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A Crystallographic and Raman Spectroscopic Study of 1,12-Diaminododecane Dihydrochloride Monohydrate

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Structural features of the crystal state of the bolaform 1,12-diaminododecane-dihydrochloride were ascertained by X-ray crystallography, and laser Raman spectroscopy, and the data are compared to these found for dodecylamine hydrochloride. The study shows that water molecules are bonded to the head groups of the bolaform, and that the methylene chain structure is normal for an all trans structure, similar to monobasic surfactants and crystalline polyethylene. These data throw some light on the nature of the bolaform aggregates formed in aqueous solution.

INTRODUCTION

During the course of studies of cationic surfactants carried out in this laboratory, we became interested in the behavior of alkyl chains with a cationic group at each end. Specifically, we wanted to determine what effects the symmetry of the molecules and the possibility of hydrogen bonding would have on the structural properties of these molecules.

Fundamental to this work was an understanding of the structure, as revealed by X-ray crystallography. Some work had been done on molecules of this general type, including studies of 1,6-hexanediamine dihydrochloride[1] and decane bis(trimethylammonium bromide).[2] In the former case, however, the presence of water of hydration was not considered. We had hoped to be able to compare the structures of a dibasic salt and the corresponding monobasic salt but found that all of the studies of the latter type of compounds to date have neglected the presence of water.[3] We hope that our work will prompt others to re-investigate the mono-basic surfactants.

We were also interested in the effects of hydrogen bonding on the Raman spectrum, the spectra of the *n*-alkanes[4] and dodecylamine hydrochloride[5] having been published.

EXPERIMENTAL SECTION

Preparation and characterization 1,12-Diaminododecane (Eastman Chemical Co.) dissolved in ethanol was reacted with an excess of concentrated aqueous hydrogen fluoride (Fisher Chemical Co.), chloride (Fisher Chemical Co.), bromide (Mallinbrodt Chemical Co.), or iodide (Fisher Chemical Co.). The products were recrystallized from water, as was *n*-dodecylamine hydrochloride (Eastman Chemical Co.). For the deuterated species, the purified salts were recrystallized from D_2O (Bio-Rad Laboratories).

The parent diamine was purified by formation of the dihydrochloride salt dissolution in water (H_2O or D_2O), and neutralization by a concentrated aqueous solution of NaOH or NaOD (formed from sodium in H_2O or D_2O).

The ^{13}C NMR spectrum of a 0.2 M solution of the dihydrochloride, obtained on a Varian XL-100-15 with a Nicolet TT-100 package, contained peaks at 40.02, 29.06, 28.95, 28.63, 27.16, and 26.02 ppm downfield from TMS, measured using internal dioxane and correcting to TMS with $\delta(\text{TMS}) = \delta(\text{dioxane}) + 66.94$, the value obtained from several micellar solutions containing both TMS and dioxane. The peaks are assigned to carbons 1, 6, 5, 4, 2, and 3, respectively, the carbon α to the nitrogen being 1. No impurities were detected.

^{13}C spin-lattice relaxation times of the above solution, measured using the π-ζ-$\pi/2$-D sequence, were 1.24, 1.11, 0.81, 0.84, 0.74, 0.74, and 0.66 sec. for carbons 1–6, respectively. It is noteworthy that the T_1 values follow a smooth progression and that the peak widths were the same for all the carbons. These results are in sharp contrast to the results obtained for the bolaforms, compounds of the formula $\bar{B}\,\bar{r}(CH_3)_3\overset{+}{N}(CH_2)_n\overset{+}{N}(CH_3)_3\bar{B}r, n = 10, 12, 16$.[6] Attempts made to observe the ^{13}C spectrum and measure the T_1's of 1,12-diaminododecane dihydrobromide were unsuccessful due to the very low stability of this compound in water.

