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FT-IR SPECTROSCOPIC INVESTIGATION OF SOME M(BENZOYL CHLORIDE)$_2$Ni(CN)$_4$ COMPLEXES (M = Co, Ni and Cd)

Neslihan KAYA[1], *Zeki KARTAL[2] , Semiha BAHÇELI[3]

[1] Department of Physics, Graduate School of Applied and Natural Sciences, Süleyman Demirel University Isparta, nsl_ky@yahoo.com
[2] Department of Physics, Faculty of Art and Science, Dumplupınar University Kütahya / TÜRKİYE, zkartal@dumlupinar.edu.tr
[3] Department of Physics, Faculty of Art and Science, Süleyman Demirel University Isparta / TÜRKİYE, bahceli@fef.sdu.edu.tr

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ABSTRACT

New Hofmann-type complexes formulated as M(Benzoyl chloride)$_2$Ni(CN)$_4$ (M = Co, Ni and Cd) have been obtained in the powder form and their FT-IR spectra have been reported in the range of (4000-400) cm^{-1}. The thermal behaviour of these complexes has been also investigated by thermo-gravimetric analysis (TGA), differential thermal analysis (DTA) and derivative thermal gravimetric analysis (DTG) in the range of (30-700) ^{o}C. The spectral and thermal features suggest that the compounds substantially isostructural to that of the already known the Hofmann type complexes. The Hofmann-type complexes consist of the corrugated $|M–Ni(CN)_4|_\infty$ polymeric layers which are held in parallel through the Van der Walls interactions of (-M-Benzoyl chloride...Benzoyl chloride-M-) in our new Hofmann type complexes formed by bounding electrons of oxygen-donor atoms of the carbonyl group of Benzoyl chloride ligand to transition metal atoms.

Keywords: *Hofmann-type complexes, Infrared spectroscopy, Benzoyl chloride, Polymeric layers, Tetracyanonickelate, Transition metals, Thermal analysis.*

BAZI M(BENZOİL KLORÜR)$_2$Ni(CN)$_4$ BİLEŞİKLERİNİN FT-IR SPEKTROSKOPİK İNCELENMESİ (M = Co, Ni ve Cd)

ÖZET

M(Benzoil klorür)$_2$Ni(CN)$_4$ (M = Co, Ni ve Cd) formülüyle verilen bazı yeni Hofmann tipi bileşikler toz halinde elde edildi ve (4000–400) cm^{-1} spektral aralığında FT-IR spektrumları alındı. Bunun yanında (30–700) ^{o}C sıcaklık aralığında ısıl davranışları (TGA, DTA ve DTG) da incelendi. Yapılan incelemeler neticesinde bileşiklerin bilinen Hofmann tipi bileşiklerle benzer yapıda oldukları görüldü. Hofmann tipi bileşikler $|M–Ni(CN)_4|_\infty$ polimerik tabakalardan meydana gelir. Bu polimerik tabakalar van der Walls etkileşimleri ile paralel olarak tutulur. Yeni Hofmann-tipi bileşiklerimizde Benzoil klorür ligand molekülü geçiş metali atomlarına karbonil grubunun elektron verici oksijen atomundan bağlanmasıyla oluşmuştur (-M-benzoil klorür ... Benzoil klorür-M).

Anahtar Kelimeler: *Hofmann tipi bileşikler, İnfrared spektroskopisi, Benzoil klorür, Polimerik tabakalar, Tetrasiyononikel iyonu, Geçiş metalleri, Isıl analizler.*

1. INTRODUCTION

The Hofmann-type complexes, given by the general formula of ML$_2$Ni(CN)$_4$were produced by using N-donor ligand molecules, O-donor ligand molecules and S-donor ligand molecules. In the formulas given above, M (Mn, Fe, Co, Ni, Cu and Zn) is a transition metal atom having the valence +2, and L is either a bidentate or two monodentate ligand molecules [1, 2]. The transition metals are defined as those elements that have partly filled d (or f) shells, either in the elemental state or in their important compounds. The transition metals react with a variety of molecules or groups, called ligands, to form transition metal complexes. The Hofmann-type complexes are built by stacking the two dimensional networks, in which M(II) takes an octahedral coordination, satisfied by four N-terminals of the cyano groups and two donor atoms of ligand molecules protruding above and below the network, and the square planar Ni(CN)$_4$ shares vertices with four M(II) octahedral to build up the 2D-network [1-3]. Using various ligands, different structural cavities are formed between these polymeric layers as depending on altered shape and size of ligand molecule and metal atoms. The cavities within the Hofmann-type host structures can provide empty voids of varying shapes and dimensions, in which guest molecules may be imprisoned. The water molecules can enter into these cavities because the complexes are prepared in water environment. Besides this, the water molecules from the moisture of air can also enter into these cavities. The purpose of this study is to offer new Hofmann-type benzoyl chloride complexes and to investigate the coordination ability of benzoyl chloride and to examine coordination sensitive ligand modes, by studying isostructural complexes of benzoyl chloride.

In this study three Hofmann-type benzoyl chloride complexes M(Benzoyl chloride)$_2$Ni(CN)$_4$ (M = Co, Ni and Cd) were prepared and their FT-IR spectra are reported for the fist time. We have also reported elemental analysis. Additionally, we have studied the thermal behavior of these complexes with TGA, DTA and DTG techniques.

