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An Infrared Spectroscopic Study on the Hofmann-diam-type 1,9-Diaminononanemetal(II) Tetracyanonickelate(II)-Aromatic Guest Clathrates: $M(H_2N(CH_2)_9NH_2)Ni(CN)_4\cdot G$ (M = Cd or Ni; G = Benzene, Naphthalene, Anthracene or Phenanthrene

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Abstract

IR spectra of M(1,9-Diaminononane)$Ni(CN)_4\cdot G$ (M = Cd or Ni; G = benzene, naphthalene, anthracene or phenanthrene) are reported. The spectral data suggest that the host structures in these clathrates are similar to those of Hofmann-α, ω-diaminoalkane type clathrates.

Introduction

A number of Hofmann-diam-type clathrates $Cd[NH_2(CH_2)_nNH_2]Ni(CN)_4\cdot x$Guest have been prepared and their x-ray structures determined [1, 2]. Of these, the structure of $Cd[NH_2(CH_2)_9NH_2]Ni(CN)_4\cdot$ 0.5o-xylene [1] reveals a three-dimensional host framework built of the layers of a two dimensional catena-[cadmium tetra-μ-cyanonickelate] network and of the ambidentate 1,9-diaminononane (danon) bridging the Cd atoms in adjacent networks. The guest o-xylene molecule is accommodated in the cavity formed in the interlayer space. The skeleton of the danon in the Cd-NC_9N-Cd linkage takes a $(gauche)_2(trans)_8$ conformation twisted at the $(gauche)_2$ part. The twisted $(gauche)_2$ part of the danon skeleton occupies the interlayer space to such an extent that the stoichiometric number of the guest per formula unit of the host is 0.5. The model of the Hofmann-diam-type host structure is schematically illustrated in Figure 1.

We have now prepared eight compounds of the form M(1,9-diaminononane)$Ni(CN)_4\cdot G$ (M = Cd or Ni; G = benzene (bz), naphthalene (npt), anthracene (ant) or phenanthrene (pht), (abbr. M-danon-Ni-G)) and their infrared spectra are reported. It should be noted that several unsuccessful attempts have been made to synthesize the clathrate $Cd[NH_2(CH_2)_9NH_2]Ni(CN)4\cdot$ 0.5o-xylene according to the method given by Hashimoto *et al.* [1] and the clathrates with biphenyl guest.

Experimental

All chemicals were reagent grade (Merck) and were used without further purification. The clathrates M-danon-Ni-G (M = Cd or Ni) were synthesized by adding 3 mmol of G and 1 mmol of danon solution in ethanol and 1 mmol of $K_2Ni(CN)_4$ in solution in water to a 1 mmol solution of MCl_2 (M = Cd or Ni) in water while stirring. The precipitate formed was filtered, washed with water, ethanol and ether, successively and kept in a desiccator containing saturated guest vapour.

The freshly prepared compounds were analysed for C, H and N with the following results (found%/calculated%).

$Cd(C_9H_{22}N2)Ni(CN)_4\cdot C_6H_6$: C = 43.82/44.61, H = 5.18/5.52, N = 15.44/16.43
$Ni(C_9H_{22}N2)Ni(CN)_4\cdot C_6H_6$: C = 49.06/49.84, H = 5.72/6.16, N = 17.20/18.35
$Cd(C_9H_{22}N2)Ni(CN)_4\cdot C_{10}H_8$: C = 48.78/49.19, H = 5.36/5, 38 N = 14.21/14.96
$Ni(C_9H_{22}N2)Ni(CN)_4\cdot C_{10}H_8$: C = 54.30/54.38, H = 5.46/5.95, N = 15.97/16.54
$Cd(C_9H_{22}N2)Ni(CN)_4\cdot C_{14}H_{10}$: C = 52.44/53.01, H = 5.21 /5.27, N = 13.61/13.74
$Ni(C_9H_{22}N2)Ni(CN)_4\cdot C_{14}H_{10}$: C = 57.55/58.12, H = 5.67/5.78, N =14.83/15.06
$Cd(C_9H_{22}N2)Ni(CN)_4\cdot C_{14}H_{10}$: C =53.07/53.01, H = 4.84/5.27, N = 14.26/13.74
$Ni(C_9H_{22}N2)Ni(CN)_4\cdot C14H_{10}$: C = 57.96/58.12, H = 5.36/5.78, N = 14.64/15.06

These analytical results are often poor for the samples obtainable in powder form owing to partial decomposition. The infrared spectra were recorded between 4000-400 cm^{-1} on a Mattson 1000 FTIR spectrometer, which was calibrated using polystyrene and an indene/camphor/cyclohexane standard solution. The samples were prepared as mulls in nujol and hexachlorobutadiene and as KBr discs.

