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Synthesis and characterization of Ru (II) and Ru (III), complexes with poly functional amides

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Abstract
A new poly-functional amide ligands were used to afford new mononuclear Ru (II) and Ru (III) coordination compounds. All the complexes were characterized by IR, ^{1}H NMR, ^{13}C NMR, mass, ESR, Electronic spectra and their structures have been proposed.

Keywords: Poly-functional amide, octahedral Ruthenium complexes

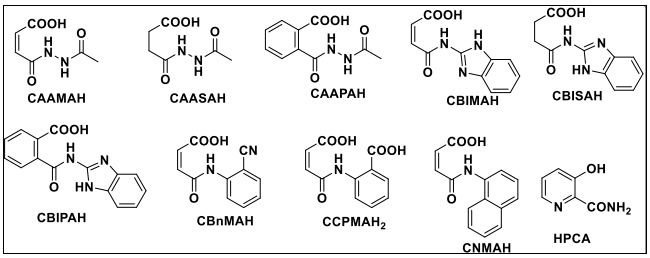
Introduction

Ruthenium is one of the rarest elements in the earth's crust, yet it exhibits the most diverse coordination chemistry among transition metals, with oxidation states ranging from -2 to +8, most commonly Ru (II) and Ru (III). Ruthenium complexes typically adopt square planar, trigonal bipyramidal, or octahedral geometries. As a 4d transition metal, ruthenium forms low-spin complexes with large ligand field splitting, even with weak ligands like water and ammonia. Ru (II) complexes are diamagnetic with a low-spin t2g^6 configuration, while Ru (III) complexes are paramagnetic with unpaired electrons. Their electronic spectra show multiple d-d transitions, influenced by the strong crystal field.

Various ruthenium complexes have been synthesized and characterized, exhibiting distorted octahedral geometry with monodentate ligands such as pyrazole. Some complexes have been studied for their antitumor and antimetastatic effects in mice. Ruthenium's electron-withdrawing properties make it a promising candidate for catalyzing urea hydrolysis, unlike some other metal complexes. Linkage isomerization involving ambidentate ligands is well-documented in Ru (II) and Ru (III) complexes.

Ruthenium (II) polypyridyl complexes attract attention for their optical and electronic properties, useful in photochemical sensitizers for solar energy conversion. Ruthenium (II) diamine complexes serve as sensitive photophysical probes for solids, surfaces, and biopolymers. Peptides and amides are important ligands in coordination chemistry, forming stable complexes with transition metals.

Ruthenium complexes also catalyze olefin hydrogenation and isomerization reactions, including selective cis-trans isomerization without polymerization or hydrogenation. Ligands containing heteroatoms have expanded the structural and catalytic diversity of ruthenium complexes. Substituted amides find extensive applications in agriculture and medicine, including as plant growth regulators and antiepileptic agents. Various Ru(II) and Ru(III) complexes with multifunctional amide ligands have been synthesized and studied, with ongoing research to further explore their properties and applications.



Results and Discussion

Characterization of ligands

The ligands are prepared as per the procedures described in experimental section and characterized by elemental analysis, mass, ^{1}H-NMR and IR spectral data. These amide ligands are fairly soluble in common organic solvents. In general7, condensation of an amine with an anhydride leads to the formation of corresponding amide or imide. In our laboratory we have isolated and characterized both the structures of amide and imide and reported earlier[40]. As a part of our investigation poly functional amides are synthesized and characterized and presented in this dissertation. The analytical data of all carboxy amide ligands are in good agreement with the proposed molecular formulae (Table - 1).

The IR spectrum of CAAMAH (Fig.1) shows characteristic absorption bands at 3400, 3150, 1710, 1675 and 1620 cm^{-1} corresponding to NH_{asy}, NH_{sym}. COOH and CO (amide) respectively. The ^{1}H - NMR spectrum (Fig.2) exhibits signals at 2.01 (s) (3H , CH_3): 6.2-6.6 doublet of doublet (2H , CH = CH) ; 11.8 & 3.0 (2H , NH) and sharp signals at 10.6 (1H , COOH). These integrated signals and their assignments are in good agreement with the proposed structural formula. The mass spectrum of the compound and the other exhibits a peak at m /z = 172 (molecular ion) intense peaks corresponding to different fragments arising from a molecule of the CAAMAH ligand.The IR spectra of CAASAH and CAAPAH exhibit similar characteristic absorption bands as we observed for CAAMAH with amide form.

