 (5.42)	---	---		---
1	$[bCar]_3[CrCl_6]$	Green dark	250	91	65.83 (65.31)	4.62 (4.51)	4.04 (3.87)	17.78 (17.43)	42.67 (42.31)	226
2	$[bCar]_2[CoCl_4]$	Brown	258	84	63.60 (63.20)	4.46 (4.32)	3.90 (3.26)	18.09 (17.89)	41.65 (41.22)	65
3	$[bCar]_2[NiCl_4]$	Pale brown	233	87	63.62 (63.22)	4.46 (4.09)	3.91 (3.73)	17.98 (17.53)	42.59 (42.09)	79
4	$[bCar]_2[CuCl_4]$	Olive	190	81	63.19 (63.09)	4.43 (4.22)	3.88 (3.61)	16.87 (16.54)	41.88 (41.65)	67
5	$[bCar]_2[ZnCl_4]$	Pale green	186	76	63.63 (63.40)	4.42 (4.25)	3.87 (3.69)	15.87 (15.04)	40.43 (40.05)	76
6	$[bTpp]_3[CrCl_6]$	Pale green	193	85	77.22 (77.01)	5.66 (5.48)	---	17.43 (17.05)	42.76 (42.32)	237
7	$[bTpp]_2[CoCl_4]$	blue	211	74	66.10 (65.96)	4.84 (4.57)	---	18.56 (18.49)	41.56 (41.28)	75
8	$[bTpp]_2[NiCl_4]$	Pale green	218	86	66.12 (65.03)	4.87 (4.74)	---	17.55 (17.04)	42.59 (42.27)	77

No.	Compound	Color	m.p (°c)	Yield %	Analysis, (calc.)%					(Λ) cm^2.ohm^{-1}.mol^{-1}
					C	H	N	M	Cl	
9	$[bTpp]_2[CuCl_4]$	orange	222	79	65.77 (65.39)	4.82 (4.33)	---	17.88 (17.26)	41.89 (41.69)	80
10	$[bTpp]_2[ZnCl_4]$	White	239	95	65.64 (65.29)	4.81 (4.74)	---	(16.55 (16.12)	40.33 (40.08)	76

d= decomposition temperature

4. Conclusion

In summary, we present the findings on creating and studying the ligand along with its metal compound involving chromium, cobalt, nickel, copper, and zinc. The outcomes of our analysis using spectrophotometry, elemental assay (C.H.N.S), infrared spectroscopy (I.R.), ultraviolet spectroscopy (U.V.) conduction tests well, as the determination of chlorine and metal content aligned with the proposed structure showing both tetrahedral and octahedral configurations.

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