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NOTE

Studies on Thermal Behaviour of Cobalt(II), Nickel(II) and Copper(II) Complexes

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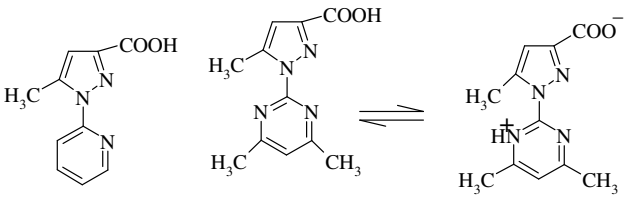
The thermogravimetric analyses of some Co(II), Ni(II) and Cu(II) complexes with two pyrazole carboxylic acids (**I** & **II**) furnish the interesting weight loss phenomena and thermal stability. The total loss is found in the cases of free ligands and for the complexes, the end products are their oxides as expected.

Key Words: Thermal analysis, Cobalt (II), Nickel (II), Copper(II), Pyridyl-pyrazole carboxylic acid, Pyrimidyl-pyrazole carboxylic acid.

There is continued interest in the studies of the complexes thermally. The present communication reports the thermogravimetric studies of some complexes of cobalt(II), nickel(II) and copper(II) complexes with biologically important two substituted pyrazole carboxylic acids (**I** & **II**)[1]. With the increase in temperature different fragments are released and finally static nature is observed at elevated temperature where corresponding oxides are formed.



PyPzCH PymPzCH HPymPzC

I **II**

All the materials used in different stages, synthesis of the ligands, synthesis of the complexes and characterization were similar as reported earlier[1].

TGA curves were taken on a Shimadzu-50, DTA-50 thermal analyzer under nitrogen atmosphere with heating rate of 10-20 ºC/min using α-Al_2O_3 as a standard, at Jadavpur University, Kolkata.

TABLE-1
THERMOGRAVIMETRIC ANALYSES DATA OF THE LIGAND, PyPzCH AND ITS METAL(II) COMPLEXES

Compound	Temp. (ºC)	Volatile products/ Fragments formed	% of wt. loss		% Yield of oxide	
			Found	Calcd.	Found	Calcd.
PyPzCH	200-250	CO_2	21.42	21.67	-	-
	250-300	$C_9H_9N_3$	78.58	78.33	-	-
$[Ni(PyPzCH)_2Cl_2]\cdot 4H_2O$	100-200	$4\ H_2O$	11.85	11.58	-	-
	200-380	$2CO_2$	52.96	53.81	-	-
	380-600	$C_9H_9N_3$	35.51	37.33	-	-
$[Ni(PyPzCH)_2(SCN)_2]$	200-280	$2CO_2$	13.95	13.50	-	-
	280-550	$C_9H_9N_3$, $2SO_2$	52.96	53.81	-	-
$[Cu(PyPzCH)_2Cl_2]$	220-280	$2CO_2$	13.95	13.50	-	-
	280-320	$C_9H_9N_3$	37.83	35.13	-	-
$[Cu(PyPzCH)_2(NO_3)_2]$	150-330	$2CO_2$,½NO_2, $2C_9H_9N_3$	89.81	86.60	-	-
	>330	-	-	-	12.21	13.31

TABLE-2
THERMOGRAVIMETRIC ANALYSES DATA OF THE LIGAND, PymPzCH AND ITS METAL(II) COMPLEXES

Compound	Temp. (ºC)	Volatile products/ Fragments formed	% of wt. loss		% Yield of oxide	
			Found	Calcd.	Found	Calcd.
PymPzCH	160-255	CO_2	18.75	18.97	-	-
	255-290	$C_{10}H_{12}N_4$	81.25	81.03	-	-
$[Co(HPymPzC)_2Cl_2]$	120-250	2HCl	13.39	12.30	-	-
	250-450	$2CO_2$, $C_{10}H_{11}N_4$	51.54	49.77	-	-
	450-750	$C_{10}H_{11}N_4$	69.14	71.39	-	-
	> 750	-	-	-	12.94	13.51
$[Ni(HPymPzC)_2(NO_3)_2].2H_2O$	180-280	$2H_2O$	4.52	5.27	-	-
	280-430	$2HNO_3$, $2CO_2$, $2C_{10}H_{11}N_4$	88.14	90.84	-	-
	> 430	-	-	-	9.45	10.23
$[Ni(HPymPzC)_2SO_4]\cdot 2H_2O$	100-180	$2H_2O$	5.83	5.53	-	-
	180-480	H_2SO_4, $2CO_2$, $C_{10}H_{11}N_4$	58.92	57.70	-	-
	480-600	$C_{10}H_{11}N_4$	68.94	71.45	-	-
	> 600	-	-	-	11.41	12.01
$[Ni(HPymPzC)_2Cl_2]\cdot 2\ H_2O$	80-120	$2H_2O$	5.97	5.71	-	-
	120-180	2HCl	12.69	11.29	-	-
	180-300	$2CO_2$	14.59	13.82	-	-
$[Cu(HPymPzC)_2Br_2]$	110-310	2HBr	23.55	23.56	-	-
	310-510	$2CO_2$	13.85	13.66	-	-
	510-570	$2C_{10}H_{11}N_4$	90.03	89.23	-	-
	> 570	-	-	-	10.82	11.34
$[Cu(HPymPzC)_2(NO_3)_2]\cdot 2H_2O$	145-245	$2\ H_2O$	6.07	5.87	-	-
	245-330	$2\ HNO_3$, $2CO_2$, $C_{10}H_{11}N_4$	57.00	5823	-	-
	330-450	$C_{10}H_{11}N_4$	70.57	70.16	-	-
	> 450	-	-	-	10.93	11.27

Thermal behaviour of the ligand, PyPzCH and some of its complexes *viz.*, chloro- and thiocyanato-species of nickel(II) and chloro- and nitrato-species of copper(II) are shown in Table-1 and the same for PymPzCH with some of its complexes *viz.*, chloro-species of cobalt(II) and nickel(II), nitrate-species of nickel(II) and copper(II), sulfato-species of nickel(II) and bromo-species of copper(II) are reflected in Table-2, respectively. It is found that the ligands start weight loss through decarboxylation[2] and ultimately suffer complete volatization making total loss in line with expectation. The species suffer weight loss at various stages through removal of H_2O (when hydrated species), CO_2 through decarboxylation[2], several volatile fragments *viz.*, $C_9H_9N_3$ (PyPz: one unit of 5-methyl-1-(2′-pyridyl)pyrazole radical, $C_{10}H_{11}N_4$ (one unit of 5-methyl-1-(4′,6′-dimethyl-2′-pyrimidyl)-pyrazole radical (as the cases might be), HCl, HNO_3, H_2SO_4, NO_2, O_2, *etc*. are believed to be removed to explain the mass loss phenomena. The final product in some of the species is NiO[3], CuO[3] and Co_3O_4[4], respectively (where experiments extended to elevated temperature). The results of the thermogravimetric analyses, thus, furnish suggestive information regarding the thermal stability of the species and the mode of cleavage of certain side chains of the primary ligand molecules.

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