Table1: Physical and Analytical Data of Amide Ligands

S. No	Ligand	Molecular Formula	Colour	Melting point (° C)	Elemental Analysis Found (Cald.)		
					C	H	N
1.	CAAMAH	$C_6H_8N_2O_4$	White	180	41.92 (41.86)	4.70 (4.65)	16.50 (16.27)
2.	CAASAH	$C_{11}H_8N_30_3$	White	175	41.38 (41.37)	5.82 (5.74)	16.25 (16.09)
3.	CAAPAH	$C_{10}H_9N_3O_3$	White	125	54.12 (54.05)	4.58 (4.50)	12.72 (12.61)
4.	CBIMAH	$C_{11}H_{10}N_2O_4$	Light Yellow	185	57.50 (57.14)	3.95 (3.89)	18.40 (18.18)
5.	CBISAH	$C_{11}H_{11}N_3O_3$	White	225	56.92 (56.65)	4.80 (4.72)	18.20 (18.00)
6.	CBIPAH	$C_{15}H_{11}N_3O_3$	Cream Colour	198	64.50 (64.05	4.00 (3.91)	15.00 (14.94)
7.	CBnMAH	$C_{11}H_9N_1O_5$	White	130	61.12 (61.11)	3.73 (3.70)	12.98 (12.96)
8.	$CCPMAH_2$	C11HGN105	Cream colour	176	56.20 (56.17)	3.86 (3.82)	6.00 (5.95)
9.	CNMAH	$C_{14}H_{11}N_1O_3$	Light Yellow	188	69.80 (69.70)	4.62 (4.56)	5.86 (5.80)
10	HPCA	$C_6H_6N_2O_2$	White	194	52.25 (52.17)	4.38 (4.34)	20.30 (20.28)

^{1}H - NMR spectrum of CAASAH shows signals at ∂ 2.0 6 (3H, CH_3); 4.78 (4H , CH_2 - CH_2) 7.6 (br, 2H, NH) 10.5(s) (1H , COOH). Mass spectral data confirm the proposed structure by possessing the molecular ion peak at m/z 174 and other peaks corresponding to suitable fragments. ^{1}H - NMR spectrum of CAAPAH shows resonance in at 2.00 ∂ (3H , CH3); 7.0-8.0 multiplet (4H, Ar ring) 8.6 (br, 2H, NH) respectively. These signals are in agreement with data proposed structural formula. Mass spectral data support the proposed structure by possessing the molecular signals the ion peak at m /z = 222 . The IR spectrum exhibits CBnMAH of significant frequencies around 3250 , 3180 (NHasy, 1700 NHsym); 1700 cm $^{-1}$ (COOH) : 1660 cm $^{-1}$CO (amide) groups . These bands are in harmony with the proposed structure.

^{1}H - NMR spectrum of CBnMAH resonates at ∂ 6.3-6.6 (d, d) (2H , CH = CH); 3.40 (br , 1H , NH); 7.0-7.8 multiplet(4H, Ar) with a sharp signal at 10.60 (1H , COOH). These signals and their alignment are in good agreement with the proposed structure. The structure of this compound is further confirmed by the mass spectrum by which shows the molecular ion peak at m/z = 216 and base peak at m/z 118. The are IR spectra of CBIMAH , CBISAH and CBIPAH exhibit characteristic vibrations assignable to NH (amide),NH (benzimidazole) C=N (benzimidazole ring), COOH , CO table are recorded in (Table 2) along with the IR frequencies of other ligands.The ^{1}H - NMR spectral data of the ligands are recorded in Table - 3. The ^{1}H - NMR spectrum of the ligand $CCPMAH_2$was presented. The elemental analysis data and spectral data are in good agreement with the proposed structure of all the ligands.

Table 2: Ir Selected Stretching Frequencies of Ligands (Cm^{-1})

Compound	N-H	COCH	co (amide)	C – N
CAAMAH	3400 , 3150	1710	1675 , 1620	1580
CAASAH	3200 , 3040	1690	1640	1580
CAAPAH	3210 , 3040	1700	1650 , 1620	1585
CBIMAH	3300 , 3150	1690	1635	1590
CBISAH	3340 , 3120	1700	1640	1575
CBIPAH	3200 , 3060	1690	1660	1575
CBnMAH	3220 , 3180	1700	1650	1550
$CCPMAH_2$	3360 , 3150	1710	1635	1560
CNMAH	3250 , 3020	1700	1650	1550
HPCA	3380 , 3200	--	1680	--

* C = N of benzimidazole is observed at 1660 cm^{-1}
# C = N is observed at 2200 cm^{-1}
$ OH is observed at 3500, 2650 cm -1.