A conductivity study of 1,12-diaminododecane dihydrochloride using a Wayne Kerr 641 apparatus showed that no aggregation occurs below 0.4 M. The study was carried out to the solubility limit (about 0.2 M) at room temperature and to 0.4 M at 45°C.

The Raman spectra were obtained on a Spek Ramalog at scan rates and slit widths such that the resolution was ± 3 cm^{-1}. The light source was a Spectra Physics argon ion laser operated at 400 mW. Comparison of the spectra of the dihalides with the spectrum of the parent diamine showed the absence of the latter compound.

X-ray crystallographic data The crystal structure of the dihydrochloride was found to be monoclinic, with cell dimensions $a = 15.284(3)$, $b = 5.123(1)$, $c = 10.729(2)$ Å, $\beta = 90.72(2)°$, $V = 840$ Å^3. The space group was determined to be P2/c with $Z = 2$. Comparison of the calculated and observed (1.115 g cm^{-3}) densities showed that the species was a monohydrate, of formula $C_{12}H_{24}N_2H_6Cl_2 \cdot 1H_2O$.

Diffraction data were collected at 292 K on a Syntex $P\bar{1}$ diffractometer using graphite-monochromated Mo Kα radiation. One quadrant of data was collected by the $\theta - 2\theta$ scan technique with speeds varying 2 to 12° min^{-1}, to the limit $2\theta < 60°$. The intensities of four standard reflections, monitored at regular intervals, showed no significant fluctuation during the collection procedure. A total of 1899 independent reflections having $|F_o| > 3\sigma(F_o)$ was used for solution and refinement of structure.

Solution and refinement of the structure The positions of the chlorine atoms were determined from a three-dimensional Patterson function calculated from intensity data, and the oxygen, nitrogen, carbon, and N- and O-hydrogen atoms were located from a series of difference Fourier maps. Full-matrix least squares refinement was based on minimization of the function $\Sigma w(|F_o| - |F_c|)^2$, with the weights w taken as $\sigma(F_o)^{-2}$, where F_o and F_c are the observed and calculated structure factor amplitudes, respectively. Atomic scattering factors were taken from Cromer and Waber.[7] Anisotropic temperature factors were introduced for all non-hydrogen atoms. The chain hydrogen atoms were assigned fixed positions ($C - H = 0.95$ Å; $B_{iso} = 1 + B_{iso}(C_n)$); the position of the amine and water protons were refined.

The final agreement factors were $R = 0.042$ and $R_w = 0.053$,[8] and the estimated standard deviation of an observation of unit weight was 1.62. The final difference Fourier map was essentially flat, with a maximum of 0.12 e/Å^3. The ratio of data to parameters was 20.2. Atomic coordinates and thermal parameters for all atoms are listed in Table I. Interatomic distances and intermolecular angles are shown in Figure 1. Torsional angles are listed in Table II.

The crystal structure of 1,12-diaminododecane dihydrochloride monohydrate(1)

Chain structure The backbone of *1*, shown in Figure 1, is very much like those of similar compounds. For example, the C—N bond length in *1*, 1.485(2) Å, is the same as the distance measured for methyl ammonium chloride[9] and 1,6-diaminohexane dihydroiodide[10] but considerably shorter than that (1.52 Å) reported in an early study of 1,6-diaminohexanedihydrochloride.[1] The C—C bonds are all in the range expected.

The torsional angles are close to 180° (Table II), indicating an all-*trans* structure, like that of 1,12-bis(trimethylammonium) decane dibromide[2] but curiously differing from that reported for 1,6-diaminohexanedihydroiodide.[10]

TABLE 1

Atomic parameters and their standard deviations[a] for $Cl^- \dot{N}H_3(CH_2)_{12}\dot{N}H_3\ Cl^- \cdot H_2O$

