



Vibrational Spectroscopic Studies on the Hofmann Type Clathrates: M(1,6-diaminohexane)Ni(CN)₄.G (M = Co, Ni or Cd; G = chlorobenzene, 1,2-, 1,3- or 1,4-dichlorobenzene)

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Abstract

New Hofmann-diaminohexane(dahxn)-type clathrates of the form M(1,6-dahxn)Ni(CN)₄.G (M = Co, Ni or Cd; G = chlorobenzene, 1,2-, 1,3 or 1,4-dichlorobenzene) were prepared in powder form and their infrared spectra are reported. The spectral data suggest that these compounds are similar in structure to those of the Hofmann-diam-type clathrates. Their structure consists of planar polymeric layers, {M–Ni(CN)₄}_∞, formed from Ni(CN)₄ anions coordinated to the bridging 1,6-diaminohexane molecules bound directly to the metal (M). The M atoms are bound to four N atoms of the CN ions and, the Ni atoms are surrounded by four C atoms of the CN groups in a square-planar layer.

Introduction

A series of three-dimensional host lattices, Cd(α,ω -diaminoalkane)Ni(CN)₄, have been developed from the two-dimensional Hofmann-type host lattices, Cd(NH₃)₂Ni(CN)₄, by replacing the ammonia molecules by bidentate ligands [1–5]. Bidentate α,ω -diaminoalkane ligands with long carbon chains give three-dimensional host structures by bridging between the Cd metal atoms in the adjacent two-dimensional {Cd–Ni(CN)₄}_∞ layers [1–3]. In the previous studies, Iwamoto *et al.* determined the crystal structures of the Cd(dahxn)Ni(CN)₄.G (dahxn = 1,6-diaminohexane, G = *o*-toluidine [2], and *p*-toluidine [3] in the monoclinic space group P2/m, G = *m*-toluidine [4] in the monoclinic space group P1) by single crystal X-ray diffraction methods. These crystal structure studies show that the clathrates in this series are not isomorphous with each other, but they have Hofmann-type structures and may be comparable to the Hofmann-type and analogous series which are isostructural. In our previous papers [6, 7], the infrared (IR) spectra of the Hofmann-diam-type compounds, M(diam)Ni(CN)₄.nG (M: metal atom and G: guest molecule), showed that their structures consist of corrugated polymeric layers {M–Ni(CN)₄} with the diam molecules bound to the metal, similar to the host framework of Hofmann-type clathrates [6, 7, 10, 11].

In the present work, we report the infrared spectra of newly prepared M(1,6-diaminohexane)Ni(CN)₄.G (M = Co, Cd or Ni; G = chlorobenzene, 1,2-, 1,3- and 1,4-dichlorobenzene) (abbreviated M–Ni–G or M–Ni–Cl, M–Ni–2Cl, M–Ni–3Cl and M–Ni–4Cl) clathrates. The infrared

wavenumbers of the diaminohexane molecule relative to its interaction with different guest molecules and to the presence of the different transition metals are given in detail.

Experimental

All chemicals used were reagent grade (Merck) and used without further purification. The compounds M–Ni–G were prepared by adding slightly more than one mmol of 1,6-diaminohexane to one mmol of K₂Ni(CN)₄ and one mmol of MCl₂ solution in water saturated with the guest molecules. The precipitate was filtered, washed with water, ethanol and ether, successively, and kept in a desiccator containing molecular sieve and saturated guest molecule vapour.

IR spectra of the compounds were recorded in the range of 4000–400 cm⁻¹ on a Mattson 1000 FTIR spectrometer which was calibrated using polystyrene film and indene solution. The samples were prepared as mulls in Nujol and hexachlorobutadiene between CsI windows and as KBr discs.

