

## Vibrational Spectroscopic Study of Creatinine Hofmann-T<sub>d</sub>-Type Complexes

CELAL BAYRAK

Department of Physics Education, Hacettepe University, 06800 Beytepe, Ankara, Turkey

Corresponding author: E-mail: cbayrak@hacettepe.edu.tr

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New Hofmann-T<sub>d</sub>-type complexes in the form of M(creat)<sub>2</sub>M'(CN)<sub>4</sub> (M = Mn and Cd; M' = Zn, Cd and Hg; creat = creatinine = 2-amino-1-methyl-5H-imidazol-4-one) were prepared in powder form and their FT-IR (4000-400 cm<sup>-1</sup>), far-IR (400-50 cm<sup>-1</sup>), FT-Raman (4000-50 cm<sup>-1</sup>) spectra and elemental analyses are reported. Creatinine molecules are found to involve coordination through one of the imidazole ring nitrogen atoms. The spectral features of the compounds studied are found to be each other indicating that they have analogous structures.

**Key Words:** Creatinine, FT-IR, Far-IR, FT-Raman spectra, Tetracyanometallate.

### INTRODUCTION

Creatinine (C<sub>4</sub>H<sub>7</sub>N<sub>3</sub>O) is produced from creatine, a molecule of major importance for energy production in muscles. Creatinine is transported through the bloodstream to the kidneys. The kidneys filter out most of the creatinine and dispose of it in the urine<sup>1</sup>. An abnormal level of creatinine in biological fluids is an indicator of various disease states<sup>2</sup>. Creatinine has two tautomeric forms, the imine (I) and the amine (II) (Fig. 1). *Ab initio* calculations have shown that in the gas phase, the imine form is preferred but the solid state, the amine form is observed and is predicted theoretically<sup>3-5</sup>. The fundamental vibrations for the creatinine molecule have assigned using the generalized valence force field approximation<sup>6</sup>. More recently, the study of complexes of this bioligand are attracting considerable interest. The X-ray crystal structure and the coordination chemistry with transition metal ions of creatinine have been widely studied<sup>6-11</sup>. Although the infrared spectra of Pt(creat)<sub>2</sub>(NO<sub>2</sub>)<sub>2</sub>, Pt(creat)<sub>2</sub>(ClO<sub>4</sub>)<sub>2</sub> have been completely analyzed<sup>6</sup>, no complete spectral characterizations have been done on the other complexes. Muradlihan *et al.*<sup>12</sup> investigated M(creat)<sub>2</sub>X<sub>2</sub> (M = Zn, Cd or Hg, X = Cl, Br or I) complexes using IR, NMR and TG. They only reported M-N and M-Cl frequencies for these complexes. In our previous study we reported the spectra of metal halogen complexes of creatinine M(creat)<sub>2</sub>X<sub>2</sub> (M = Zn, Cd or Hg, X = Cl or Br) and it was concluded that coordination through imidazol ring nitrogen<sup>13</sup>.

We have also prepared five new complexes of the form M(creat)<sub>2</sub>M'(CN)<sub>4</sub> (M = Mn or Cd; M' = Zn, Cd or Hg). These complexes are analogous to the Hofmann-T<sub>d</sub>-type complexes<sup>14-18</sup>

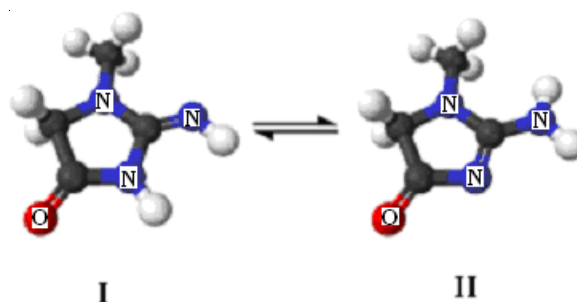


Fig. 1. Imine (I) and amine (II) tautomeric forms of creatinine

and clathrates<sup>19-21</sup>. In these structures, the host framework is formed from the infinite -Cd-L<sub>2</sub>-Cd- chains extending along the a- and b-axes alternately and tetrahedral M'(CN)<sub>4</sub> ions arranged between the consecutive crossing -Cd-L<sub>2</sub>-Cd- chains with the connections of the N-ends bound to the Cd atoms in the chains<sup>14-21</sup>. The compounds possessing this type of the host framework reported to date were confined to the Mn or Cd metal atom in an octahedral environment and to the Cd or Hg metal atom in a tetrahedral group<sup>16-21</sup>.