	Coordinates			Anisotropic Thermal Parameters[b]						
Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}	B[c] Å^2
Cl	0.10280(3)	0.13987(8)	0.09070(4)	4.42(2)	3.01(2)	4.17(2)	−0.11(1)	0.98(1)	−0.27(1)	3.74
N	0.1087(1)	0.3666(3)	0.4124(2)	3.14(5)	3.20(6)	4.13(6)	−0.67(5)	0.87(5)	0.69(6)	3.27
C_1	0.2005(1)	0.3398(3)	0.3709(1)	3.12(6)	2.62(5)	3.81(6)	−0.15(4)	0.92(4)	0.35(4)	3.05
C_2	0.2336(1)	0.5918(3)	0.3154(2)	3.23(6)	2.43(5)	4.25(6)	−0.21(4)	1.34(5)	0.27(5)	3.05
C_3	0.3159(1)	0.5524(3)	0.2402(2)	3.23(6)	2.75(6)	4.02(6)	−0.12(5)	1.20(5)	0.41(5)	3.44
C_4	0.3513(1)	0.8052(3)	0.1869(1)	3.14(6)	2.54(6)	3.66(6)	−0.23(4)	1.14(4)	0.17(4)	2.95
C_5	0.4264(1)	0.7660(3)	0.0967(1)	3.33(6)	2.75(6)	3.85(6)	−0.12(5)	1.27(5)	0.32(5)	3.12
C_6	0.4627(1)	1.0197(3)	0.0457(1)	3.51(6)	2.86(6)	4.05(7)	−0.04(5)	1.43(5)	0.43(5)	3.24
O	0	−0.2887(4)	1/4	4.36(8)	3.33(8)	4.33(7)	0	1.04(1)	0	3.90

[a] The number following each datum is the estimated standard deviation in the least significant figure.
[b] B_{ij} is related to the dimensionless β_{ij} employed during refinement by $B_{ij} = 4\beta_{ij}/a_i^* a_j^*$.
[c] Isotropic thermal parameter calculated from $B = 4[v^2 det(\beta_{ij})]^3$.

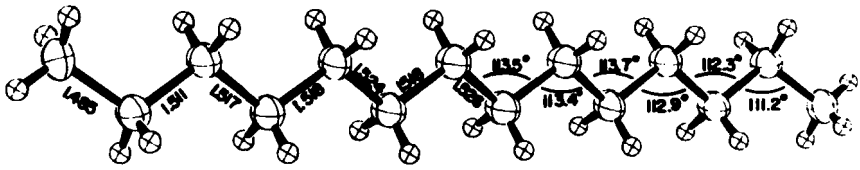

FIGURE 1 The backbone of 1,12-diaminododecane dihydrochloride. Estimated standard deviations on distances are 0.002–0.003 Å; on angles 0.1°. The required center of symmetry is at midpoint of the center C—C bond.

TABLE II

Torsional angles (deg.)

$NC_1C_2, C_1C_2C_3$	163.8(1)
$C_1C_2C_3, C_2C_3C_4$	178.4(1)
$C_2C_3C_4, C_3C_4C_5$	172.1(1)
$C_3C_4C_5, C_4C_5C_6$	178.9(1)
$C_4C_5C_6, C_5C_6C_6,$	179.5(2)

Binnie and Robertson[1] determined that 1,6-diaminohexanedihydrochloride is essentially flat, but that the nitrogens are flared a few degrees out of the plane, the same structure we observed for *1*.

Packing As can be seen from Figures 2 and 3 the molecules are packed with adjacent hydrocarbon chains crossed rather than parallel, in much the same fashion as 1,6-diaminohexanedihydrochloride[1] and 11-aminoundecanoic acid hydrobromide[11] but distinctly different from the parallel structure reported for 1,10-bis(trimethylamino)decane dibromide.[2] Hydrogen bonding to a water molecule situated between them ties together the ends of two neighboring molecules along an axis parallel to the N—C_1 bonds, while hydrogen bonding to chlorides located between chains along the other two directions holds neighboring pairs of chains in place. Each water molecule has symmetric surroundings, donating its protons to one chloride on each side of the plane bisecting the H—O—H angle, and accepting a hydrogen bond from one proton of each of two —NH_3^+ groups in the plane. Each chloride ion accepts hydrogen bonds from one water proton and one proton from each of three —NH_3^+ groups. The results, listed in Table III, are in accord with literature values.[1,3,9–14] It should be noted that the presence of water was considered in only a few cases,[11,13,14] even though the solvent used for recrystallization contained water.