The freshly prepared compounds were analyzed for C, H and N by a Leco CHNS-932 analyzer and the following results (found%/calculated%) were obtained:

- Co(C₆H₁₆N₂)Ni(CN)₄.C₆H₅Cl:
C = 41.51/42.66, H = 4.42/4.70, N = 18.80/18.65
- Ni(C₆H₁₆N₂)Ni(CN)₄.C₆H₅Cl:
C = 41.36/42.68, H = 4.51/4.70, N = 18.88/18.66
- Cd(C₆H₁₆N₂)Ni(CN)₄.C₆H₅Cl:
C = 37.37/38.13, H = 4.02/4.20, N = 16.24/16.67
- Co(C₆H₁₆N₂)Ni(CN)₄.(1,2-C₆H₄Cl₂):
C = 38.61/39.62, H = 4.06/4.16, N = 17.49/17.33

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Table 1. The vibrational wavenumbers (cm^{-1}) of 1,6-diaminohexane (dahxn) in the M-Ni-G compounds

Assignment ^a	Liquid dahxn ^b	Ni-Ni-G ^b	Co-Ni-Cl	Cd-Ni-Cl	Ni-Ni-Cl	Ni-Ni-2Cl	Cd-Ni-2Cl	Ni-Ni-2Cl	Co-Ni-3Cl	Cd-Ni-3Cl	Ni-Ni-3Cl	Co-Ni-4Cl	Cd-Ni-4Cl	Ni-Ni-4Cl
ν (NH ₂)	3377s	3348s	3343s	3350s	3347s	3044s	3348s	3346s	3350s	3351s	3350s	3361s	3354s	3348s
ν (NH ₂)	3286s	3288s	3284s	3290m	3289m	3284m	3287m	3286m	3290m	3291m	3291m	3289m	3293m	3290m
ν (CH ₂)	2927vs	2927vs	2924m	2920vs	2924vs	2918vs	2919s	2920vs	2924s	2923vs	2923m	2923s	2923vs	2916vs
ν (CH ₂)	2854vs	2860vs	2854vs	2853vs	2856vs	2850s	2852s	2852vs	2853m	2856m	2853m	2854m	2855s	2854m
δ (NH ₂)	1578s	1587m	1575w	1580m	1574w	1579m	1584m	1582m	1586m	1586m	1585m	1578s	1586m	1579s
δ (CH ₂)	1485s	1495w	1496w	1489w	1489w	1489w	1487w	1487w	1489w	1491vw	1489w	1489w	1489vw	1495vw
δ (CH ₂)	1437w	1443vw	1437vw	1426vw	-	1436m	1436m	1435w	1437m	1437m	1437w	1437w	1439w	1437w
ρ_w (CH ₂)	1389w	1383vw	1391vw	1383vw	1382vw	1392vw	1394vw	1390vw	1393vw	1403w	1403w	1403vw	1403vw	1403vw
ρ_w (CH ₂)	1338w	1338vw	1340w	1337w	1336vw	1339vw	1338vw	1335vw	1338vw	1340vw	1338vw	1339vw	1338vw	1342w
ρ_r (NH ₂)	1305vw	-	1305w	1301vw	1307vw	1302vw	1305vw	1307w	1294vw	1303vw	1306vw	1294w	1307vw	1306vw
ν (CN)	1075m	1092m	-	-	-	1081m	1069s	1083s	-	1078m	1072m	-	1070m	-
ρ_w (NH ₂)	1052vw	1066w	1057w	1054vw	1056w	1056w	1054vw	1057w	1051w	-	1055vw	1051w	-	1054w
?	-	1016vw	-	-	-	1019vw	1018vw	1019vw	-	1013vw	-	-	1009w	-
ρ_r (CH ₂)	975vw	972m	961m	959s	970s	961s	954m	965s	-	-	-	-	-	-
ρ_r (CH ₂)	832m,br	868vw	847w	859w	859vw	851vw	846vw	831vw	847w	848vw	847w	845w	845vw	850m
ρ_r (CH ₂)	725w	736w	739w	728w	748s	731m	739m	728s	722s	723m	726m	731s	723m	729w

^a Taken from Ref. [8].^b Ni(1,6-diaminohexane)Ni(CN)₄benzene[6].

v = very strong, s = strong, m = medium, w = weak, br = broad.