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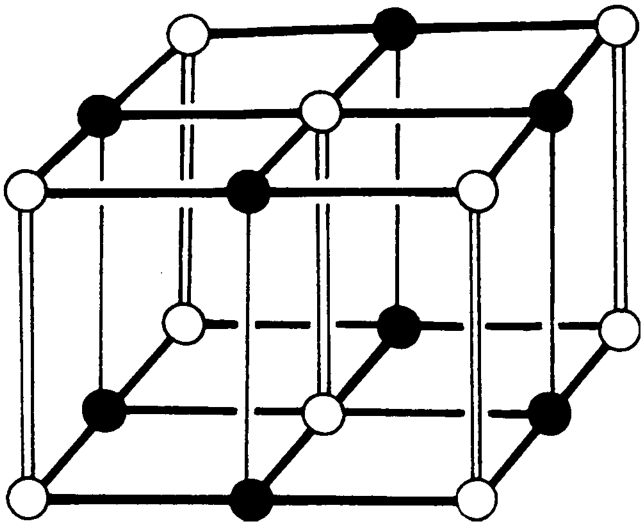

Figure 1. The model for the Hofmann-diam-type host structure (open circle: 6-coordinate M, solid circle: square-planar Ni, open column: an ambident ligand, thick line : CN bridge and thin line : edge of cavity.

Results and discussion

Before embarking upon the vibrational features of the compounds, a word may be said about the number of guest molecules accommodated. In spite of our efforts to trap more guest molecules into the cavities of the present clathrates, we have only obtained the composition $M(danon)Ni(CN)_4 \cdot G$ (M = Cd or Ni). The decrease of the number of guest molecules from 2 in the Hofmann-en-type clathrates [2] to 1 in our case may be due to the flexibility of the host framework with long diamine ligands.

The spectral features of the host moities in the clathrates with similar M atoms (Cd or Ni) are found to be similar to each other, suggesting that they also have similar structural features. But the spectral features of the Cd-danon-Ni host group differ substantially from those of the Ni-danon-Ni host group implying that the conformation of the ligand molecules and the distortion in the two dimensional $[M\text{-}Ni(CN)_4]_\infty$ sheets are different in the two groups of clathrates.

Owing to the lack of structural data the assignment was made by treating the 1,9-diaminononane ligand, the guest molecules and the $Ni(CN)_4$ ions as isolated units. The wavenumbers of the bands arising from the $Ni(CN)_4$ units, from the diamine ligand and from the guest species are tabulated in Tables 1–4 respectively, together with some relevant spectral data for comparison.

$Ni(CN)_4$ group vibrations

The fact that in the host framework of the Hofmann-type clathrates and Hofmann-type host complexes [3] the metal atom in $Ni(CN)_4$ is surrounded by four C atoms of CN groups in a square-planar environment suggests that the host framework of the clathrates studied here also have square-planar $Ni(CN)_4$ units. In order to assign the bands attributable to the $Ni(CN)_4$ ion in the spectra of our compounds, we refer to the work of McCullough *et al.* who

Table 1. The vibrational wavenumbers (cm^{-1}) of the $Ni(CN)_4$ group for the M-danon-Ni-G clathrates

Assignment[a]	$Na_2Ni(CN)_4$[a]	Cd-Ni-G G = Benzene	Ni-Ni-G G = Benzene	Cd-Ni-G G = Naphthalene	Ni-Ni-G G = Naphthalene	Cd-Ni-G G = Anthracene	Ni-Ni-G G = Anthracene	Cd-Ni-G G = Phenanthrene	Ni-Ni-G G = Phenanthrene
$\nu_8(CN)$, E_u	2132	2148s	2160vs	2148vs	2166vs	2152s	2160vs	2150vs	2166vs
Hot band?	2128	–	–	–	–	–	–	–	–
$\nu_9(NiC)$, E_u	543	523w	–	542w	–	538w	–	–	494w
$\pi(NiC)$, A_{2u}	448	470vw	470vw	–	476w	472w	469w	461vw	474w
$\delta(NiCN)$, E_u	433	422s	438vs	424vs	440vs	424m	436vs	424vs	440vs

[a] Taken from Ref. [7]. v = very, s = strong, m = medium, w = weak.

