Journal of Thermal Analysis, Vol. 14 (1978) 291–298

COMPLEXES OF THE ETHYLPYRIDINES WITH THE HALIDES OF COBALT(II), NICKEL(II) AND COPPER(II)

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(Received May 16, 1977)

A series of complexes has been prepared with the halides of cobalt(II), nickel(II), copper(II) and 2-,3-,4-ethylpyridine. The compounds were essentially all octahedral with the exception of those formed between 2-ethylpyridine and the cobalt(II) halides. The stereochemical configurations were deduced using spectral and magnetic properties. The decomposition of the complexes was studied by thermogravimetry and differential thermal analysis.

This work is a continuation and an extension of previously reported studies [1–9] on the coordination compounds formed between the halides of cobalt(II), nickel(II) copper(II) and 2-,3-,4-ethylpyridine. The variation of the ethyl constituent in the 2-, 3- and 4- position of the pyridine ring means that both the donor tendency of the N ring atom and the steric influence of the substituted pyridine molecule can be further investigated. The thermal decomposition of the compounds has been studied. Information regarding the stereochemistry of the compounds was obtained using electronic absorption spectra, far infrared spectra and magnetic measurements over the temperature range 300–100 K.

Experimental

The complexes were prepared by dissolving the appropriate metal halide in hot ethanolic solution and adding an excess of the nitrogen-base dissolved in ethanol. The precipitated complexes were washed with ethanol and air-dried. The complexes prepared by this method are given in Table 1. References are given to previous preparations; where no reference is given it is believed that these are new compounds.

Thermal decomposition studies were carried out in air on a Stanton Redcroft Model TR-01 Thermobalance with a DTA attachment.

Electronic spectra were obtained on a Beckman ACTA M-IV spectrophotometer using 1 cm cells. The solvent used was AR Chloroform dried over molecular sieves. Far infrared spectra were obtained on a Beckman IR 720 M interferometer using a polythene matrix to support the complexes.

Table 1

Analysis of compounds

Compound	Metal (found)	Halide (found)	Metal (theory)	Halide (theory)	Reference
$Co(2EtPy)_2Cl_2$	17.51	20.30	17.12	20.60	[1]
$Co(2EtPy)_2Br_2$	13.97	36.42	13.61	36.90	[1, 2]
$Co(2EtPy)_2I_2$	11.61	48.78	11.18	48.16	[1, 2]
$Co(3EtPy)_2Cl_2$	17.10	20.60	17.12	20.60	[1, 2]
$Co(3EtPy)_4(H_2O)_2Br_2$	8.60	23.40	8.63	23.43	[3]
$Co(3EtPy)_4Br_2$	9.00	24.52	9.10	24.69	[3]
$Co(3EtPy)_4(H_2O)_2I_2$	7.56	32.62	7.58	32.69	[3]
$Co(3EtPy)_4I_2$	7.86	34.02	7.95	34.23	[3]
$Co(4EtPy)_4Cl_2$	10.59	12.54	10.55	12.70	
$Co(4EtPy)_4Br_2$	9.65	24.80	9.10	24.69	
$Co(4EtPy)_4I_2$	8.50	34.12	7.95	34.23	
$Ni(3EtPy)_4Cl_2$	10.52	12.72	10.52	12.70	
$Ni(3EtPy)_4Br_2$	9.03	24.61	9.07	24.69	
$Ni(3EtPy)_4I_2$	7.53	34.16	7.92	34.24	
$Ni(4EtPy)_4Cl_2$	10.09	12.86	10.52	12.70	
$Ni(4EtPy)_4Br_2$	8.84	24.78	9.07	24.69	
$Ni(4EtPy)_4I_2$	7.46	34.38	7.92	34.24	
$Cu(2EtPy)_2Cl_2$	18.36	20.21	18.22	20.33	[6]
$Cu(2EtPy)_2Br_2$	14.45	36.38	14.52	36.51	[6, 8]
$Cu(3EtPy)_2Cl_2$	18.28	20.36	18.22	20.33	[4, 5]
$Cu(3EtPy)_2Br_2$	14.42	36.32	14.52	36.51	[5, 8]
$Cu(4EtPy)_2Cl_2$	18.39	20.44	18.22	20.33	[4, 5, 7]
$Cu(4EtPy)_2Br_2$	14.38	36.45	14.52	36.51	[8]