### EXPERIMENTAL

**Preparation of complexes:** The complexes Mn-creat-M (M = Zn, Cd or Hg) were synthesized by adding 2 mmol of creatinine and 1 mmol of K<sub>2</sub>M(CN)<sub>4</sub> solution in water. The precipitate formed was filtered, washed with water, ethanol and ether successively and kept in a dessicator. The Cd-creat-Cd and Cd-creat-Hg complexes were prepared using a method analogous to that given in the literature<sup>16-21</sup>.

The freshly prepared compounds were also analyzed for C, H and N by a LECO CHNS-932 analyzer with the following results (found %/calcd. %).  $\text{Cd}(\text{C}_4\text{H}_7\text{N}_3\text{O})_2\text{Cd}(\text{CN})_4$ : C = 42.73/42.94, H = 4.35/4.46, N = 14.11/14.31;  $\text{Cd}(\text{C}_4\text{H}_7\text{N}_3\text{O})_2\text{Hg}(\text{CN})_4$ : C = 42.73/42.94, H = 4.35/4.46, N = 14.11/14.31;  $\text{Mn}(\text{C}_4\text{H}_7\text{N}_3\text{O})_2\text{Zn}(\text{CN})_4$ : C = 42.73/42.94, H = 4.35/4.46, N = 14.11/14.31;  $\text{Mn}(\text{C}_4\text{H}_7\text{N}_3\text{O})_2\text{Cd}(\text{CN})_4$ : C = 42.73/42.94, H = 4.35/4.46, N = 14.11/14.31;  $\text{Mn}(\text{C}_4\text{H}_7\text{N}_3\text{O})_2\text{Hg}(\text{CN})_4$ : C = 35.78/37.34, H = 3.47/3.87, N = 12.29/12.44. The analytical results were agreement with the proposed formula.

**Physical measurements:** The FT-IR spectra recorded between 4000-400  $\text{cm}^{-1}$  on Perkin Elmer 1330 and Mattson 1000 FT-IR spectrometers, which were calibrated using indene and polystyrene film. The samples were prepared as mulls in nujol and KBr pellets. Far-infrared (400-50  $\text{cm}^{-1}$ ) spectra between polyethylene plates as Nujol mulls of the compounds were recorded *via* a Bruker Optics IFS66v/s FT-IR spectrometer with 2  $\text{cm}^{-1}$  resolution in vacuum. FT-Raman spectra of the compounds were recorded using a Bruker Senterra Dispersive Raman microscope spectrometer with 532 or 633 nm excitations from a 3B diode laser having 3  $\text{cm}^{-1}$  resolution in the region of 4000 and 50  $\text{cm}^{-1}$ .

## RESULTS AND DISCUSSION

The FT-IR, FT-Raman and far-IR spectra of  $\text{Cd}(\text{creat})_2\text{Cd}(\text{CN})_4$  and  $\text{Mn}(\text{creat})_2\text{Zn}(\text{CN})_4$  compounds are given in Figs. 2-4, respectively. Because of the lack of structural data on the compounds studied, the assignment was made by treating the ligand molecules and the  $\text{M}(\text{CN})_4$  ( $\text{M} = \text{Zn}, \text{Cd}$  or  $\text{Hg}$ ) ions as isolated units. The wavenumbers and assignments made are given for ligand molecules and  $\text{M}(\text{CN})_4$  ions in Tables 1 and 2, respectively, together with some relevant spectral data for comparison.