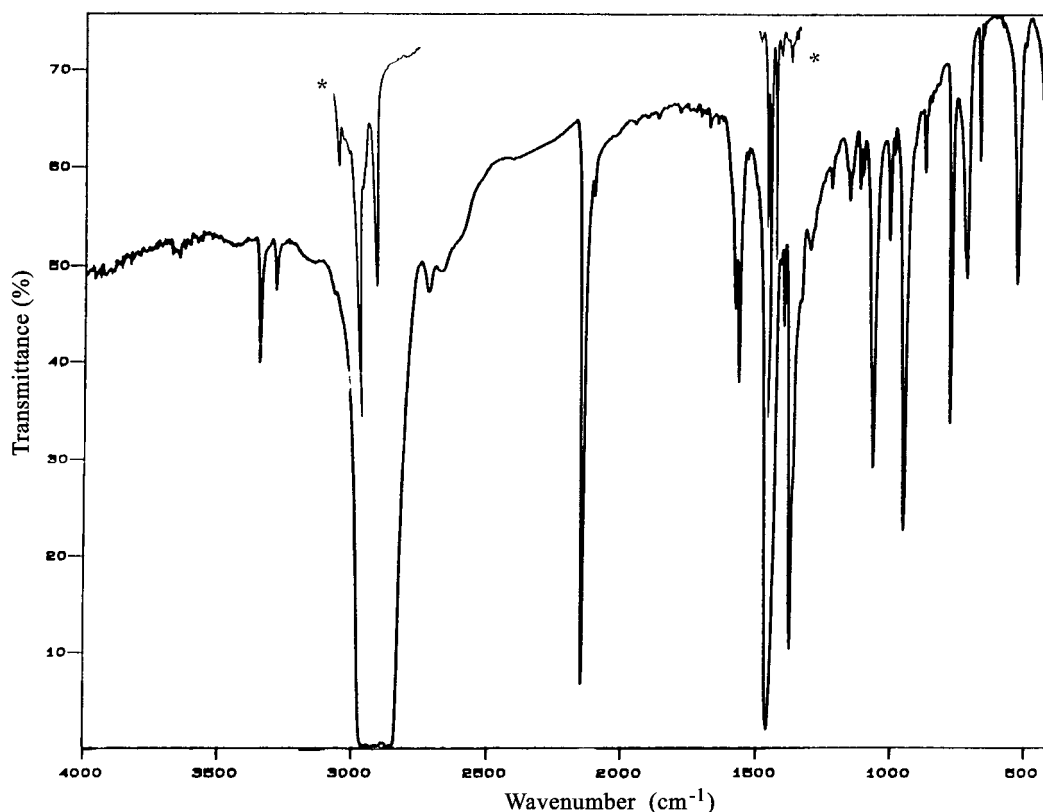


Figure 1. The Infrared spectrum of the $\text{Cd}(\text{dahxn})\text{Ni}(\text{CN})_4 \cdot 1,3\text{-dichlorobenzene}$ compound in nujol (*in a KBr disc).