Raman spectra

Common features Figures 4 through 9 are the Raman spectra of 1,12-diaminododecane, its dihydrohalide salts (x = F, Cl) and the hydrochloride salt

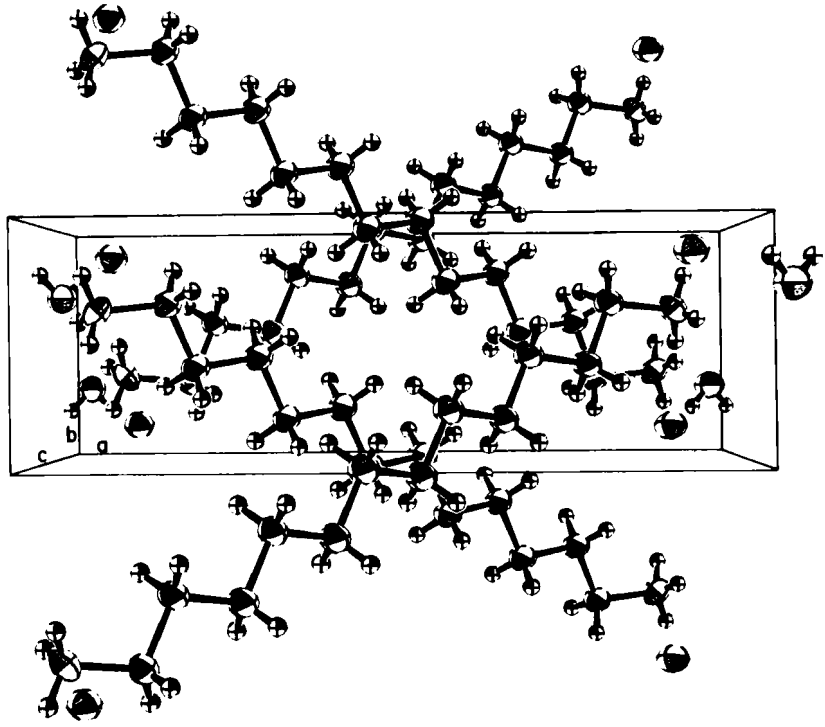

FIGURE 2 Contents of a unit cell of crystalline 1,12-diaminododecane dihydrochloride monohydrate (*I*).

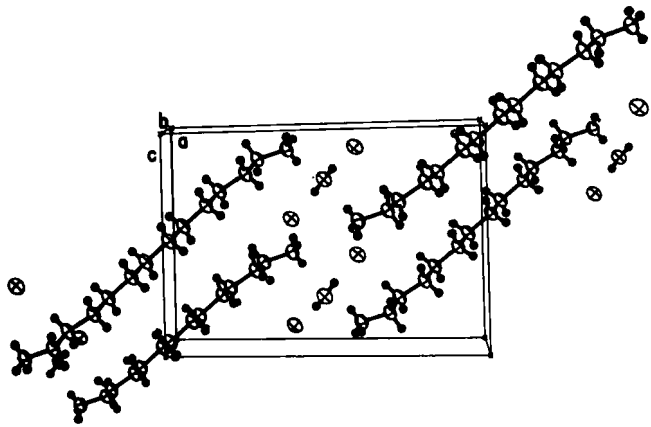

FIGURE 3 Second view of contents of unit-cell.