Magnetic susceptibility measurements were made by the Gouy method using a Newport variable temperature balance over the range 300 – 100 K at two different field strengths, with calibration by $Hg[Co(CNS)_4]$. Diamagnetic corrections were applied using the atomic diamagnetic susceptibilities tabulated by Figgis and Lewis [10]. The results were processed on the Napier College CTL Modular One computer using programme MAG [11].

Results

The cobalt compounds isolated from ethanolic solution for 3-,4-ethylpyridine have stoichiometry CoL_4X_2 while the 2-ethylpyridine compounds have stoichiometry CoL_2X_2. The reaction of 3-ethylpyridine with cobalt (II) chloride in ethanolic solution produced a pink compound $Co(3EtPy)_4Cl_2$ which decomposed readily in air to give blue $Co(3EtPy)_4Cl_2$ while the reaction with cobalt(II) bromide and cobalt(II) iodide gave orange compounds of stoichiometry $Co(3EtPy)_4(H_2O)_2X_2$ ($X = Br^-, I^-$). On drying at 318K overnight blue anhydrous compounds were obtained. Thermal analysis showed that all the CoL_4X_2 species decomposed via CoL_2X_2 intermediates by loss of two then a further two molecules of ligand.

The CoL_4X_2 compounds have electronic spectra which are typical of octahedrally coordinated cobalt atoms with weak multiple absorption bands near 18000 cm^{-1} and 7500 cm^{-1}. The assignment of the bands around 18000 cm^{-1} is not easy because of the poor resolution of the bands due to the interaction between the $^4A_{2g}(F)$ and $^4T_{1g}(P)$ states. The bands at around 7500 cm^{-1} are assigned to the transitions $^4T_{1g}(F) \rightarrow {}^4T_{2g}(F)$ while the multiple bands between 15000–18500 cm^{-1} are assigned to the transition $^4T_{1g}(F) \rightarrow {}^4A_{2g}(F)$, $^4T_{1g}(P)$.

Table 2

Thermal decomposition products

Starting material	Decomposition temperature, K	Resulting compound	Metal (found)	Halide (found)	Metal (theory)	Halide (theory)
$Co(3EtPy)_4Br_2$	345 (ENDO)	$Co(3EtPy)_2Br_2$	13.54	36.72	13.61	36.90
$Co(3EtPy)_4I_2$	353 (ENDO)	$Co(3EtPy)_2I_2$	11.00	48.12	11.18	48.16
$Co(4EtPy)_4Cl_2$	340 (ENDO)	$Co(4EtPy)_2Cl_2$	17.04	20.71	17.12	20.60
$Co(4EtPy)_4Br_2$	373 (ENDO)	$Co(4EtPy)_2Br_2$	13.52	36.78	13.61	36.90
$Co(4EtPy)_4I_2$	380 (ENDO)	$Co(4EtPy)_2I_2$	11.06	48.28	11.18	48.16
$Ni(3EtPy)_4Cl_2$	393 (ENDO)	$Ni(3EtPy)_2Cl_2$	17.06	20.60	17.08	20.66
	458 (ENDO)	$Ni(3EtPy)Cl_2$	24.72	29.90	24.80	29.94
	523 (ENDO)	$Ni_3(3EtPy)_2Cl_6$	29.06	35.12	29.20	35.26
$Ni(3EtPy)_4Br_2$	353 (ENDO)	$Ni(3EtPy)_2Br_2$	13.50	36.92	13.57	36.98
	450 (ENDO)	$Ni(3EtPy)Br_2$	18.00	49.10	18.03	49.07
$Ni(3EtPy)_4I_2$	380 (ENDO)	$Ni(3EtPy)_2I_2$	11.10	48.06	11.14	48.18
$Ni(4EtPy)_4Cl_2$	410 (ENDO)	$Ni(4EtPy)_2Cl_2$	17.02	20.64	17.08	20.66
	520 (ENDO)	$Ni(4EtPy)Cl_2$	24.74	29.90	24.80	29.94
$Ni(4EtPy)_4Br_2$	420 (ENDO)	$Ni(4EtPy)_2Br_2$	13.48	36.90	13.57	36.98
$Ni(4EtPy)_4I_2$	423 (ENDO)	$Ni(4EtPy)_2I_2$	11.06	48.14	11.14	48.18