**Creatinine (creat) vibrations:** Creatinine (2-amino-1-methyl-5H-imidazol-4-one) has 15 atoms and 39 normal modes. It has methyl, methylene, amino and C=O groups. The methyl group as a united atom in the creatinine molecule considered by Trendafilova *et al.*<sup>6</sup> and 30 vibrational modes discussed. Costa *et al.*<sup>7</sup> reported some selected IR bands of creatinine. The detailed vibrational assignments of fundamental modes of creatinine along with the calculated at B3LYP levels using the triple split valence basis set along with diffuse and polarization functions, 6-311++G (d,p) and normal mode

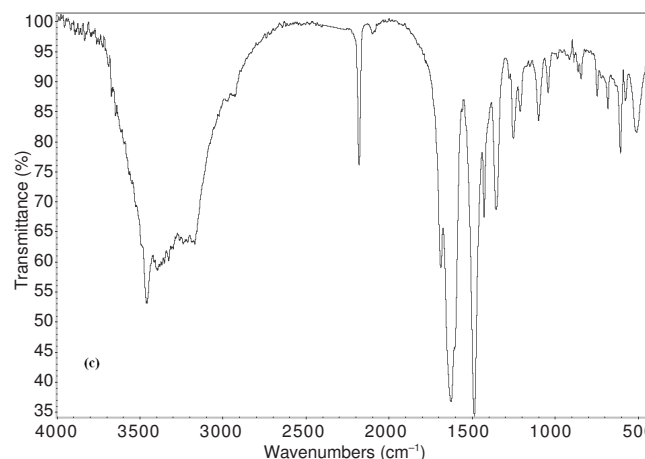
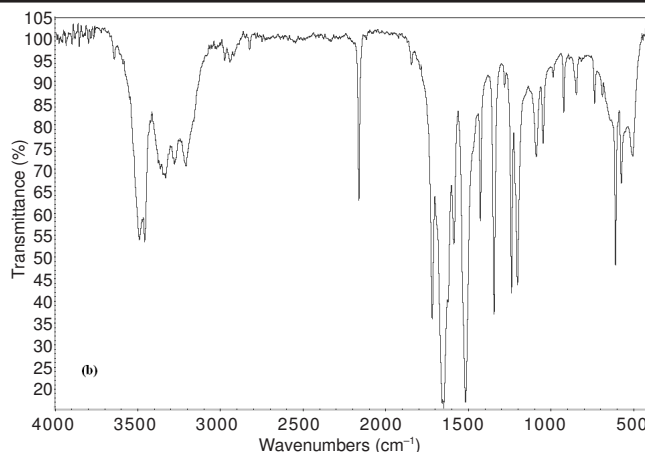
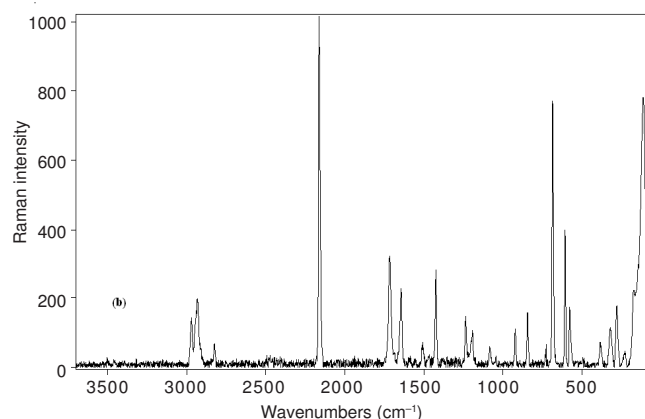
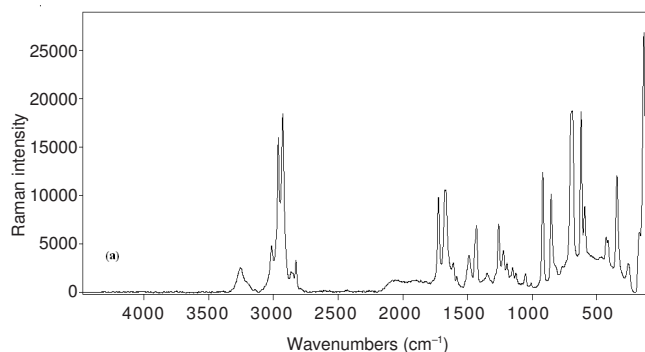
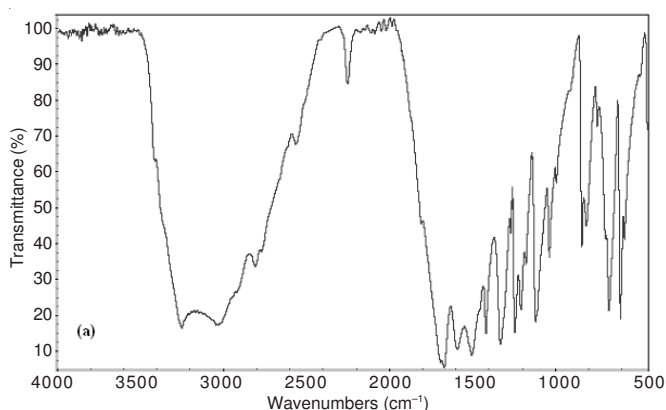