TABLE III

Hydrogen bond distances in $C_{12}H_{24}N_2H_6Cl_2 \cdot 1H_2O$

Bond (A—H . . . B)	H . . . B Distance (Å)	Number of Bonds
N—H . . . O†	2.32(2)	2
N—H . . . Cl‡	2.19(3)	1
N—H . . . Cl‡	2.33(2)	1
O—H . . . Cl†	2.28(2)	1

† A,H,B not collinear.
‡ A,H,B collinear.

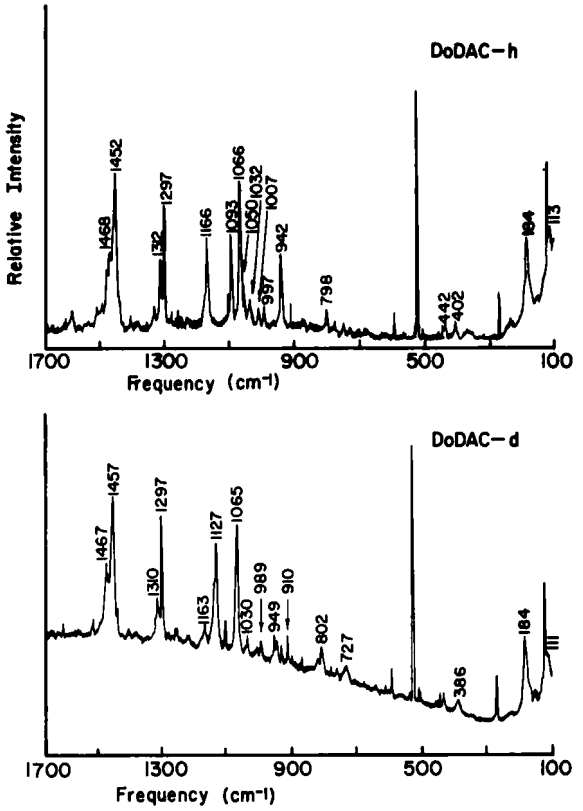

FIGURE 4 The Raman spectra of $C_{12}H_{24}N_2H_6Cl_2 \cdot H_2O$ (top) and $C_{12}H_{24}N_2H_6Cl_2 \cdot D_2O$ (bottom).