Table 2. The vibrational wavenumbers (cm^{-1}) of 1,9-diaminononane in the M-danon-Ni-G clathrates

Assignment[a]	Danon in CCl_4	Cd-Ni-G G = Benzene	Ni-Ni-G G = Benzene	Cd-Ni-G G = Naphthalene	Ni-Ni-G G = Naphthalene	Cd-Ni-G G = Anthracene	Ni-Ni-G G = Anthracene	Cd-Ni-G G = Phenanthrene	Ni-Ni-G G = Phenanthrene
$\nu_a(NH_2)$	3361vs	3349vs	3356vs	3340s	3357vs	3338vs	3344vs	3344vs	3343vs
$\nu_s(NH_2)$	3325vs	3290vs	3296vs	3286s	3296vs	3292vs	3286s	3294vs	3284s
$\nu_a(CH_2)$	2926vs	2924vs	2922vs	2924vs	2922vs	2921vs	2924vs	2923vs	2924vs
$\nu_s(CH_2)$	2856vs	2852vs	2852vs	2854vs	2847vs	2853vs	2856vs	2853vs	2843vs
$\delta(NH_2)$	1581vs	1587m	1587vs	1581s	1581s	1587m	1583s	1581s	1583vs
$\delta(CH_2)$	1489vs	1456s	1460vs	1462vs	1464vs	1464vs	1463vs	1466vs	1469vs
$\delta(CH_2)$	1472vs	–	1472s	–	1470m	–	–	–	–
$\rho_w(CH_2)$	1390s	–	–	–	–	–	–	–	–
$\rho_w(CH_2)$	–	1377s	1377s	1373s	1377s	1377vs	1377vs	1376vs	1377vs
$\rho_t(CH_2)$	1313m	1302vw	1307m	1308vw	–	–	1311m	1311m	1298m
$\rho_t(NH_2)$	1225w	1205w	1205w	1213w	1214vw	1205vw	1207vw	1205m	1203m
$\nu(CN)$	1097vw	1109m	1106vw	–	–	1097m	1107s	1109m	1103s
$\nu(CN)$	1070w	1068s	1067s	1068s	–	1068m	1067m	1068s	1069m
$\rho_w(NH_2)$	–	–	1014w	1014m	1010m	1020m	1014s	1018vs	1012vs
$\rho_r(CH_2)$	931m	966m	964vw	962m	958w	960m	966m	964s	962s
$\rho_r(CH_2)$	877m	866s	854m	863s	866w	860s	876s	864s	864m
$\rho_r(CH_2)$	721m	717m	723s	721s 721m	723m	727s	721s	725vs	
δ(skeletal)	576m	577m	575m	573m	586m	577m	588m	577m	573m

[a] Taken from Ref. [20]. v = very, s = strong, m = medium, w = weak.

presented vibrational data for the Ni(CN)$_4$ ion in the salt Na$_2$Ni(CN)$_4$ in the solid state [7]. In the salt Ni(CN)$_4$ ions are not coordinated to K ions; therefore, they can be treated as isolated units with D_{4h} symmetry and thus used as references to comment on vibrational changes when M(Cd or Ni)–NC coordination takes place. The infrared spectral data for Ni(CN)$_4$ groups are given in Table 1, along with the spectral data of Na$_2$Ni(CN)$_4$ [7]. The assigned wavenumbers of the Ni(CN)$_4$ units of the clathrates studied appear to be much higher than that for the isolated Ni(CN)$_4$ ion in Na$_2$Ni(CN)$_4$ (Table 1). Such frequency shifts have been observed for Hofmann-type clathrates [4–6] and Hofmann-type complexes [8–11], and are attributed to the mechanical coupling of the internal modes of Ni(CN)$_4$ with the metal M–NC vibrations.

1,9-diaminonane vibrations

Up to the time of writing, there have been no reports of the spectral data for the danon molecule. However, relevant vibrational spectral data exist for some other α,ω-diaminoalkanes: ethylenediamine in the liquid phases [12] and in the en-Td-type benzene clathrates [13], 1,3-trimethylenediamine in solution in CCl$_4$ and in the tn-Td-type clathrates [14], 1,4-diaminobutane as a liquid and in the Hofmann-type clathrates [15], 1,5-diaminopentane as a liquid and in the Hofmann-type clathrates [16], 1,6-diaminohexane as a liquid and in the Hofmann-type clathrates [17], 1,7-diaminoheptane as a liquid and in the Hofmann-type clathrates [18], 1,8-diaminooctane as a liquid and in the Hofmann-type clathrates [19].