2. EXPERIMENTAL

2.1. Materials and Syntheses of the Complexes

All the chemicals, namely Benzoyl chloride (as abbreviated PhCOCl) (C_7H_5ClO; Fluka, 98.5%), $CoCl_2.6H_2O$ (Fluka, 99%), $NiCl_2.6H_2O$ (Fluka, 98%), $CdCl_2.2H_2O$ (Fluka, 96%) and $K_2[Ni(CN)_4].aq$ (Fluka, 98%), were used without further purification. The complexes were prepared as follows: first, 1 mmol $K_2[Ni(CN)_4]$ was dissolved in distillated water and 2 mmol PhCOCl ligand molecule were added to the solution under stirring rapidly during 2-3 minutes. Then, 1 mm MCl_2 (M = Co, Ni and Cd) solution in distillated water added to this mixture and the whole was stirred during 5 days. The precipitates formed were filtered and washed with distillated water, ethanol and diethyl ether successively kept in desiccators containing silica gel [4].

2.2. Instrumentation

The FT-IR spectra of these complexes were recorded in the range of (4000 – 400) cm^{-1} at room temperature on a Perkin-Elmer spectrum One FT-IR (Fourier Transformed Infrared) Spectrometer having the resolution of 2 cm^{-1} in the transmission mode. The TGA, DTA and DTG curves of the complexes were recorded under air at a heating rate of 10°C/min in the temperature area (20-700) °C using platinum crucibles on a SII EXSTAR 6000 TG/DTA 6300 thermal analyzer. The complexes were analyzed for metals with Perkin-Elmer optima 4300 DV ICP-OES and for C, H and N amounts with CHNS-932 (LECO) elemental analyzer. The results of elemental analyses are given in Table 1. According to the result of elemental and spectral analysis, there is small amount molecule water in the all complexes.

3. RESULTS AND DISCUSSION

The FT-IR spectral features of the complexes M(PhCOCl)$_2$Ni(CN)$_4$ (M = Co, Ni and Cd) are found to be similar to each other. The similar spectral features of the complexes suggest that they have similar analogous structural features [5]. The FT-IR spectra of bulk PhCOCl and M(PhCOCl)$_2$Ni(CN)$_4$ (M = Co, Ni and Cd) complexes are illustrated in Fig 1.

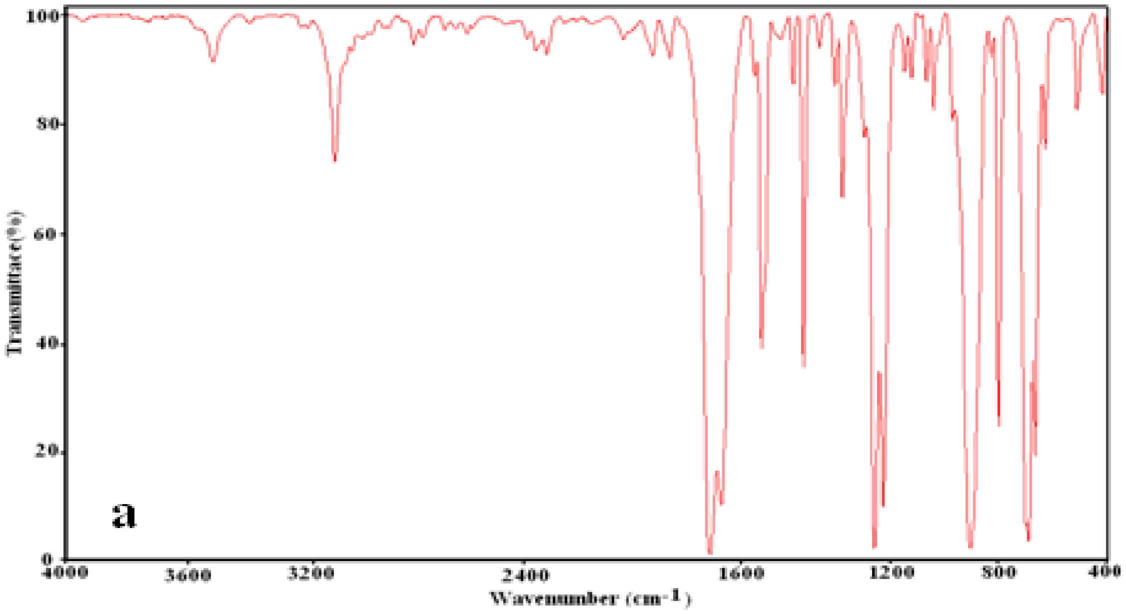
Table1. Elemental analysis of the M(PhCOCl)$_2$Ni(CN)$_4$ (M = Co, Ni and Cd) complexes.