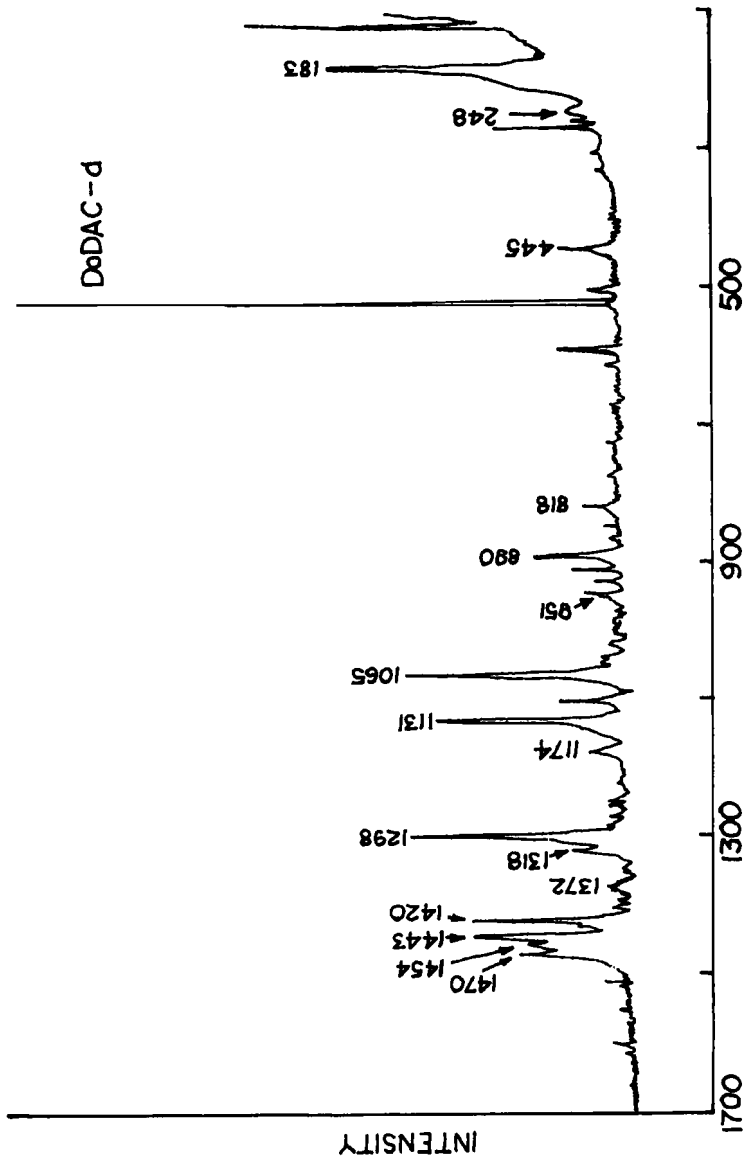

FIGURE 5 The Raman spectra of $C_{12}H_{25}NH_3Cl \cdot nH_2O$ (top) and $C_{12}H_{25}NO_3Cl \cdot nD_2O$ (bottom).