- $\text{Ni}(\text{C}_6\text{H}_{16}\text{N}_2)\text{Ni}(\text{CN})_4 \cdot (1,2\text{-C}_6\text{H}_4\text{Cl}_2)$:
C = 38.88/39.64, H = 4.00/4.16, N=17.55/17.34
- $\text{Cd}(\text{C}_6\text{H}_{16}\text{N}_2)\text{Ni}(\text{CN})_4 \cdot (1,2\text{-C}_6\text{H}_4\text{Cl}_2)$:
C = 34.98/35.69, H=3.45/3.74, N=15.68/15.61
- $\text{Co}(\text{C}_6\text{H}_{16}\text{N}_2)\text{Ni}(\text{CN})_4 \cdot (1,3\text{-C}_6\text{H}_4\text{Cl}_2)$:
C = 39.12/39.62, H=4.21/4.16, N=17.10/17.33
- $\text{Ni}(\text{C}_6\text{H}_{16}\text{N}_2)\text{Ni}(\text{CN})_4 \cdot (1,3\text{-C}_6\text{H}_4\text{Cl}_2)$:
C = 38.86/39.64, H=3.96/4.16, N=17.56/17.34
- $\text{Cd}(\text{C}_6\text{H}_{16}\text{N}_2)\text{Ni}(\text{CN})_4 \cdot (1,3\text{-C}_6\text{H}_4\text{Cl}_2)$:
C = 35.45/35.69, H=3.98/3.74, N=15.89/15.61
- $\text{Co}(\text{C}_6\text{H}_{16}\text{N}_2)\text{Ni}(\text{CN})_4 \cdot (1,4\text{-C}_6\text{H}_4\text{Cl}_2)$:
C = 38.96/39.62, H=4.10/4.16, N=17.45/17.33
- $\text{Ni}(\text{C}_6\text{H}_{16}\text{N}_2)\text{Ni}(\text{CN})_4 \cdot (1,4\text{-C}_6\text{H}_4\text{Cl}_2)$:
C = 39.14/39.64, H=4.06/4.16, N=17.15/17.34
- $\text{Cd}(\text{C}_6\text{H}_{16}\text{N}_2)\text{Ni}(\text{CN})_4 \cdot (1,4\text{-C}_6\text{H}_4\text{Cl}_2)$:
C = 35.40/35.69, H=3.92/3.74, N=15.49/15.61

These analytical results are poor for the samples obtainable in powder form because of partial decomposition.

Results and discussion

The compounds studied show no major difference in the IR spectra between the nujol (or hexachlorobutadiene) mulls and KBr pellet. As a representative, the infrared spectrum of the Cd-Ni-1,3-CIBz clathrate is given in Figure 1. The IR spectra of the compounds M-Ni-G (M = Co, Cd or Ni; G = chlorobenzene, 1,2-dichlorobenzene, 1,3-dichlorobenzene and 1,4-dichlorobenzene) are very similar, suggesting that they also have similar structural features

and the degrees of the interactions of the guest molecules, the 1,6-diaminohexane ligand and the $\text{Ni}(\text{CN})_4$ species with their surroundings are almost the same for each clathrate. It will be most convenient to divide the vibrations into three groups arising from the *dahxn* ligands, from the $\text{Ni}(\text{CN})_4$ units and from the guest moieties, respectively. The vibrational wavenumbers of the bands in the spectra of these species are tabulated in Tables 1–4, respectively, together with some relevant spectral data for comparison.

The assignments and the wavenumbers of the vibrational bands of the *dahxn* observed in the spectra of the clathrate compounds under study are given in Table 1, together with the spectral data for *dahxn* in the liquid phase and in solution in CCl_4 on which the assignments are based [8]. The assignments were made in terms of characteristic group frequencies. The vibrational wavenumbers of the $\text{Ni}(\text{dahxn})\text{Ni}(\text{CN})_4 \cdot \text{Bz}$ clathrate [7] are given in the third column in Table 1 for comparison.

Some vibration bands of the ligand molecule show upwards or downwards shifts when compared to the same vibrations in the pure ligand as shown in Table 1. Similar observations were also reported for different clathrates [6, 7, 10, 11]. For example: the band observed in the range $3343\text{--}3284\text{ cm}^{-1}$ (Table 1) assigned to the NH_2 symmetric and asymmetric stretching frequencies are lower than the corresponding values of the free *dahxn* molecule and characteristic of coordinated $-\text{NH}_2$ groups. It shows that the ligand molecules are bound directly to the metal M (M = Co, Ni or Cd). Also, the absence of the splitting of these bands indicates that ligands are bonded in a bidentate manner. As

Table 2. The vibrational wavenumbers (cm^{-1}) of the $\text{Ni}(\text{CN})_4$ group for the M–Ni–G compounds