Table 3: H - NMR Spectral Data of Ligands

Ligand	-CH3 (S)	CH_2 - CH_2 (m)	-CH = CH- (d)	- NH (br)	-Ar (m)	-COOH (s)
CAAMAH	2.01 (3H	--	6.2-6.6 (2H)	11.8 & 3.01	--	10.80
CAASAH	2.0 (3H)	4.8 (4H)	--	7.6 (2H)	--	10.50
CAAPAH	2.0 (3H)	--	--	8.6 (2H)	7.31-8.0 (4H)	--
CBIMAH	--	--	6.0-6.6 (2H)	5.4 (1H)	7.0-7.5 (4H)	--
CBISAH	--	3.5 (4H)	--	4.5 (1H)	6.9-7.8 (4H)	--
CBIPAH	--	--	--	4.6	6.8-7.8 (4H)	10.8
CBnMAH	--	--	6.2-6.6 (2H)	3.4 (M)	6.9-7.6 (4H)	10.57
$CCPMAH_2$	--	--	6.0-6.6 (2H)	5.4	7.2-8.6 (4H)	11.4
CNMAH	--	--	6.2 - 6.6 (2H)	4.8	6.8-7.8 (8H)	10.50
HPCA	--	--	--	3.6	--	--

* COOH signal is not observed which may be due to rapid exchange of proton with that of the solvent

Characterisation of Ru (II) and Ru (III) Complexes

Physical characteristics of the complexes:

All the Ru (II) and Ru (III) complexes are crystalline, non - hygroscopic and stable

At room temperature. The colour of the complexes vary from light green to light brown or snuff. These complexes are freely soluble in DMSO, DMF and methanol but sparingly soluble in water.

Elemental Analysis

The analytical data of the complexes of Ru (II) and Ru (III) are presented in Tables 4 & 5 respectively. The theoretical percent values of the elements in the complexes have been calculated as per the composition given in the tables. It is clear from the tables that the experimental values are in agreement with the calculated ones. The complexes may, therefore, be formulated as shown in the tables.

Table 4: Physical and Analitical Data of Ru (II) Complexes

S. No	Complex	Molecular Formula	Colour	A Analytical Data Found (cald)%			MOhm^{-1} cm^2 mol^{-1}	u eff (B.M)
				C	H	N		
1.	[Ru(CAAMA)$_2$ (DMSO) $_2$]	$C_{16}H_{26}N_4O_{10}S_2Ru$	Pale Green	32.20 (32.01)	4.36 (4.33)	9.50 (9.88)	9.0	Diamagnetic
2.	[Ru(CARSA) $_2$ (DMSO) $_2$]	$C_{16}H_{30}N_4O_{10}S_2Ru$	Snuff	31.75 (31.84)	4.98 (4.96)	9.40 (9.24)	8.8	Diamagnetic
3.	[Ru(CAAPA)$_2$]	$C_{20}H_{18}N_4O_8S_2Ru$	Snuff	44.20 (44.14)	3.52 (3.31)	10.16 (10.29)	8.3	Diamagnetic
4.	[Ru(CBIMA)$_2$ (DMSO) $_2$]	$C_{26}H_{28}N_6O_8S_2Ru$	Pale Green	44.00 (43.47)	3.82 (3.90)	11.85 (11.70)	8.2	Diamagnetic
5.	[Ru(CBISA)$_2$ (DMSO) $_2$]	$C_{26}H_{32}N_6O_8S_2Ru$	Thick Violet	43.47 (43.52)	4.62 (4.43)	11.21 (11.30)	9.1	Diamagnetic
6.	[Ru(CBIPA)$_2$ (DMSO) $_2$]	$C_{30}H_{20}N_6O_6S_2Ru$	Thick Violet	43.52 (43.27)	3.10 (3.02)	12.72 (12.59)	8.5	Diamagnetic
7.	[Ru(CBnMA)$_2$ (DMSO)$_2$]	$C_{16}H_{26}N_2O_{12}S_2Ru$	Pale brown	54.55 (54.40)	3.80 (3.78)	8.21 (8.15)	8.7	Diamagnetic
8.	[Ru(CCPMAH)$_2$ (DMSO) $_2$]	$C_{26}H_{28}N_4O_{10}S_2Ru$	Pale Green	45.00 (44.97)	4.15 (4.03)	4.05 (4.03)	8.6	Diamagnetic
9.	[Ru(CNMA)$_2$ (DMSO) $_2$]	$C_{32}H_{32}N_2O_8S_2Ru$	Pale Green	54.52 (54.41)	4.60 (4.53)	3.98 (3.98)	8.4	Diamagnetic
10.	[Ru(HPCA)$_2$ (DMSO) $_2$]	$C_{16}H_{22}N_4O_6S_2Ru$	Snuff	36.24 (36.11)	4.16 (4.13)	10.60 (10.53)	9.2	Diamagnetic