The temperatures quoted are those of the maximum rate of decomposition.

The magnetic data for the compounds are given in Table 3 and agree with the suggestion of an octahedral arrangement of ligands around the cobalt atom with temperature dependent magnetic moments in the range 4.8–5.2 B.M. The compounds obey the Curie–Weiss Law over the temperature range 300–100 K.

The far infrared spectra supports an octahedral configuration for the compounds [12 infrared spectra of compounds $Co(3EtPy)_4(H_2O)_2Br_2$ and $Co(3EtPy)_4(H_2O)_2I_2$ show weak bands at 315 cm^{-1}. These bands are not observed in the anhydrous compounds $Co(3EtPy)_4Br_2$ and $Co(3EtPy)_4I_2$ and are attributed to a $Co-OH_2$ stretching mode [3].

The CoL_2X_2 compounds have electronic spectra which would suggest tetrahedral structures. The observed transitions are $^4A_2(F) \rightarrow {}^4T_1(F)$ and $^4A_2(F) \rightarrow {}^4T_1(P)$. It is possible that the complexity of the $^4A_2(F) \rightarrow {}^4T_1(P)$ band is due to the presence of a strongly tetragonal component in the ligand field rather than due entirely to spin-orbit coupling and the interaction between states since the symmetry is C_{2v} and not T_d. The magnetic data reported in Table 3 for the compounds

Table 3

Magnetic data

Compound	Temp, K	Diamagnetic correction/10	Corr $Xm\cdot10^{-6}$	μ, B.M.	Θ
$Co(2EtPy)_2Cl_2$	297	113.12	8882	4.61	3.9
$Co(2EtPy)_2Br_2$	291	134.12	9137	4.63	4.2
$Co(2EtPy)_2I_2$	294	162.12	9013	4.62	3.6
$Co(3EtPy)_2Cl_2$	295	113.12	8379	4.47	2.3
$Co(3EtPy)_4Br_2$	295	194.24	9646	4.79	9.2
$Co(3EtPy)_4I_2$	295	222.24	9865	4.85	14.3
$Co(4EtPy)_4Cl_2$	296	173.24	9849	4.85	16.5
$Co(4EtPy)_4Br_2$	295	194.24	11020	5.12	20.2
$Co(4EtPy)_4I_2$	295	222.24	11053	5.13	17.4
$Ni(3EtPy)_4Cl_2$	294	173.24	4011	3.08	−3.6
$Ni(3EtPy)_4Br_2$	291	194.24	3493	2.86	−2.4
$Ni(3EtPy)_4I_2$	298	222.24	4054	3.12	−2.5
$Ni(4EtPy)_4Cl_2$	292	173.24	3848	3.01	−4.7
$Ni(4EtPy)_4Br_2$	294	194.24	3592	2.92	−3.4
$Ni(4EtPy)_4I_2$	297	222.24	4015	3.10	−3.8
$Cu(2EtPy)_2Cl_2$	295	113.12	1324	1.78	−11.7
$Cu(2EtPy)_2Br_2$	296	134.12	1383	1.82	−6.6
$Cu(3EtPy)_2Cl_2$	295	113.12	1395	1.82	10.1
$Cu(3EtPy)_2Br_2$	295	134.12	1301	1.76	24.5
$Cu(4EtPy)_2Cl_2$	295	113.12	1365	1.80	−1.0
$Cu(4EtPy)_2Br_2$	299	134.12	1248	1.75	7.7

isolated from solution support a tetrahedral configuration with magnetic moments between 4.2–4.8 B.M., as does the far infrared spectra with cobalt-chlorine modes in the region 350–300 cm^{-1}, cobalt-bromine modes in the region 300–250 cm^{-1} and the cobalt-iodine modes around 200 cm^{-1} [12].