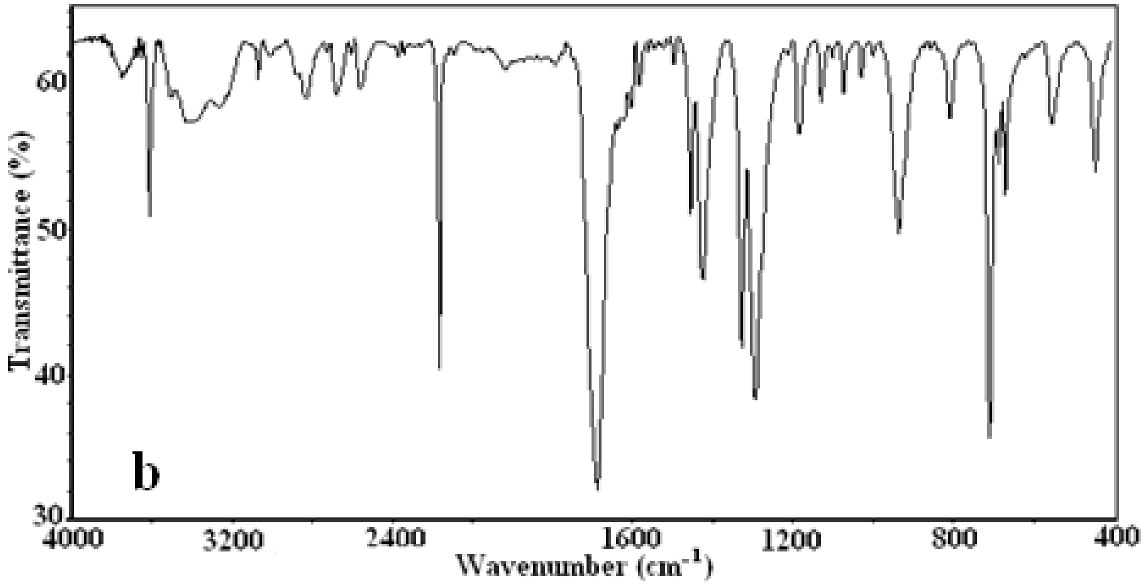
Abbreviations of complexes and M_r (g)	**Elemental analysis, Found (%)/ (Calculated) (%)**					
	C	**H**	**N**	**Co**	**Ni**	**Cd**
$Co(C_7H_5OCl)_2Ni(CN)_4$ Co-PhCOCl-Ni, M_r = 520.85	41.33 (41.51)	2.40 (2.32)	10.46 (10.76)	11.03 (11.31)	10.96 (11.27)	- (-)
$Ni(C_7H_5OCl)_2Ni(CN)_4$ Ni-PhCOCl-Ni, M_r = 520.60	41.09 (41.53)	2.47 (2.32)	10.60 (10.76)	- (-)	22.24 (22.55)	- (-)
$Cd(C_7H_5OCl)_2Ni(CN)_4$ Cd-PhCOCl-Ni, M_r = 574.32	37.06 (37.42)	2.18 (2.11)	9.82 (9.76)	- (-)	9.94 (10.22)	19.13 (19.57)

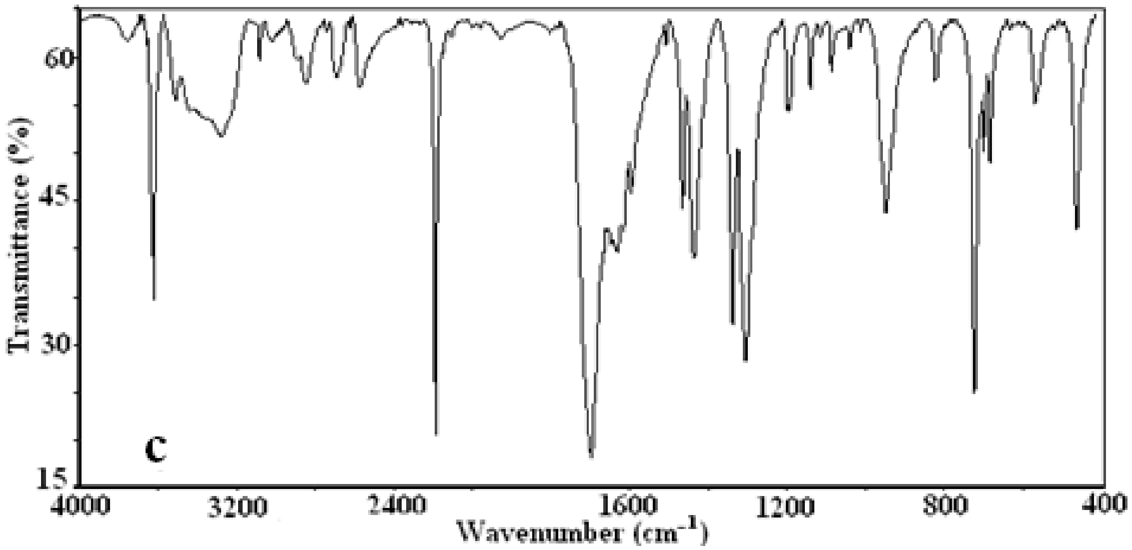
The FT-IR spectral data obtained can be interpreted two parts by considering the vibrations of ligand PhCOCl molecules and the $[Ni(CN)_4]^{2-}$ ion units.

3.1. PhCOCl Vibrations

Benzoyl chloride, also known as benzenecarbonyl chloride (PhCOCl), is an organochlorine compound with the formula C_6H_5COCl. It is a colorless, fuming liquid with an irritating odor. A carbonyl group is a functional group composed of a carbon atom double-bonded to an oxygen atom (C=O). The PhCOCl molecule contains a carbonyl group having an oxygen atom which can donate electrons. Hence, it expects that the binding takes place with the oxygen atom of the carbonyl group to the transition metal atoms. The PhCOCl molecule is mainly useful for the production of peroxides but is generally useful in other areas such as in the production of dyes, perfumes, pharmaceuticals, and resins. It is also used in photography; as gasoline gum inhibitors, and in the manufacture of synthetic tannins. It was formerly employed as an irritant gas in chemical warfare.