Fig. 2. FT-IR spectra of the creatinine (solid) (a), Cd-creat-Cd (b) and Mn-creat-Zn (c) complexes



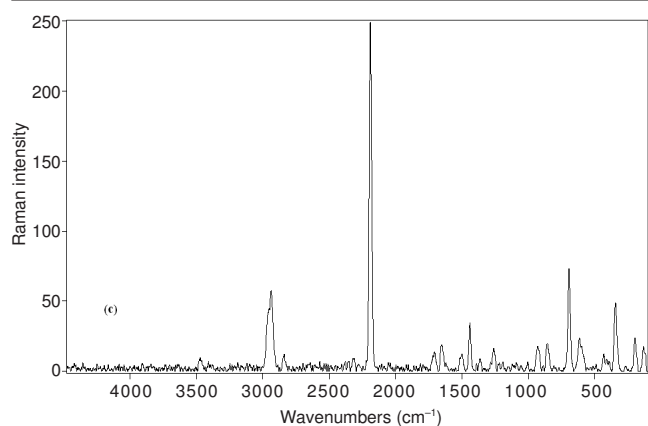


Fig. 3. FT-Raman spectra of the creatinine (solid) (a), Cd-creat-Cd (b) and Mn-creat-Zn (c) complexes

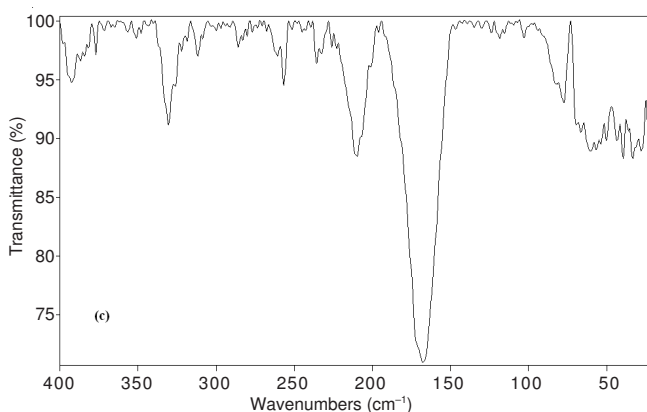
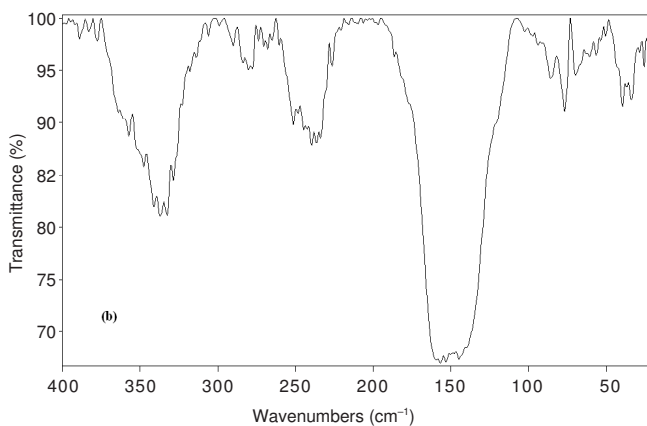
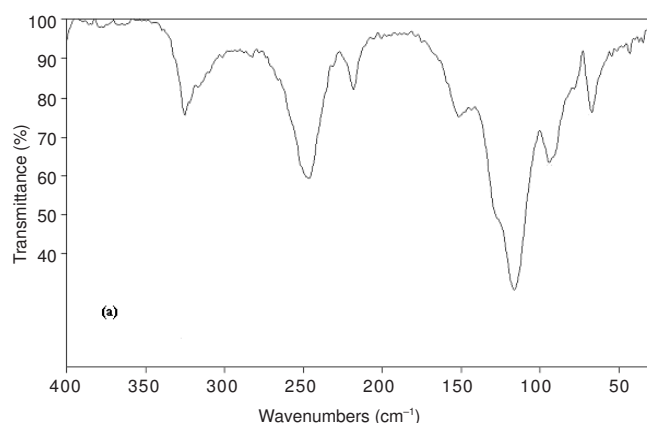