Table 5: Physical and Analitical Data of Ru (III) Complexes

S. No	Complex	Molecular Formula	Colour	A Analytical Data Found (cald)%			M Ohm^{-1} cm^2 mol^{-1}	u eff (B.M)
				C	H	N		
1.	[Ru(CAAMA)$_3$]	$C_{18}H_{21}N_6O_{12}Ru$	Pale Green	35.26 (35.13)	3.46 (3.41)	13.50 (13.66)	9.2	1.73
2.	[Ru(CARSA)$_3$]	$C_{18}H_{27}N_6O_{12}Ru$	Snuff	34.62 (34.80)	4.45 (4.35)	13.60 (13.54)	8.4	1.75
3.	[Ru(CAAPA)$_2$]Cl	$C_{20}H_{18}N_4O_8ClRu$	Snuff	41.21 (41.42)	3.15 (3.10)	9.54 (9.66)	38.5	1.65
4.	[Ru(CBIMA)$_3$]	$C_{33}H_{24}N_9O_9Ru$	Pale Green	50.44 (50.01)	3.00 (3.03)	16.00 (15.9)	8.2	1.80
5.	[Ru(CBISA)$_3$]	$C_{33}H_{20}N_9O_9Ru$	Thick Violet	49.85 (49.64)	3.81 (3.76)	15.5 (15.7)	8.9	1.72
6.	[Ru(CBIPA)$_2$]Cl	$C_{30}H_{20}N_6O_6ClRu$	Thick Violet	51.88 (51.63)	2.76 (2.86)	12.10 (12.04)	36.0	1.66
7.	[Ru(CBnMA)$_3$]	$C_{33}H_{21}N_6O_9Ru$	Pale brown	53.00 (52.89)	2.85 (2.81)	11.42 (11.24)	8.2	1.78
8.	[Ru(CCPMAH)$_3$]	$C_{33}H_{24}N_3O_{15}Ru$	Pale Green	49.55 (49.27)	2.88 (2.98)	5.28 (5.22)	8.0	1.64
9.	[Ru(CNMA)$_3$]	$C_{42}H_{30}N_3O_3Ru$	Pale Green	61.56 (61.33)	3.55 (3.65)	5.15 (5.22)	8.5	1.63
10.	[Ru(HPCA)$_3$]	$C_{18}H_{15}N_6O_6Ru$	Snuff	44.20 (36.11)	2.91 (2.82)	15.70 (15.82)	9.6	1.82

Mass Spectra

Mass spectra of two of the ruthenium complexes were recorded and the representative spectrum of [Ru (CBIMA)$_2$(DMSO)$_2$] is presented. The mass spectrum of the above complex exhibits the molecular ion peak at m/z 717 and the base peak at m/z 133 along with the other intense peaks corresponding to different fragments arising from a molecule of the complex. The fragmentation.