Assignment ^a	$\text{Na}_2\text{Ni}(\text{CN})_4$ ^a	Ni–Ni–G ^b	Co–Ni–Cl	Cd–Ni–Cl	Ni–Ni–Cl	Co–Ni–2Cl	Cd–Ni–2Cl	Ni–Ni–2Cl
$\nu_8(\text{CN})$, E_4	2132	2160vs	2157vs	2146vs	2158vs	2157vs	2150vs	2163vs
Hot band?	2128	–	–	–	–	2134	–	2136
$\nu_9(\text{NiC})$, E_4	543	584m	553m	538m	580vs	554m	532m	576m
$\nu_9(\text{NiC})$, E_4	–	551w	485vw	503vw	549vw	–	–	549vw
$\pi(\text{NiC})$, A_{24}	448	453vw	451vw	457vw	454vw	451vw	467vw	454w
$\delta(\text{NiC})$, E_4	433	436s	436s	423s	436s	437vs	426vs	439s
Assignment ^a	$\text{Na}_2\text{Ni}(\text{CN})_4$ ^a	Ni–Ni–G ^b	Co–Ni–3Cl	Cd–Ni–3Cl	Ni–Ni–3Cl	Co–Ni–4Cl	Cd–Ni–4Cl	Ni–Ni–4Cl
$\nu_8(\text{CN})$, E_4	2132	2160vs	2163vs	2147	2162vs	2164vs	2144vs	2169vs
Hot band?	2128	–	2136	2107	–	2132	2130	2132w
$\nu_9(\text{NiC})$, E_4	543	584m	567m	534m	577m	568m	547m	584m
$\nu_9(\text{NiC})$, E_4	–	551w	555w	–	552vw	–	–	556vw
$\pi(\text{NiC})$, A_{24}	448	453vw	490m	–	444w	–	490m	464w
$\delta(\text{NiC})$, E_4	433	436s	437vs	424vs	438s	437vs	423vs	441vs

^aTaken from Ref. [9].^bTaken from Ref. [6].

v = very strong, s = strong, m = medium, w = weak.

Table 3. The vibrational wavenumbers (cm^{-1}) of chlorobenzene in the M–Ni–G compounds

Assignment ^a	Chlorobenzene in CCl_4 or CS_2 ^a	Cd–Ni–Cl	Co–Ni–Cl	Ni–Ni–Cl
$\nu(\text{CH})$, A_1	3087w, sh	3088vw	3087vw	3088vw
$\nu(\text{CH})$, B_2	3072s	3075vw	3070vw	3072vw
$\nu(\text{CH})$, A_1	3060w, sh	3061vw	3061vw	3061vw
$\nu(\text{CH})$, A_1	3030w, sh	3030vw	3029vw	3034vw
$\nu(\text{CH})$, B_1	3006w	–	–	–
$\nu(\text{CC})$, B_2	1586s	1587m	1583m	1582m
$\nu(\text{CC})$, A_1	1571w, sh	1560w	1560w	1561w
$\nu(\text{CC})$, A_1	1479vs	1476m	1476m	1476m
$\nu(\text{CC})$, B_2	1447s	1443w	1443w	1443w
$\beta(\text{CH})$, A_1	1123w	1125vw	1134w	1124vw
$\beta(\text{CH})$, B_2	1084s	1082m	1081m	1084s
$\beta(\text{CH})$, B_2	1069vw	–	–	–
$\beta(\text{CH})$, A_1	1023m	1019m	1020w	1018m
$\gamma(\text{CH})$, B_1	1003vw	1000vw	1006vw	1004vw
$\gamma(\text{CH})$, B_1	902vw	904vw	903vw	908vw
$\gamma(\text{CH})$, B_1	741vs	738m	747m	749s
$\delta(\text{ring})$, B_1	702s	703m	703m	703m
$\delta(\text{ring})$, B_2	687s	688m	689m	689m
$\delta(\text{ring})$, B_1	471m	472w	472m	472m

^aTaken from Refs. [12, 13].

v = very strong, s = strong, m = medium, w = weak, sh = shoulder.

seen in Table 1, four bands are assigned to two kinds of CH_2 groups (one for asymmetric CH stretching and one for symmetric CH stretching) in two different environments. Based on the present spectral data it is not possible to describe the conformation of the *dahxn* ligand in our compounds.