Fig. 4. Far-IR spectra of the creatinine (solid) (a), Cd-creat-Cd (b) and Mn-creat-Zn (c) complexes

descriptions (characterized by TED) based on SQM force field calculations are reported of Bayrak *et al.*<sup>13</sup> The FT-IR, FT-Raman and far-IR spectra of creatinine is illustrated in Figs. 2a, 3a and 4a.

Creatinine can coordinate through the ring nitrogen, the C=O and/or -NH<sub>2</sub> groups. In metal complexes, creatinine typically coordinates to the metal *via* the ring nitrogen<sup>6-8,11,22</sup>. The N-H stretching vibration of NH<sub>2</sub> group are much affected and found to shift to lower wavenumbers on coordination<sup>23,24</sup>. The shifts to lower wavenumbers of the  $\nu(\text{C}=\text{O})$  in the complexes can be attributed to the effect of coordination through the oxygen atoms of these groups to the metal<sup>25</sup>. When the aromatic ring nitrogen coordinates to metal, the ring stretching wavenumbers shift to higher wavenumbers<sup>25</sup>. In order to determine the coordination site of creatinine in  $\text{M}(\text{creat})_2\text{M}'(\text{CN})_4$  ( $\text{M} = \text{Mn}$  or  $\text{Cd}$ ;  $\text{M}' = \text{Zn}$ ,  $\text{Cd}$  or  $\text{Hg}$ ) complexes, the wavenumbers of creatinine in complexes are compared with those of free creatinine. Some selected fundamental modes of complexes are reported in Table-1.

We observed four broad bands corresponding to stretching vibrations  $\nu(\text{NH}_2)$  and their wavenumbers are found to be higher in value than those of free creatinine. A positive shift of these absorptions is usually regarded as signifying that the ligand is not NH<sub>2</sub>-bonded. This band indicates the presence of creatinine in the metal halogen complexes in its amine form and the rather broad character of the NH<sub>2</sub> vibration bands is suggestive of H bond participation<sup>26</sup>. In addition, NH<sub>2</sub> scissoring mode of creatinine is observed at 1670 cm<sup>-1</sup> for creatinine and around 1650 cm<sup>-1</sup> in the FT-IR spectra (1645 and 1626 cm<sup>-1</sup> FT-Raman, respectively) for complexes. These results suggested that the NH<sub>2</sub> groups of creatinine are not involved in the coordination with the metal ions and are in good agreement with those reported in the literature<sup>6,7</sup>.

The  $\nu(\text{C}=\text{O})$  mode is observed at 1692 cm<sup>-1</sup> for free creatinine and around 1720 cm<sup>-1</sup> in the infrared spectra and at 1716 cm<sup>-1</sup> in the FT-Raman spectra of complexes, indicating that the ligand does not coordinate to the metal ions through (C=O) group. These bands in the FT-IR spectra at 1503, 1208, 1177, 841, 813, 677 and 608 cm<sup>-1</sup> with ring contribution exhibit intensity changes and shift to higher (1517, 1210, 1192, 923, 847, 688 and 610 cm<sup>-1</sup> FT-IR, respectively) wavenumbers in complexes. In our previous study we observed the ring stretching mode in the Raman spectra at 1151 cm<sup>-1</sup> and the ring deformation mode 852 cm<sup>-1</sup> in free creat molecules. On the other hand, these modes at 1151 and 852 cm<sup>-1</sup> are observed at 1192 and 923 cm<sup>-1</sup> in FT-Raman. The above mentioned complexes, the ring bending mode complexes at 687 (very strong band) and 610 cm<sup>-1</sup> (medium band) observed in the FT-Raman spectra (Table-1). All of these data suggest binding between the metal(II) and the ring N atom of the creatinine. Similar shifts have been observed in metal-coordinated creat compounds<sup>6-13</sup>.