Nickel(II) forms compounds of stoichiometry NiL_4X_2 with 3,4-ethylpyridine. No stoichiometric 2-ethylpyridine compounds could be prepared using the present method. The results of the thermogravimetric studies are given in Table 2. All of the nickel compounds would appear to have an octahedral configuration about the metal. Thus by analogy with assigments made for other nickel compounds of this type [12] the bands are assigned at 8000–11500 cm^{-1} to the $^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$ transition, at 15000–17000 cm^{-1} to the $^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$ transition and the band at 24000–25500 cm^{-1} to the $^3A_{2g}(F) - {}^3T_{1g}(P)$ transition. For all the NiL_4X_2 species the $^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$ transition was split into two bands corresponding to the splitting of the excited $^3T_{2g}(F)$ level into $^3B_{2g} + {}^3E_g$ components [13]. The prediction of an octahedral structure for these compounds is further supported by the fact that the compounds have temperature independent magnetic moments in the range 2.8–3.2 B.M. The compounds obey the Curie–Weiss Law over the temperature range 300–100 K. The far infrared spectra also supports an octahedral configuration for these compounds [12].

Table 4

Electronic spectra, cm^{-1}
Cobalt compounds
(i) Octahedral environment (ground state $^4T_{1g}(F)$)

Compound	$^4T_{1g}(F) \rightarrow T_{2g}(F)$	$^4T_{1g}(F) \rightarrow {}^4A_{2g}(F), {}^4T_{1g}(P)$
$Co(3EtPy)_4Br_2$	6024, 7052, 9381	15 504, 15 873(sh), 16 807
$Co(3EtPy)_4I_2$	6887, 9091	14 599, 15 267, 16 051(sh)
$Co(4EtPy)_4Cl_2$	6289, 7042(sh), 9709	16 367, 17 301
$Co(4EtPy)_4Br_2$	6173(sh), 7032, 9363	15 504, 15 873(sh), 16 807
$Co(4EtPy)_4I_2$	6793, 9009	14 684, 15 385, 16 155

(ii) Tetrahedral environment (ground state $^4A_2$ (F))

Compound	$^4A_2(F) \rightarrow {}^4T_1(F)$	$^4A_2(F) \rightarrow {}^4T_1(P)$
$Co(2EtPy)_2Cl_2$	6993, 7225, 8621(sh)	15 798, 17 241(sh)
$Co(2EtPy)_2Br_2$	6944, 8475	15 723, 16 392(sh), 16 722(sh)
$Co(2EtPy)_2I_2$	6711, 8000	14 085(sh), 15 267, 16 181(sh)
$Co(3EtPy)_2Cl_2$	6309, 7194(sh), 9709	15 748(sh), 16 340, 17 271
$Co(3EtPy)_2Br_2$	6098(sh), 7042, 9416	15 504, 15 873(sh), 16 807(sh)
$Co(3EtPy)_2I_2$	6780, 8929	14 641, 15 314, 16 103(sh)
$Co(4EtPy)_2Cl_2$	6305, 7143(sh), 9756	15 773(sh), 16 340, 17 301
$Co(4EtPy)_2Br_2$	6006(sh), 7042, 9363	15 504, 15 873(sh), 16 807(sh)
$Co(4EtPy)_2I_2$	6803, 8969	14 620, 15 291, 16 103(sh)

Table 4 (cont)

Nickel compounds
Octahedral environment (ground state $^3A_{2g}(F)$)