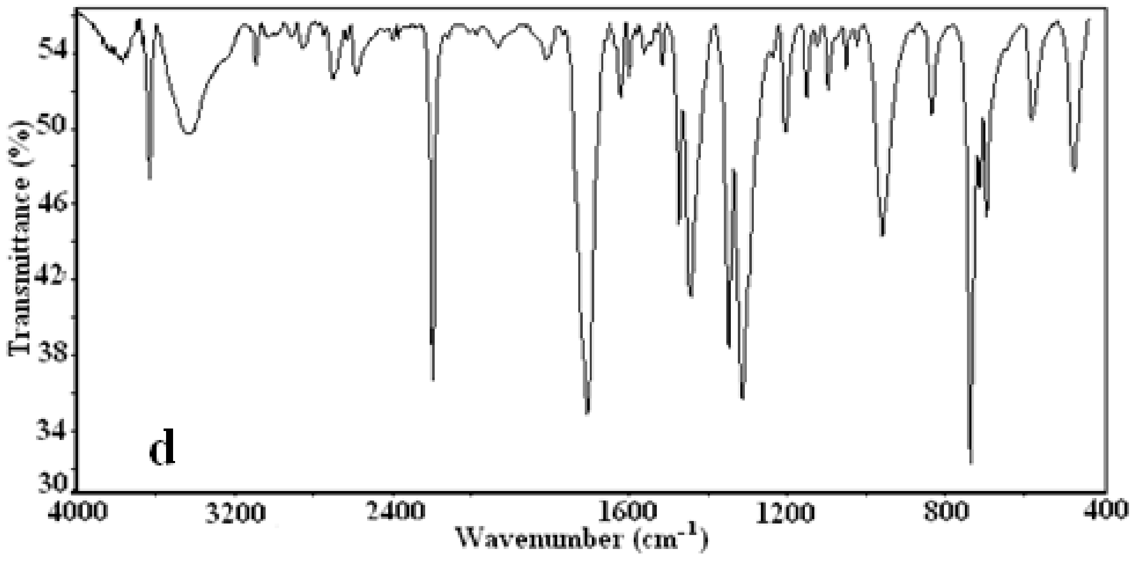


Fig. 1. The FT-IR spectra of the bulk PhCOCl (a), Co(PhCOCl)$_2$Ni(CN)$_4$ (b), the Ni(PhCOCl)$_2$Ni(CN)$_4$ (c), the Cd(PhCOCl)$_2$Ni(CN)$_4$ (d) complexes.

In this study, we use for the first time the PhCOCl molecule as a ligand to produce new Hofmann-type complexes in the of M(PhCOCl)$_2$Ni(CN)$_4$, where M is a transition metal atom Co, Ni and Cd. The molecular structure of PhCOCl is shown in Figure 2. There are various vibrational (IR and Raman) studies reported for the PhCOCl molecule [6-13].

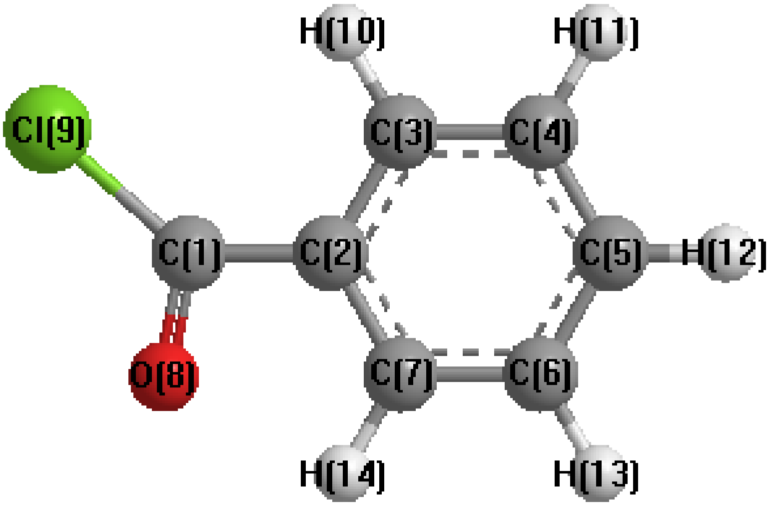


Fig. 2 The molecular structure of the PhCOCl.

The PhCOCl has a planar structure and belongs to the C_s point group. The planar conformation allows the overlap of the p orbitals in the ring and that of the carbonyl carbon. In this way, the electrons of the carbonyl group of the PhCOCl molecule have the possibility of moving in a broader region. Assuming this geometry, 36 normal vibration modes are expected to appear in the vibrational (IR and Raman) spectra. The 25 modes of these 36 normal modes are in-plane and 11 out of plane vibration. Among the 36 normal modes 30 fundamentals are due to the aromatic ring. Their distribution is as follows: 21 are in-plane and nine are out-of-plane vibrations. By the rest six normal modes the substituent group is characterized. For the –COCl group there are four in-plane vibrations: ν(C=O), ν(C-Cl), O=C-Cl in-plane scissoring and O=C-Cl in-plane rocking modes. The two out of plane modes are the O=C-Cl bending and the C-C=OCl torsion. These normal modes are observed in the liquid phase spectrum of the PhCOCl molecule [11, 12]. The frequencies of for the bulk PhCOCl vibrations and in the M(PhCOCl)$_2$Ni(CN)$_4$ (M = Co, Ni and Cd) complexes are assigned on the basis of the work of Condit et al [11]. The assignments and wavenumbers of the infrared bands for the PhCOCl molecule observed in the FT-IR spectra of the complexes are given in Table 2, together with the vibrational wavenumbers of bulk the PhCOCl. The observed small frequency shifts are due to the changes of the surround of the PhCOCl molecule and the pairing of the internal vibration of the PhCOCl molecule with the vibrations of (M–O) bond. Same small frequency shifts were reported in other studies [14, 15]. The noticeable frequency shifts were observed at frequencies of the ν(C=O), ν(C-C), X-sensitive, Ø(C-C-C) ν(C-Cl), and γ(OCCl).