Infrared Spectra

IR Selected Stretching Frequencies of Ru (II) Complexes

Table 6:

S. No.	Complex	N - H	C=O (amide)	COO- ASY	COO- SYM
1.	[Ru(CAAMA)$_2$(DMSO)$_2$] *	3380 , 3160	1640, 1620	1550	1360
2.	[Ru (CAASA)$_2$ (DMSO)$_2$]	3200, 3060	1620	1540	1380
3.	[Ru(CAAPA)$_2$]	3220 , 3050	1650, 1600	1570	1390
4.	[Ru (CBIMA)$_2$(DMSO)$_2$]	3280	1620	1560	1390
5.	[Ru (CBISA)$_2$(DMSO)$_2$]	3320 , 3140	1630	1540	1370
6.	[Ru(CBIPA)$_2$]	3220 , 3050	1650, 1600	1580	1380
7.	[Ru(CBNMA)$_2$ (DMSO)$_2$]	3220. 3100	1620	1570	1360
8.	[Ru(CCTMNH) 2 (DMSO) 2)]	3240 , 3100	1610	1530	1360
9.	[Ru (CIMA) $_2$ (DMSO) $_2$]	3280 , 3160	1620	1540	1370
10.	[Ru (HPCA) 2 (DMSO) 2]	3400 , 3220	1650		

The elemental analysis of all Ru (II) complexes except 3 and 6 indicate the presence of two DMSO molecules in every complex, which is further confirmed by ^{1}H - NMR spectra. The IR spectrum of DMSO exhibits a very strong

band at 1055 cm^{-1} attributed to (S=O). On coordination to the metal ion this absorption band shifts either to high or low frequency side depending on the atoms (S/O) involving in the bonding. A low frequency absorption is reported when DMSO is coordinating through oxygen while a high frequency absorption is reported when Sulphur coordinates to the metal ion.[45] In the spectra of the above Ru (II) complexes a band is observed at 1070-1100 cm^{-1} indicating the coordination of sulphur of DMSO to the metal ion.

^{1}H - NMR Spectra

The ^{1}H - NMR spectrum of [Ru $(CBISA)_2$ ($DMSO)_2$] is recorded in D_6 - DMSO and presented in Fig. 13. The integral intensity of each signal in the ^{1}H - NMR spectra of ligand and complex is found to agree withthe number of different types of protons present. The presence the of DMSO in coordination sphere is confirmed by characteristic methyl protons resonance signal at 2.8. The NMR spectrum of the above Ru (II) complex exhibits resonance signals at 3.4 (4H, -CH_2 - CH_2). 4.4 (1H , N - H) , 6.6-7.3 (4H , Aromatic) . The disappearance of carboxylic oxygen proton signal in the complex confirms the coordination of carboxylic after deprotonation. The broad signal of N - H proton virtually shows no significant change in its shift confirming the non participation of this group in chelation.

ESR Spectra

ESR Data of Ruthenium (III) Complexes

Table 9:

Complex	g_1	g_2	g_3	g_{ave}	A_{ave}
[Ru$(CBIMA)_3$] *	2.35	2.22	1.93	2.16	-
[Ru$(OCPMAH)_3$]*	2.40	2.18	1.99	2.19	-
[Ru$(CNMA)_3$]@	2.32 1.99	2.33 1.92	2.12 1.84	2.07	135
[Ru$(CBIMA)_3$]@	2.36 2.04	2.23 1.93	2.14 1.85	2.09	150

*=Spectra recorded at room temperature. @= Spectra recorded at liquid nitrogen tarperature.

The magnitude of hyperfine coupling constant depends on the factors like (I) isotropic contact term, (II) direct dipolar and (III) indirect dipolar contributions. The isotropic contact term is due to the core polarization which results from the exchange of d and s electrons, and causes magnetic field around the nucleus. Increase in covalency of the metal - ligand bond results in the decrease in g ' value because the direct dipolar contribution. Indirect dipolar contribution depends on the g_{aniso} which is more for less symmetric structures. Higher A_{ave} values for all these complexes than that of Ru (III) free ion (A_{ave} = 54.3) suggest the covalency of metal - ligand bond. The magnetic and electronic spectral data of the present complexes indicate them to be low - spin octahedral.

The anisotropic nature associated with the spectra indicates that the complexes are distorted. The distortion may be due to non - identical coordination sites present around the metal ion. Based on the spectral evidences and foregoing discussions an octahedral geometry has been proposed for the complexes. The assigned tentative structures of Ru (II) and Ru (III) complexes are shown in Figs. 19, 20 respectively.

Conclusion

In conclusion, a new poly-functional amide ligands were used to afford new mononuclear Ru(II) and Ru(III) coordination compounds. All the complexes were characterized by IR, ^{1}H NMR, ^{13}C NMR, mass, ESR studies and their structures have been proposed.

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