Compound	$^3A_{2g}(F) \rightarrow {}^3T_{2g}(F)$	$^3A_{2g}(F) \rightarrow {}^3T_{1g}(F)$	$^3A_{2g}(F) \rightarrow {}^3T_{2g}(P)$
$Ni(3EtPy)_4Cl_2$	8 460, 10 638	14 749	24 096
$Ni(3EtPy)_2Cl_2$	10 695	13 986	23 810
$Ni(3EtPy)Cl_2$*	8 757	15 552	25 773
$Ni_3(3EtPy)_2Cl_6$*	8 741	15 385	25 641
$Ni(3EtPy)_4Br_2$	7 911, 10 638	17 500	25 063
$Ni(3EtPy)_2Br_2$	10 331	18 182	22 883
$Ni(3EtPy)Br_2$*	8 681	15 385	25 641
$Ni(3EtPy)_4I_2$	10, 101, 10 989	17 094	25 000
$Ni(3EtPy)_2I_2$	10 000, 10 917	17 094	25 000
$Ni(4EtPy)_4Cl_2$	8 503, 11 111	15 267	24 876
$Ni(4EtPy)_2Cl_2$	10 870	13 569	23 256
$Ni(4EtPy)Cl_2$*	8 726	15 385	25 773
$Ni(4EtPy)_4Br_2$	7 813, 11 628	16 260	25 000
$Ni(4EtPy)_2Br_2$	10 471	18 349	23 095
$Ni(4EtPy)_4I_2$	10 050, 10 989	17 241	25 316
$Ni(4EtPy)_2I_2$	10 040, 10 870	17 301	25 575

* — spectra recorded in water

Table 4 (cont)

Copper compounds

Octahedral environment (ground state ^2E_g)

Compound	$^2E_g \rightarrow {}^2T_{2g}$
$Cu(2EtPy)_2Cl_2$	14 085
$Cu(2EtPy)_2Br_2$	13 193
$Cu(3EtPy)_2Cl_2$	13 245
$Cu(3EtPy)_2Br_2$	14 286
$Cu(4EtPy)_2Cl_2$	12 937
$Cu(4EtPy)_2Br_2$	12 987

Table 5

Far infrared spectra

Compound	$\nu(M-X)$, cm^{-1}	$\nu(M-N)$, cm^{-1}
$Co(2EtPy)_2Cl_2$	306, 331	237
$Co(2EtPy)_2Br_2$	261	233
$Co(2EtPy)_2I_2$	214	240
$Co(3EtPy)_2Cl_2$	314, 343	245
$Co(3EtPy)_4Br_2$	190, 224	275
$Co(3EtPy)_4(H_2O)_2Br_2$	190, 225	—
$Co(3EtPy)_4I_2$	188, 224	242
$Co(3EtPy)_4(H_2O)_2I_2$	188, 220	—
$Co(4EtPy)_4Cl_2$	190, 222	—
$Co(4EtPy)_4Br_2$	195, 237	270
$Co(4EtPy)_4I_2$	192, 233	—
$Ni(3EtPy)_4Cl_2$	190, 220	240(sh)
$Ni(3EtPy)_4Br_2$	175, 214	247
$Ni(3EtPy)_4I_2$	173, 210	244
$Ni(4EtPy)_4Cl_2$	144, 221	260
$Ni(4EtPy)_4Br_2$	176, 220	252
$Ni(4EtPy)_4I_2$	193, 217	260(sh)
$Cu(2EtPy)_2Cl_2$	318	242
$Cu(2EtPy)_2Br_2$	286	248
$Cu(3EtPy)_2Cl_2$	290	262
$Cu(3EtPy)_2Br_2$	264	232
$Cu(4EtPy)_2Cl_2$	301	247
$Cu(4EtPy)_2Br_2$	275	245

The copper compounds have stoichiometry CuL_2X_2. Thermogravimetric studies show that no intermediate compounds are formed and that the isolated compounds decompose with loss of two molecules of the ethylpyridine. The magnetic data listed in Table 3 are unable to differentiate between octahedral and tetrahedral arrangements and consequently the evidence for structural assignment is from spectral data. The compounds exhibit a broad absorption band in the region 13000 – 14500 cm^{-1} and it is thus suggested that the compounds have a distorted octahedral configuration involving halogen bridging [14, 15]. The far infrared spectra are also characteristic of compounds with polymeric octahedral structures [4, 12]. Two infrared active metal-halogen vibrations are expected but only one is observed in the present investigation.