The carbonyl stretching mode, ν(C=O) of the PhCOCl has been reported to be in Fermi resonance with a combination tone [6]. In 1962, Rao and Venkataraghava assigned the doublet to Fermi resonance; later the detailed description of the spectrum was also published [8]. There is agreement in the literature that the doublet at 1775 cm^{-1} and 1734 cm^{-1} caused by Fermi resonance between the (C=O) stretching and overtone of the X-sensitive mode vibration at 873 cm^{-1} in the bulk PhCOCl vibrations [6-11]. A Fermi resonance is the shifting and splitting of the energies and intensities of absorption bands in a vibrational (IR or Raman) spectrum. It is a consequence of quantum mechanical mixing. The phenomenon was explained by the physicist Enrico Fermi. The method of Langseth and Lord to correct Fermi resonance was used to obtain the unperturbed ν(C=O) frequency in the bulk PhCOCl [16]. The corrected frequency of the ν(C=O) at 1750±1.73 cm^{-1} for the bulk PhCOCl molecule shifted to low frequency region around 62–67 cm^{-1} in the complexes. Similar low frequency shifts are also observed for the other different ligands having carbonyl group in the structure [17-20]. The frequency of the ν(C-C) at 1316 cm^{-1} for the bulk PhCOCl molecule shifted to low frequency region around 24–25 cm^{-1} in the complexes. The frequency of the X-sensitive mode at 873 cm^{-1} for the bulk PhCOCl molecule shifted to low frequency region around 18–20 cm^{-1} in the complexes.

Table 2. The IR wavenumbers (cm^{-1}) of PhCOCl in the M(PhCOCl)$_2$Ni(CN)$_4$ (M = Co, Ni and Cd) complexes along with bulk PhCOCl.

Assignment, Sym. species [a]	**PhCOCl** [a]	**Co-PhCOCl-Ni**	**Ni-PhCOCl-Ni**	**Cd-PhCOCl-Ni**
2ν(C=O)	3491 w	3507 w	3501 w	3506 w
ν(C-H)	3070 m	3072 w	3072 w	3072 w
ν(C=O) [b]	1750 s	1688 s	1687 s	1683 w
ν(C-C)	1619 m	1620 w	1618 w	n.o.
ν(C-C)	1595 m	1603 w	1584 w	1582 m
ν(C-C)	1487 w	1497 w	1496 w	1496 w
ν(C-C)	1451 s	1454 m	1454 m	1453 s
β(C-H)	1343 m	1326 s	1326 s	1326 s
ν(C-C)	1316 m	1293 s	1293 s	1292 s
X-sensitive	1205 s	1213 w	1211 vw	1213 w
β(C-H)	1175 s	1184 m	1186 m	1180 m
β(C-H)	1101 w	1128 m	1128 m	1099 m
β(C-H)	1078 w	1072 m	1073 w	1072 m
β(C-H)	1027 w	1027 w	1027 w	1026 w
Ring def.	1001 w	999 w	1001 w	995 w
X-sensitive	873 s	853 w	853 vw	855 sh
γ(C-H)	775 s	772 vw	764 vw	n.o.
Ø(C-C-C)	692 m	708 s	708 s	707 s
ν(C-Cl)	665 s	684 w	684 m	682 w
γ(OCCl)	645 s	668 m	668 m	667 m
α(C-C-C)	615 m	618 w	618 w	615 sh
δ(OCCl) scissors	505 w	501 vw	503 vw	503w

s, strong; m, medium; w, weak; sh, shoulder; n.o., not observed
[a] Taken from Ref. [11].
[b] The corrected wavenumber for Fermi resonance according to Ref. [16].

The FT-IR spectral data for the PhCOCl in the complexes are consistent with all the vibrational features the *ν*(C=O) group of a coordinated ligand. The *ν*(C=O) stretching frequency should decrease due to the consecutive effects and coordination. Hence, the (C=O) bonds should become weaker. This shift shows that PhCOCl molecule is connected directly to transition metals atoms through the oxygen of the carbonyl group. These observed frequencies shifts may be interpreted as a weakening of the (C=O) bonds resulting from the electron draining of the oxygen atom on account of its bridging coordination between the metal atoms (M = Co, Ni and Cd) [20, 21].

The frequency shifts to higher frequencies were also observed for the complexes. For example, the frequency of the Ø(C-C-C) at 692 cm^{-1} for the bulk PhCOCl molecule shifted to high frequency region around 15–16 cm^{-1} in the complexes. The frequency of the *ν*(C-Cl) at 665 cm^{-1} for the bulk PhCOCl molecule shifted to high frequency region around 17–19 cm^{-1} in the complexes. The frequency of the γ(OCCl) at 645 cm^{-1} for the bulk PhCOCl molecule shifted to high frequency region around 22–23 cm^{-1} in the complexes. All of these vibrations which are sensitive to connect directly to transition metals atoms. The reason is that the bonding of the (CO–M) makes difficult the C–O deformation motion for the (C=O), the Ø(C-C-C), the *ν*(C-Cl) and the γ(OCCl) groups. Similar shifts were observed in Şenyel's and Kartal's study [17-20, 22].

It is obvious that the observed FT-IR peaks between 3613 and 3100 cm^{-1} result from the water molecules in the complexes [23]. The non covalent Ni–*π* binding force among the Ni ions with aromatic systems of the PhCOCl ligand molecules is very important in stabilizing the polymeric layers of the complexes and clathrates [20, 24].