In assigning the bands characteristic of the $\text{Ni}(\text{CN})_4^{2-}$ ion in the spectra of the compounds under study, we refer to the work of McCullough et al. who presented vibrational data for the ion $\text{Ni}(\text{CN})_4^{2-}$ in $\text{Na}_2\text{Ni}(\text{CN})_4$ [9]. The vibrational wavenumbers of the $\text{Ni}(\text{CN})_4$ groups in the compounds con-

Table 4. The vibrational wavenumbers (cm^{-1}) of 1,2-dichlorobenzene in the M–Ni–G compounds

Assignment ^a	1,2-dichlorobenzene (gas phase) ^a	Cd–Ni–2Cl	Co–Ni–2Cl	Ni–Ni–2Cl
$\nu(\text{CH})$, A_1	3072	3076vw	3074vw	3071vw
$\nu(\text{CC})$, A_1	1576	1574s	1575vw	1576w
$\nu(\text{CC})$, A_1	1458	1454vs	1455vs	1456vs
$\nu(\text{CC})$, B_2	1438	–	–	–
$\beta(\text{CH})$, B_2	1252	1253w	1254vw	1255vw
X-sens, A_1	1155	1158w	1154vw	1160w
X-sens, B_2	1130	1126m	1125w	1126m
$\beta(\text{CH})$, B_2	1038	1037s	1037m	1037s
$\gamma(\text{CH})$, A_2	975	–	–	–
$\gamma(\text{CH})$, B_1	940	941vw	940vw	939vw
$\gamma(\text{CH})$, A_2	850	859vw	851vw	855vw
$\gamma(\text{CH})$, B_1	748	759s	748m	748s
X-sens, B_2	740	739w	739w	740m
X-sens, A_1	660	660m	661m	661m
X-sens, A_1	480	487vw	489w	481vw
$\phi(\text{CC})$, B_1	435	–	–	–

^aTaken from Ref. [14].

v = very strong, s = strong, m = medium, w = weak, sh = shoulder.

sidered are given in Table 2 together with the vibrational data of $\text{Na}_2\text{Ni}(\text{CN})_4$. The wavenumbers of the spectra of the $\text{Ni}(\text{CN})_4$ group in the $\text{Ni}(\text{hahxn})\text{Ni}(\text{CN})_4 \cdot \text{C}_6\text{H}_6$ compound are also listed for comparison in Table 2. The wavenumbers of the spectra of the $\text{Ni}(\text{CN})_4$ group in the compounds studied appear to be much higher than those for isolated $\text{Ni}(\text{CN})_4$ units (Table 2). Such frequency shifts have been observed for other Hofmann-diam-type clathrates [6, 7, 10, 11] in which both ends of the CN group are coordinated and explained as a mechanical coupling of the internal modes of $\text{Ni}(\text{CN})_4$ with the metal (Co, Ni or Cd)–NC vibrations. The characteristic frequencies of the $\text{Ni}(\text{CN})_4$ group are found to be similar to those of the Hofmann-type clathrates suggest-

Table 5. The vibrational wavenumbers (cm^{-1}) of 1,3-dichlorobenzene in the M–Ni–G compounds

Assignment ^a	1,3-dichlorobenzene (gas phase) ^a	Cd–Ni–3Cl	Co–Ni–3Cl	Ni–Ni–3Cl
$\nu(\text{CH})$, A ₁	3071	3074vw	3072vw	3071w
$\nu(\text{CC})$, A ₁	1580	1572s	1572s	1572s
$\nu(\text{CC})$, B ₂	1464	1460vs	1460s	1463m
$\nu(\text{CC})$, A ₁	1412	1413vw	1417vw	1417w
$\nu(\text{CH})$, B ₂	1258	1254vw	1255vw	1255w
X-sens, B ₂	1161	1161m	1161m	1161m
X-sens, A ₁	1127	1124w	1123w	1122m
$\beta(\text{CH})$, B ₂	1079	1071m	1080s	1084vs
$\gamma(\text{CH})$, B ₁	966	963s	957s	969s
$\gamma(\text{CH})$, A ₂	896	892vw	891vw	892vw
$\gamma(\text{CH})$, A ₂	–	–	–	–
X-sens, B ₂	784	784vs	783s	783vs
$\gamma(\text{CH})$, B ₁	775	–	–	–
X-sens, A ₁	663	661vw	661w	661w
X-sens, A ₁	428	–	–	–