The assignments and the wavenumbers of the fundamental bands observed in the infrared spectra of the compounds under study are given in Table 2, together with our spectral data for danon in solution in CCl$_4$ and in the solid phase, for comparison. From Table 2, it is clear that the assignments of the fundamental bands in the 4000-1800 cm^{-1} region due to the stretching vibrations of N–H and CH bands are straightforward while the assignments of the bands in the 1800-400 cm^{-1} region, only on the basis of our experimental data, are tentative. The notable spectral features are the following:

The NH$_2$ stretching frequencies of the danon molecules in the clathrate compounds are found to be lower than those of danon in the CCl$_4$ solution (Table 2). These downward frequency shifts may be explained as weakening of the N–H bonds resulting from the electron draining from the two N atoms on account of their bridging coordination between the two consecutive metal M (Cd or Ni) atoms. The other notable feature is that the asymmetric and symmetric stretching vibrations of the NH$_2$ groups appear as two single sharp bands. There are no signs of splitting for these bands. The absence of splitting implies that the ligand molecule in the compounds behaves as a bridging ligand (bidentate coordination).

Based on the present spectral data, it is not possible to describe the conformation of the danon molecules in the host framework in our clathrate compounds. However, some implications may be deduced: the spectral features of the danon molecules in the clathrates are found to be similar suggesting similar conformations. The compositions of the present compounds suggest that one of the two cavities are occupied by the skeletal carbon-chain instead of a guest molecule in such a way that the bridging diamine ligand takes an appropriate skeletal conformation.

Table 3. The vibrational wavenumbers (cm^{-1}) of benzene in the M-danon-Ni-G clathrates

Assignment[a]	Liquid Benzene[a]	Cd-Ni-G, G = Benzene	Ni-Ni-G, G = Benzene
ν_{20}, E_{1u}	3073	3080vw	3080vw
$\nu_8 + \nu_{19}$	3075	3065w	3066vw
B_{13}, B_{1u}	3048	3029w	3032w
$\nu_5 + \nu_{17}$, E_{1u}	1955	1964vw	1967vw
$\nu_{10} + \nu_{17}$	1815	1829w	1821w
ν_{19}, E_{1u}	1479	1476s	1472vs
ν_{14}, B_{2u}	1309	1302w	1307s
ν_{15}, B_{2u}	1149	1147m	1136s
ν_{18}, E_{1u}	1036	1038w	1036s
ν_{17}, E_{2u}	966	966m	964w
ν_{11}, A_{2u}	670	688m	681vs
	–	–	–
ν_{16}, E_{2u}	404	–	–

[a] Taken from Ref. [21]. v = very, s = strong, m = medium, w = weak.

Guest vibrations

The assignments and the frequencies of the bands arising from the enclathrated benzene, naphthalane, anthracene and phenanthrene are tabulated in Table 3 and Table 4, respectively, together with some relevant spectral data for comparison. The most structurally informative spectral features are the following.

Most of the vibrational bands of the isolated benzene molecules are observed in the infrared spectra of the M-danon-Ni-Bz (M = Cd or Ni) clathrates (Table 3), suggesting that the selection rules for the benzene molecule under its intrinsic symmetry (D_{6h}) are lost. This is possibly due to the lower site symmetry of the benzene molecules in the clathrates.

The CH out-of-plane mode (A_{2u}) in the spectra of the clathrates are found to be shifted to higher frequency (688 cm^{-1} for Cd-danon-Ni-Bz and 681 cm^{-1} for Ni-danon-Ni-Bz) (Table 3) from that of benzene in solution in CCl$_4$ (675 cm^{-1}) [19] or from that of liquid benzene (670 cm^{-1}) [21]. Similar positive frequency shifts were observed for Hofmann-type [4, 5, 8] and Td-type [13, 14] clathrates. Akyüz *et al.* [5, 6] explained this upward shift by the presence of a weak hydrogen bond between π electrons located above and below the plane of the benzene ring and the amine groups of the host lattice. Therefore, we may reasonably suggest that the frequency shifts in our compounds are due to the π-electron donation from the benzene ring to the hydrogen atom of the NH$_2$ group in the ligand molecule which has a more electrophilic character caused by coordination. These

Table 4. The vibrational wavenumbers (cm^{-1}) and symmetry species of naphthalene, anthracene and phenanthrene in the M-danon-Ni-G clathrates