3.2. Ni(CN)$_4$ Group Vibrations

The FT-IR wavenumbers of Ni(CN)$_4$ anions in our investigated complexes are given in Table 3 and refer to the work of McCullough and et al [25]. The diagnostic wavenumbers of the (CN) and (Ni-CN) modes are found to be similar those of Hofmann-type complexes [4, 17-20, 22, 24]. As the stretched vibrations of the CN group of the K$_2$[Ni(CN)$_4$] were observed in 2122 cm^{-1} region, the band ν(CN) E$_u$ for the complexes of M-PhCOCl-Ni shifted to a higher frequency range around 40-46 cm^{-1}. The stretched of (Ni-CN) E$_u$ mode of the K$_2$[Ni(CN)$_4$] were observed in 539 cm^{-1} region, the band of ν(Ni-CN) E$_u$ modes, for the complexes of M-PhCOCl-Ni shifted to a higher frequency range around 12-14 cm^{-1}. The same higher frequency shift range around 12–16 cm^{-1} appeared also in the in plane bending of δ(Ni-CN) E$_u$ mode, of the K$_2$[Ni(CN)$_4$ were observed in 416 cm^{-1} region for complexes. On the other hand the out of plane bending of (Ni-CN) A$_{2u}$ mode, of the K$_2$[Ni(CN)$_4$ were observed in 474 cm^{-1} region, the band of π(Ni-CN) A$_{2u}$ modes, for the complexes of M-PhCOCl-Ni shifted to a lower frequency range around 27-30 cm^{-1}. The all shifts show that both bands are connected to the transition metal atoms (M = Co, Ni and Cd). Such frequency shifts were observed and explained as the mechanical coupling of the internal modes of Ni(CN)$_4$ with the metal M-NC (M = Co, Ni and Cd) vibrations. The characteristic frequencies of Ni(CN)$_4$ group are found to be similar to those of the Hofmann-type complexes and clathrates suggesting that coordination around Ni atom is square and that $|\text{M-Ni(CN)}_4|_\infty$ layers have been conserved [4, 17-20, 22, 24]. Therefore, [Ni(CN)$_4$]$^{2-}$ anions and [M-(PhCOCl)$_2$]$^{2+}$ cations come together in square planar sheet constitution. These polymeric layers are held in parallel by Van der Waals interactions between PhCOCl molecules. The α type cavity occurs in these complexes. The cavities have volume depending on the type of transition metals. The PhCOCl molecules are located below and above the plane. Nickel atoms are surrounded by four carbon atoms of cyanide groups and M metal atoms are also surrounded by four nitrogen atoms of the cyanide groups and at the same time two oxygen atoms of carbonyl group of the PhCOCl molecules are bonded to the metal atoms in the regular square plane. The proposed shape of M-PhCOCl-Ni complexes is shown in Figure 3.

Table 3. The IR wavenumbers (cm^{-1}) of the [Ni(CN)$_4$]$^{2-}$ group in the M(PhCOCl)$_2$Ni(CN)$_4$ (M = Co, Ni and Cd) complexes.

Assignment [a]	**K$_2$[Ni(CN)$_4$]** [a]	**Co-PhCOCl-Ni**	**Ni-PhCOCl-Ni**	**Cd-PhCOCl-Ni**
ν(CN), E$_u$	2122	2162 s	2168 s	2165 s
Hot bant	2118	2122 sh	2129 sh	2124 sh
ν(Ni-CN), E$_u$	539	552 m	553 m	551 m
π(Ni-CN), A$_{2u}$	474	444 sh	447 sh	445 sh
δ(Ni-CN), E$_u$	416	432 m	431 m	428 m

s, strong; m, medium; w, weak; sh, shoulder; ν, stretching; π, out-of-plane bending; δ, in plane bending
[a] Taken from Ref. [25].

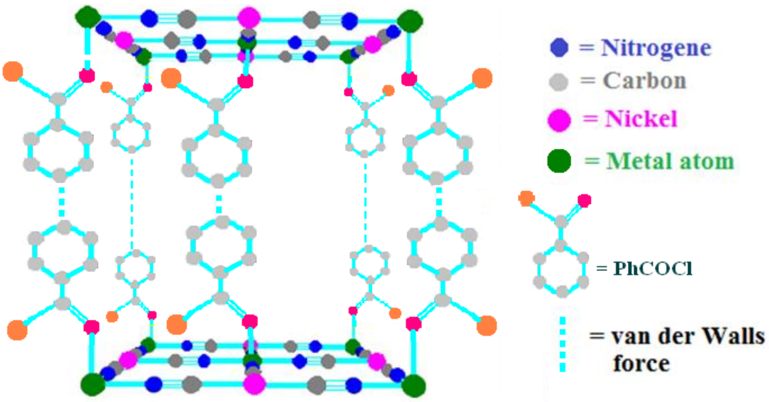


Fig. 3. Structure of the proposed shape of M-PhCOCl-Ni complexes.

3.3. Thermal Behavior of Complexes

The TGA, DTA and DTG graphics of all the complexes are similar and M(PhCOCl)$_2$Ni(CN)$_4$ (M = Co, Ni and Cd) complexes are shown in Fig. 3 as examples. The TGA results indicate that samples are stable at room temperature. By heating, however, all of complexes gradually lose their water molecules between about (69–81) °C and (98-116) °C. The water molecules in the cavities of complexes leave in the first temperature range. Then the water molecules bounded weakly to the complexes leave the complexes in the second temperature range. The decomposition temperatures of M(PhCOCl)$_2$Ni(CN)$_4$ (M = Co, Ni and Cd) complexes are given in Table 4. The Table 4 shows two decomposition stages. The first decomposition stages around (143–219) °C indicate that the PhCOCl molecules leaves from the M(PhCOCl)$_2$Ni(CN)$_4$ (M = Co, Ni and Cd) complexes. The final decomposition stage around (382–400) °C is the decomposition of cyanide to yield the respective metal oxide. The results show the formation of the Hofmann-type complex.