It is clear from Table-1 that most of the vibrational modes of coordinated creatinine in the complexes have increased in wavenumbers when compared with uncoordinated creatinine. These shifts may be explained as the coupling of M-N(creat) vibrations. Similar observations have been previously for creatinine metal halogen complexes<sup>6,13</sup> pyridine nitrogen complexes<sup>13,15,16</sup> and imidazole nitrogen clathrate<sup>19</sup>.

TABLE-1  
 VIBRATIONAL WAVENUMBERS (cm<sup>-1</sup>) OF CREATININE IN METAL COORDINATION COMPLEXES

Modes	Creatinine <sup>a</sup>			Cd-creat-Cd		Cd-creat-Hg		Mn-creat-Zn		Mn-creat-Cd		Mn-creat-Hg		TED %
	v <sub>exp.</sub>		v <sub>calc.</sub>	IR	Ra	IR	Ra	IR	Ra	IR	Ra	IR	Ra	
	IR	Ra	IR											
39	3252vs	3256w	3565	3451vs		3491vs		3463vs	3469vw	3460vs	3469vw	3461vs	3469vw	100 v <sub>a</sub> (NH <sub>2</sub> )
38	3030vs	3015m	3454	3186vs						3200vs	3189vw	3205vs	3189vw	100 v <sub>s</sub> (NH <sub>2</sub> )
37	2980vw	2963s	2990											100 v <sub>a</sub> (CH <sub>3</sub> )
36			2965	2945vw	2970 w		2972 w					2969vw		100 v <sub>a</sub> (CH <sub>2</sub> )
35	2922w	2928s	2959	2907vw	2933w	2944vw	2931w	2907vw	2938w	2906vw	2938w	2907vw	2938w	100 v(CH <sub>3</sub> )
34		2863w	2909											100 v <sub>s</sub> (CH <sub>2</sub> )
33	2809w	2826w	2875	2827vw	2824vw	2824vw	2824vw	2838vw	2839vw	2836vw	2839vw	2824vw	2839vw	100 v <sub>s</sub> (CH <sub>3</sub> )
32	1692sh	1724s	1730	1721vs	1716m	1718s	1717m	1689s	1706w	1685s	1706w	1718s	1706w	65 v(C=O)+11 v(CN) <sub>ring</sub>
31	1670vs	1673s	1664	1649vs	1645w	1655vs	1643w	1626vs	1654w	1630vs	1654w	1635vs	1654w	51 δ(NH <sub>2</sub> )+27 δ(HNC)+11 v(C-NH <sub>2</sub> )
30	1590s	1609w	1581	1588s		1589vw		1586s		1587s		1587vw		37 v(CN) <sub>ring</sub> +20 v(CNH <sub>2</sub> )+13 v(C=O)+10 δ(HNC)
29	1503s	1489w	1518	1517vs	1507vw	1518vs	1506vw	1508vs	1505w	1509vs	1506w	1504s	1503w	50 v(CN) <sub>ring</sub> +15 v(C-NH <sub>2</sub> )+10 δ(C-NH <sub>2</sub> )
28	1457sh		1474	1427w	1425m	1427m	1423m	1433w	1440w	1435w	1443w	1434w	1441w	64 δ(CH <sub>2</sub> )+12 δ <sub>ring</sub> +10 τ(H <sub>2</sub> C-NC-CH <sub>3</sub> )
27			1462											55 δ(CH <sub>3</sub> )+32 τ(CN-CH <sub>3</sub> )
26	1418s	1430m	1443	1345vw		1345m		1378vw	1364w	1377vw	1360w	1374vw	1366w	53 δ(CH <sub>3</sub> )+30 τ(CN-CH <sub>3</sub> )
25	1331s	1349w	1391					1322s		1324s		1321s		50 δ(CH <sub>3</sub> )+46 δ(N-CH <sub>3</sub> )
24	1269w	1259m	1290	1281vw				1279vw		1261w		1259w		35 δ(CH <sub>2</sub> )+32 τ(CN-CH <sub>2</sub> )+17 v(N-CH <sub>3</sub> )
23	1243s	1221w	1254	1240s	1237w	1239m	1236w							38 v <sub>ring</sub> +30 v(N-CH <sub>3</sub> )+18 δ(N-CH <sub>3</sub> )