Discussion

The compounds formed by the reaction of 2-,3-,4-ethylpyridines with the halides of cobalt(II), nickel(II) and copper(II) are given in Table 1. A wider selection of complexes was prepared when 2-,3-,4-methylpyridines were used [16–18]. However, too much significance should not be placed on the absence of certain compounds since only one method of preparation was used in this study. The presence of the ethyl group in the 2-position of the pyridine ring results in the formation of compounds containing fewer molecules of base per metal atom. This would suggest that steric factors are more dominant than the electron donating power of the nitrogen atoms.

The thermal decomposition products are listed in Table 2. The CoL_4X_2 species undergo decomposition to CoL_2X_2 and then to the metal halide. This differs from the decomposition products obtained from the corresponding chloro- and bromo-complexes of methylpyridine, but is the same in the case of the related iodo-complexes [16]. The nickel(II) complexes of methylpyridine and ethylpyridine show similar trends in their method of decomposition for the chloro- and bromo-complexes. The general decomposition scheme would appear to be:

$$\begin{array}{lllll} & NiL_4X_2 \rightarrow & NiL_2X_2 \rightarrow & Ni_3L_2X_6 \rightarrow & NiX_2 \\ (X = Cl,Br) & & (OCT) & (OCT) & (OCT) \end{array}$$

$$\begin{array}{lll} NiL_4I_2 \rightarrow & NiL_2I_2 \rightarrow & NiI_2 \\ (OCT) & (OCT) & \end{array}$$

Although a phase with a ratio of three nickel atoms to two ligand molecules could only be isolated in the case of tetrakis (3-ethylpyridine). Nickel(II) chloride, the other chloro- and bromo-compounds showed inflections in their thermal decomposition curves at points corresponding to these phases.

The CoL_4X_2 compounds have octahedral structures while the CoL_2X_2 compounds have tetrahedral structures. The NiL_4X_2 compounds have trans-octahedral structures while the NiL_2X_2, $NiLX_2$ and $Ni_3L_2X_6$ species have octahedral configurations with bridging halide ions [17]. The CuL_2X_2 compounds have polymeric octahedral configurations about the copper atom [7].

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The authors wish to thank Beckman Instruments Ltd for the use of the Far IR spectrophotometer, IR 720 M.

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RÉSUMÉ — Une série de complexes a été préparée avec les halogénures de cobalt(II), nickel(II), cuivre(II) et éthyl-2-, 3-, 4- pyridine. La plupart des composés sont octaédriques, à l'exception de ceux formés à partir de l'éthyl-2 pyridine et les halogénures de cobalt(II). Les configurations stéréochimiques ont été déduites des propriétés spectrales et magnétiques. La décomposition des complexes a été suivie par thermogravimétrie et analyse thermique différentielle.

ZUSAMMENFASSUNG — Eine Reihe von Komplexen wurde aus den Halogeniden von Cobalt(II), Nickel(II), Kupfer(II) und 2-, 3-, 4-äthylpyridin hergestellt. Die Verbindungen waren im Wesentlichen alle oktaedrisch, mit Ausnahme jener, die aus 2-Äthylpyridin und den Cobalt(II)halogeniden gebildet wurden. Die stereochemischen Konfigurationen wurden aus den spektralen und magnetischen Eigenschaften abgeleitet. Der Zerfall der Komplexe wurde durch Thermogravimetrie und Differentialthermoanalyse untersucht.

Резюме — Был получен ряд комплексов 2-, 3-, 4-этилпиридинов с галоидами кобальта (II), никеля (II) и меди (II). В основном все комплексы были октаэдрическими, за исключением, тех, которые образуются между галоидами кобальта (II) и 2-этилпиридином. Стереохимические конфигурации были выведены, используя спектралные и магнитные свойства. Разложение комплексов было изучено с помощью термогравиметрии и дифференциального термического анализа.