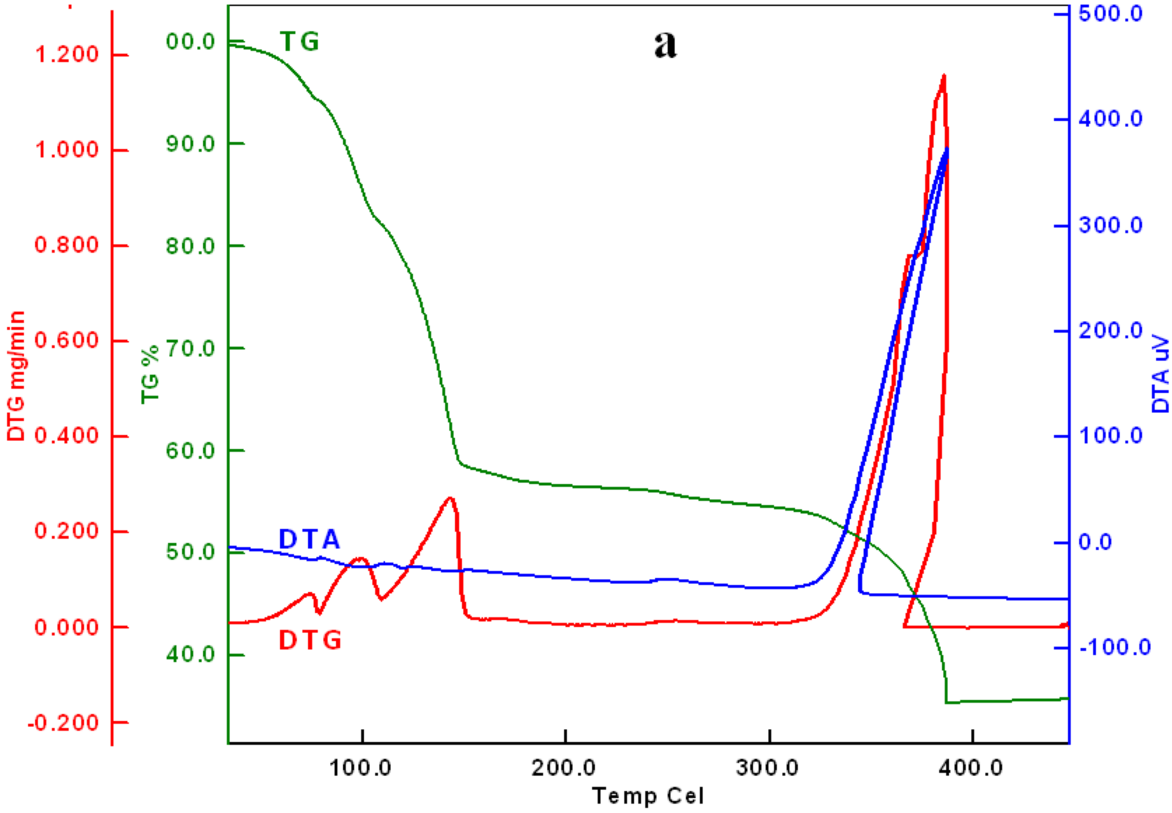
Table 4. The decomposition temperatures of the M(PhCOCl)$_2$Ni(CN)$_4$ (M = Co, Ni and Cd) complexes.

Sample	**Decomposition (°C)**			
	I	**II**	**III**	**IV**
Co(PhCOCl)$_2$Ni(CN)$_4$	73	98	143	382
Ni(PhCOCl)$_2$Ni(CN)$_4$	69	116	217	400
Cd(PhCOCl)$_2$Ni(CN)$_4$	81	116	219	398

Similar decomposition stages have been observed for other Hofmann-type complexes [26, 27 and 29] and Hofmann-type clathrates [20, 24, 28 and 30].