^aTaken from Ref. [13].

v = very strong, s = strong, m = medium, w = weak, sh = shoulder.

ing that coordination around the Ni atom is square and the {M–Ni(CN)₄} layers have been conserved.

The assignments and the wavenumbers of the bands arising from the guest molecules in the IR spectra of M–Ni–G (M = Co, Cd or Ni; G = chlorobenzene, 1,2-, 1,3- and 1,4- dichlorobenzene) compounds are given in Tables 3–6, respectively, together with the wavenumbers of the guest molecules chlorobenzene, vapour phase, in-plane vibrations [12], out-of-plane vibrations [13]; 1,2- and 1,3-dichlorobenzene [14] and 1,4-dichlorobenzene [16], on which the assignments are based. Our band assignments of the guest molecules in the compounds are in agreement with those of the literature [12–14]. As shown in these tables, shifts of the bands of the guest molecules in the compounds are very small. Host–guest interactions in the clathrate structure are not effective, and the IR spectral data of the compounds studied suggest that the guest molecules retain the symmetry of the free molecule. However, some of the CH out-of-plane bands ($\gamma(\text{CH})$) of the guest molecules in the IR spectra of the compounds are observed at higher values than those corresponding to the guest molecules in the solutions due to weak hydrogen bonds between π -electrons located above and below the plane of the benzene ring and the dahxn molecules [18]. Similar frequency shifts were observed in Hofman-type compounds [7, 18].

It should be noted that the number of guest molecules in our clathrates is one. The decrease in the number of guest molecules from 2 in the Hofmann-type [6, 9, 10] and Hofmann-Td-type [17] clathrates to 1 in our compounds is due to the fact that two of the four cavity units are occupied by the dahxn ligand, other cavity units of the host are occupied by the guest molecules [4]. Host–guest interactions

Table 6. The vibrational wavenumbers (cm^{-1}) of 1,4-dichlorobenzene in the M–Ni–G compounds

Assignment ^a	1,4-dichlorobenzene (gas phase) ^a	Co–Ni–4Cl	Ni–Ni–4Cl	Cd–Ni–4Cl
$\nu(\text{CH})$, B _{2u}	3087	3078vw	3074vw	3071vw
$\nu(\text{CH})$, B _{1u}	3078	–	1575vw	1576w
$\nu(\text{CC})$, B _{1u}	1477	1473s	1474s	1471s
$\nu(\text{CC})$, B _{2u}	1394	1392w	1392w	1390w
$\alpha(\text{CCC})$, B _{2u}	1220	1223vw	1226vw	1227w
$\beta(\text{CH})$, B _{2u}	1107	1104w, sh	1107w, sh	1109w, sh
X-sens, B _{1u}	1090	1087m	1087m	1088m
$\beta(\text{CH})$, B _{1u}	1015	1027vw	1029vw	1025vw
$\gamma(\text{CH})$, A _u	951	963m	968m	954m
$\gamma(\text{CH})$, B _{3g}	934	931vw	–	929vw
$\gamma(\text{CH})$, B _{3u}	819 ^b	825m	823m	824m
X-sens, B _{1u}	550	556vw	558vw	558vw
$\phi(\text{CC})$, B _{3u}	485 ^b	487m	488m	489m
$\phi(\text{CC})$, A _u	405	–	–	–

^aTaken from Ref. [16].

^bBand contour of vapour spectrum.

v = very strong, s = strong, m = medium, w = weak, sh = shoulder.

in the clathrate structure are not effective because of the bulkiness of the dahxn ligand molecule.

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