Naphthalene[a]	Naphthalene in		Anthracene[b]	Anthracene[b]	Anthracene in		Phenanthrene[c]	Phenanthrene[c]	Phenanthrene in	
(Solid)	Cd-danon-Ni	Ni-danon-Ni	(theory)	(in matrix)	Cd-danon-Ni	Ni-danon-Ni	(theory)	(gas phase)	Cd-danon-Ni	Ni-danon-Ni
3072(B_{3u})	3070w	3068w	3078(B_{2u})	3067–3068m	3080w	3084w	3093(A_1)	–	3092w	3094w
?	3049w	3047w	3063(B_{1u})	3055, 3062 m	3064w	3064w	3082(B_2)	–	3081w	3081w
3014(B_{2u})	3015vw	3012v	3044(B_{1u})	3032w	3043vw	3048w	3071(B_2)	–	3072vw	3071w
2976(B_{3u})	2978w	2979w	3039(B_{1u})	3017, 3022w	3040w	3037vw	3064(A_1)	3061vs	3064vw	3062vw
2947(B_{2u})	2953vw	2953vw	1620(B_{1u})	1627w	1620m	1620m	3057(A_1)	–	3056w	3056vw
1680(B_{3u})	1677w	1680vw	1534(B_{2u})	1540, 1542w	–	1533m	3045(A_1)	–	3046vw	3048w
1595(B_{2u})	1591vw	1591vw	1456(B_{1u})	1450w	1454m	1453m	1595(A_1)	1602w	1581s	1583s
1510(B_{3u})	1504vw	1507vw	1455(B_{2u})	1460w	1463m	1463m	1497(B_2)	1496w	1498s	1498s
1387(B_{2u})	1386w	1385w	1343(B_{2u})	1346 vw	1345vw	1347vw	1462(B_2)	1452m	1466s	1469s
1268(B_{2u})	1268m	1267w	1311(B_{1u})	1318 w	–	1311m	1444(A_1)	–	–	–
1211(B_{3u})	1213m	1214vw	1275(B_{1u})	1272w	1279w	1282m	1250(A_1)	1239w	1244m	1242m
1139(B_{2u})	1130m	1128vw	1169(B_{2u})	1167, 1169vw	1160w	1165w	1038(B_2)	1032m	1038s	1038s
1012(B_{3u})	1014m	1010m	1158(B_{2u})	–	–	–	950(B_1)	940, 998w	957s	962s
957(B_{1u})	962m	958w	1156(B_{1u})	1149, 1151w	1149m	1147w	871(B_1)	859m	864s	864m
876(B_{3u})	–	879vw	1001(B_{2u})	1001w	1006w	1007w	817(B_1)	806s	816s	816s
782(B_{1u})	796vs	791s	962(B_{3u})	955, 958 w	960m	966m	737(B_1)	729 vs	752s	737s
752(B_{2u})	757vw	–	908(B_{1u})	908 vw	909vw	910vw	628(B_2)	615w	–	–
618(B_{3u})	619vw	619w	885(B_{3u})	879s	–	876s	498(B_1)	486w	496w	494w
475(B_{1u})	478m	476m	730(B_{3u})	726, 729vs	723s	727s	431(B_1)	–	425s	–
			613(B_{3u})	603w	602w	603m				
			471(B_{3u})	468, 470m	472m	469m				

[a] Taken from Ref. [30].
[b] Taken from Ref. [31].
[c] Taken from Ref. [31].

experimental observations are also consistent with the theory of orbital following during molecular vibrations [22–26].

Another essential feature of the out-of-plane CH bending vibration (A_{2u}) is that it appears as a very intense single band in the spectra of the compounds (Table 3). A similar single band was observed in the infrared spectra of Cd(4,4′-bipyridyl) $(CN)_4 \cdot 2C_6H_6$ (M′ = Cd or Hg) [10]. This vibrational mode splits into a doublet in Cd(pyrazine) $(CN)_4 \cdot C_6H_6$ [27] and $M(NH_3)_2\ (CN)_4 \cdot 2C_6H_6$ (M = Mn or Cd, M′ = Cd or Hg) [28] and into a triplet in M(ethylenediamine) $(CN)_4 \cdot$ 2C6H6 (M = Mn or Cd, M′ = Cd or Hg) [13]. In the case of clathrates with triplet or doublet features, the splittings have been ascribed to crystal field effects (strong host-guest interactions) [13]. In the case of clathrates with a single band, because of the larger cavities due to the ligands, the host-guest interactions are expected not to be effective for splitting [13, 14, 27].

Based on the spectral data presented, it is not possible to determine the orientation of the benzene molecules in the host lattice. The relative orientation of the N–H bonds of danon with respect to the axis of the π cloud of benzene must be the one most favourable for hydrogen bonding [29].

The out-of-plane C–H vibrations in the other guest molecules show lower positive frequency shifts (Table 4). These shifts are decreasing in the sequence of benzene, naphthalene, anthracene and phenanthrene (Table 3, 4). These observations are due to the bulkiness of the guest molecules.

The preceding discussion considered as a whole leads us the conclusion that the host lattices of the our clathrates are similar to those of the other Hofmann-α, ω- dialkylamine-type clathrates [1–3, 14–17].

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