4. CONCLUSION

The explanation given above indicates that the complexes obtained in this study are new examples of Hofmann-type complexes. The FT-IR spectroscopic and thermo-gravimetric studies of three new Hofmann type complexes have shown that they have similar structures consisting of infinite two-dimensional polymeric layers formed with Ni(CN)$_4$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DTG mg/min
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1.000
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0.600
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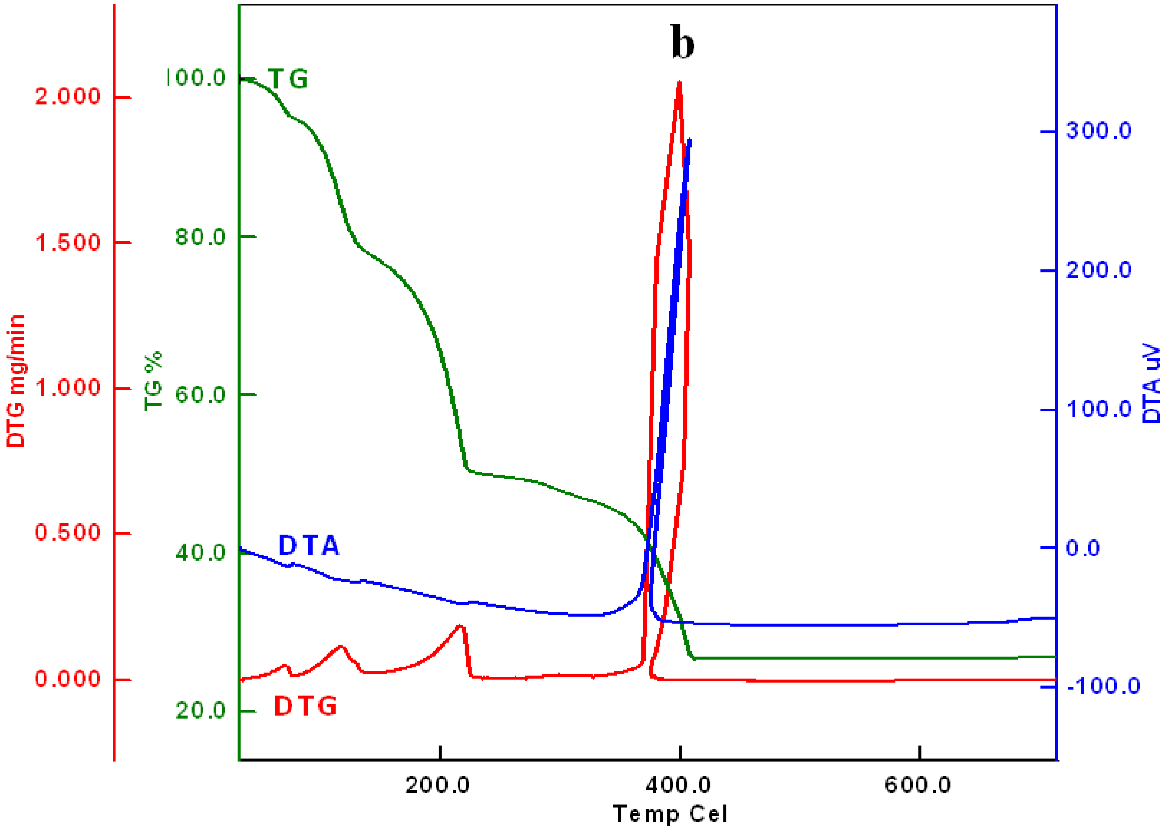
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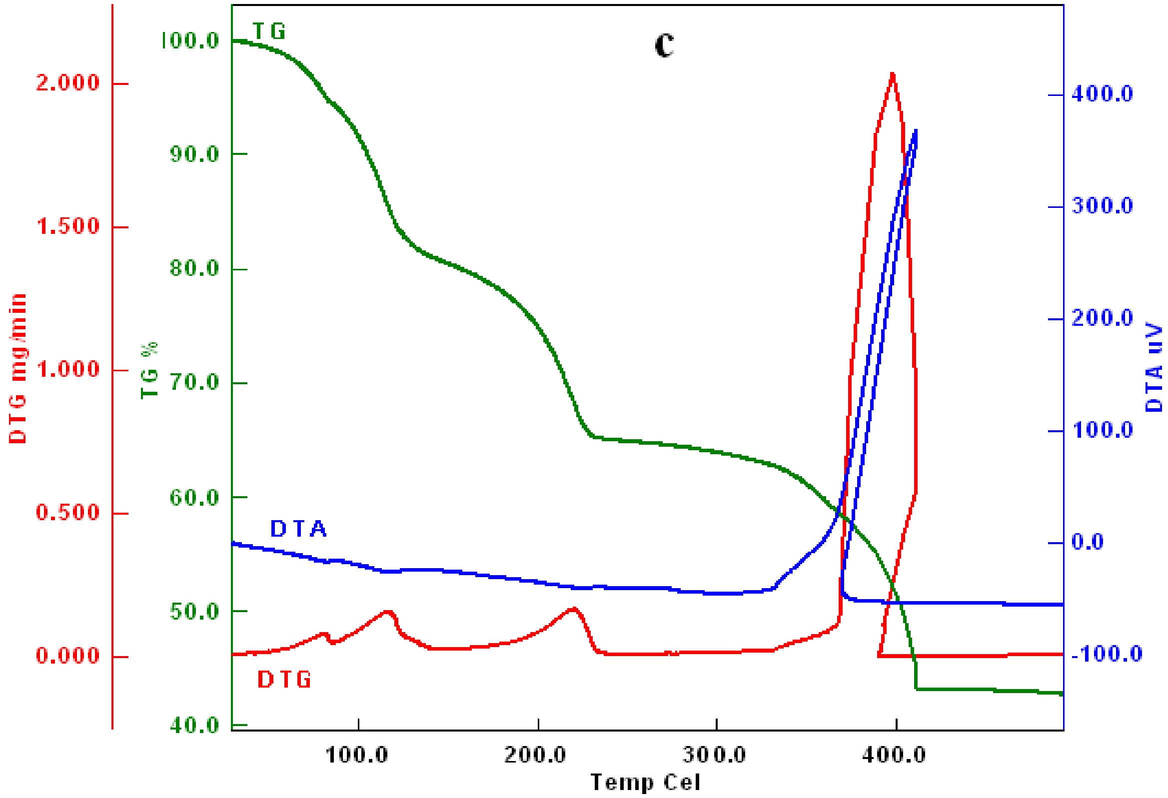

Fig. 3. The TGA, DTA and DTG curves of $Co(PhCOC)_2Ni(CN)_4$ (a), the $Ni(PhCOC)_2Ni(CN)_4$ (b), the $Cd(PhCOC)_2Ni(CN)_4$ (c) complexes.

ions bridged by $M(PhCOCl)_2$ (M = Co, Ni and Cd) cations. The PhCOCl molecules in complexes have been coordinated as a monodentate ligand bonding only through the oxygen atoms of the carbonyl group. These polymeric layers are held in parallel by Van der Walls interactions between the PhCOCl molecules. The compounds $M(PhCOCl)_2Ni(CN)_4$ (M = Co, Ni and Cd) are new examples of the Hofmann-type complexes.

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DUMLUPINAR ÜNİVERSİTESİ
KÜTAHYA
1992