22	1208m	1194w	1200	1210m		1210m		1219m		1217m		1221m		45 v <sub>ring</sub> +28 δ(CH <sub>2</sub> )+13 δ(C=O)
21	1177w	1151w	1182	1192w	1193vw	1159w	1191vw	1190m	1192w	1191m	1193w	1192m	1189w	33 v <sub>ring</sub> +17 δ(C-NH <sub>2</sub> )+15 δ(N-CH <sub>3</sub> )+14 v(NCH <sub>3</sub> )
20	1118s		1143	1091w	1084vw		1084vw	1082m		1081m		1083m		46 δ(CH <sub>2</sub> )+30 τ(NC-CH <sub>2</sub> )
19	1036m	1051w	1096	1048w		1048w		1044w	1004w	1041w	1006w	1042w	1005w	72 δ(N-CH <sub>3</sub> )+15 τ(CN-CH <sub>3</sub> )
18			1086											60 δ(C-NH <sub>2</sub> )+15 v <sub>ring</sub> +10 δ(C=O)
17			1015											44 δ(N-CH <sub>3</sub> )+35 v(CN) <sub>ring</sub>
16	992w	917s	979	988vw		988vw		988vw	930w	982vw	932w	984vw	933w	26 τ(C=O)+21 δ(C-CH <sub>2</sub> )+16 τring+13 τ(ring-CH <sub>2</sub> )
15	841s	852s	849	923vw	923w	923vw	920w	907vw	856w	904vw	855w	908vw	851w	63 v <sub>ring</sub> +16 δ <sub>ring</sub> +10 δ(C-NH <sub>2</sub> )
14	813m		801	847w	845w	856w	848w	834w	807w	838w	805w	835w	806w	40 v(CN) <sub>ring</sub> +17 v(N-CH <sub>3</sub> )+14 δring+10 v(C-NH <sub>2</sub> )
13	747mw	689s	733	736w	727vw	736vw	725vw	747vw	794w	743vw	791w	745vw	797w	50 ω(NH <sub>2</sub> )+30 τ <sub>ring</sub>
12	677vs	662s	661	688w	687vs	689w	688vs	681w	694w	680w	692w	683w	695w	43 δ <sub>ring</sub> +15 v(C-NH <sub>2</sub> )+11 v <sub>ring</sub> +10 v(C=O)
11	608vs	593m	606	610m	608s	610m	609s	665m	616w	663m	617w	666m	612w	32δ <sub>ring</sub> +18 v(N-CH <sub>3</sub> )+10 v <sub>ring</sub> +10 δ(C=O)
10	583mw		577	574m	579m	579w	577m	609w		607w		610w		30 δ(C=O)+28 τ(ring-CH <sub>2</sub> )+14 τ(C=O)+12 δ(NC-NH <sub>2</sub> )
9	455vw		531	505m		507m		509w	489m	507w	491m	511w	485m	20 δ(NC-NH <sub>2</sub> )+17 τ(ring-NH <sub>2</sub> )+13 τ(ring-CH <sub>2</sub> )+10 τ(C=O)
8	406m	427w				426s		406vw	411w	404vw	413w	407vw	414w	71 τ(ring-NH <sub>2</sub> )
7	325m	344s	350	328m	322w	387w	321w	330m	343w	332m	345w	333m	345w	87 τ(ring-NH <sub>2</sub> )
6	246s	257w	314	318m	281w	237w	285w	318w	269w	317w	271w	319w	272w	48 δ(NC-NH <sub>2</sub> )+20 δ(C=O)+13v <sub>ring</sub>
5	218m		291	215w	230w	204m	234w	223	196w	223	196w	223	196w	58 δ(CN-CH <sub>3</sub> )+15 τ(ring-NH <sub>2</sub> )
4	151mw	135vs	160	153vs	173w	157m	177w	167s	134w	166s	131w	169s	135w	27 τ(ring-CH <sub>3</sub> )+24 τ(ring-CH <sub>2</sub> )+15 τ(ring-NH <sub>2</sub> )
3	116vs		123		114s	134m	116s	116w		114w		115w		37 τ(C=O)+22 τ <sub>ring</sub> +13 τ(ring-NH <sub>2</sub> )
2	94mw		101	85w		79w		103w		108w		106w		90 τ(ring-CH <sub>3</sub> )
1	67mw		85	77w	73w	59w	76w	77w		78w		79w		46 τ(ring-CH <sub>3</sub> )+28 τ(H <sub>3</sub> C-ring-NH <sub>2</sub> )

<sup>a</sup>Taken from Ref.-13. (TED: The total energy distributions). vs: very strong, s: strong, m: medium, w: weak, vw: very weak, sh: shoulder.

TABLE-2  
VIBRATIONAL WAVENUMBERS (cm<sup>-1</sup>) OF CYANIDE GROUP FOR THE M-creat-M' COMPLEXES

Assignment <sup>a</sup>	K <sub>2</sub> Zn(CN) <sub>4</sub> <sup>a</sup>	K <sub>2</sub> Cd(CN) <sub>4</sub> <sup>a</sup>	K <sub>2</sub> Hg(CN) <sub>4</sub> <sup>a</sup>	Cd-creat-Cd	Cd-creat-Hg	Mn-creat-Zn	Mn-creat-Cd	Mn-creat-Hg
v <sub>1</sub> (CN) A <sub>1</sub>	(2157)	(2145)	(2146)	(2163vs)	(2164vs)	(2180vs)	(2185vs)	(2187vs)
v <sub>5</sub> (CN) F <sub>1</sub>	2152	2145	2146	2163vs	2164vs	2172vs	2170vs	2170vs
v <sub>2</sub> (MC) A <sub>1</sub>	(347)	(327)	(335)	(359w)	(361w)	(357w)	(358w)	(356w)
v <sub>6</sub> [v(MC)+δ(NCM)]F <sub>2</sub>	359	316	330	353s	350s	355s	354s	353s
v <sub>7</sub> [v(MC)+δ(NCM)]F <sub>2</sub>	315	250	235	261w	261w	270w	267m	268m

The band observed in the Raman spectra are in given parentheses. <sup>a</sup> Taken from Ref.-27.

**M'(CN)<sub>4</sub> (M = Zn, Cd or Hg) group vibrations:** In assigning the bands attributable to M'(CN)<sub>4</sub> (M' = Zn, Cd or Hg) ions in the spectra of our compounds, we refer to the work of Jones<sup>27</sup> who presented vibrational data for the salts K<sub>2</sub>M(CN)<sub>4</sub> (M = Zn, Cd or Hg) in the solid phase and assigned the infrared and Raman active fundamental vibrations of the M(CN)<sub>4</sub> ion on the basis of T<sub>d</sub> symmetry. The assigned wavenumbers for the M'(CN)<sub>4</sub> groups in the compounds studied appear to be much higher than those for M(CN)<sub>4</sub> groups in K<sub>2</sub>M(CN)<sub>4</sub> (M = Zn, Cd or Hg) (Table-2). Such frequency shifts have been observed for other T<sub>d</sub>-type host complexes<sup>15-18</sup> and T<sub>d</sub>-type clathrates<sup>14,19-21</sup>, in which both ends of the CN group are coordinated and explained as the mechanical coupling of the internal modes of M'(CN)<sub>4</sub> (M' = Zn, Cd or Hg) with the M-NC vibrations. It follows that the N-ends of the M'(CN)<sub>4</sub> groups are also bound to a M atom in present complexes.

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