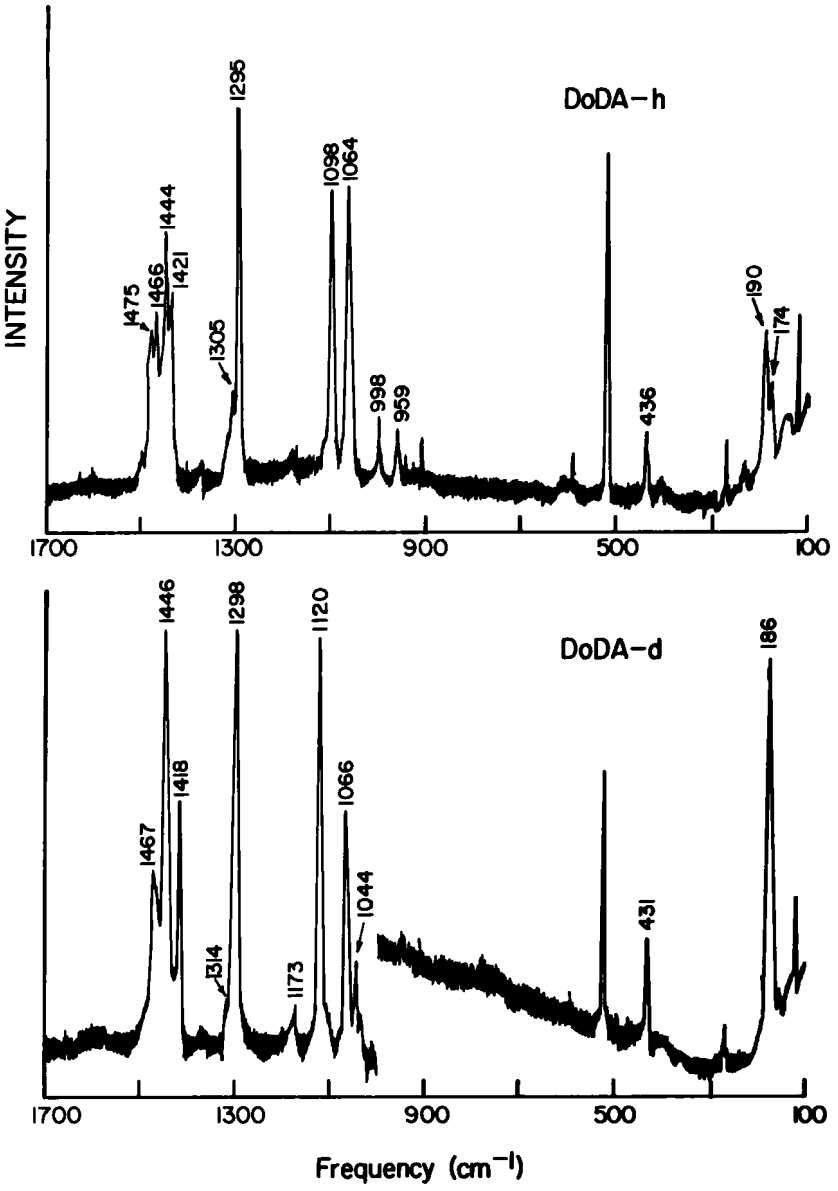

FIGURE 6 The Raman spectra of $C_{12}H_{24}N_2H_4 \cdot nH_2O$ (top) and $C_{12}H_{24}N_2D_4 \cdot nD_2O$ (bottom).

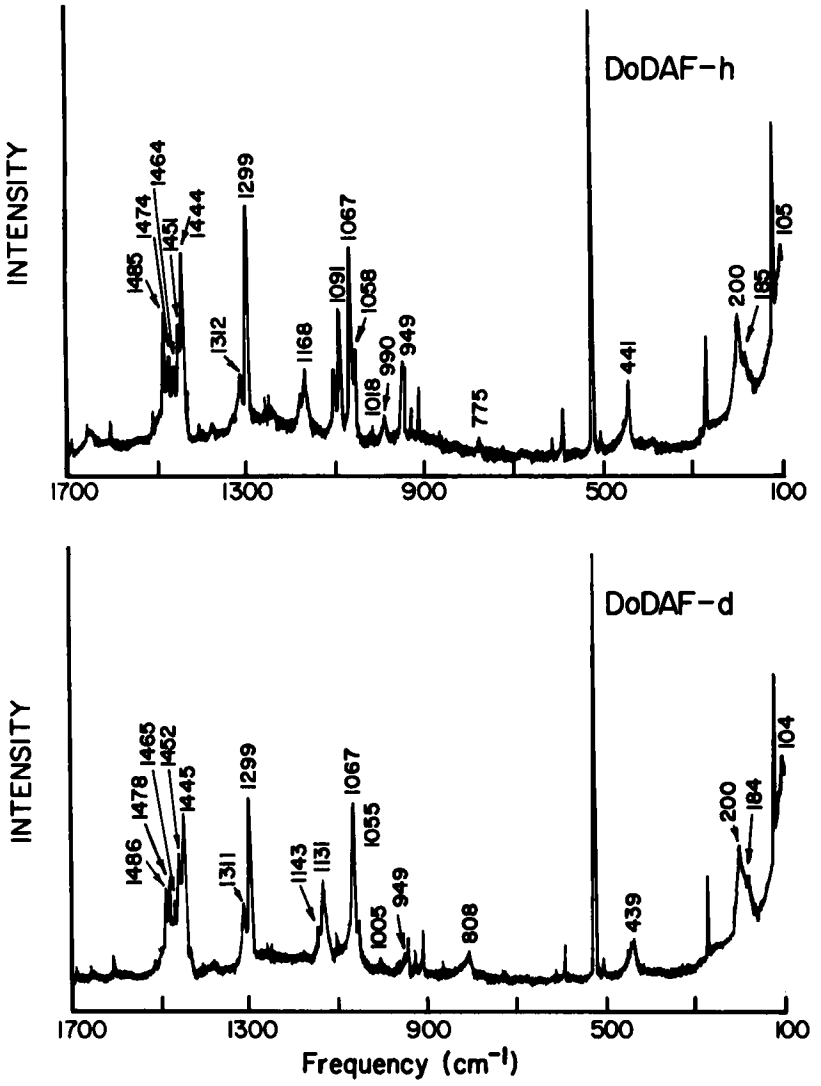

FIGURE 7 The Raman spectra of $C_{12}H_{24}N_2H_6FinH_2O$ (top) and $C_{12}H_{24}N_2D_6F_2 \cdot nD_2O$ (bottom).

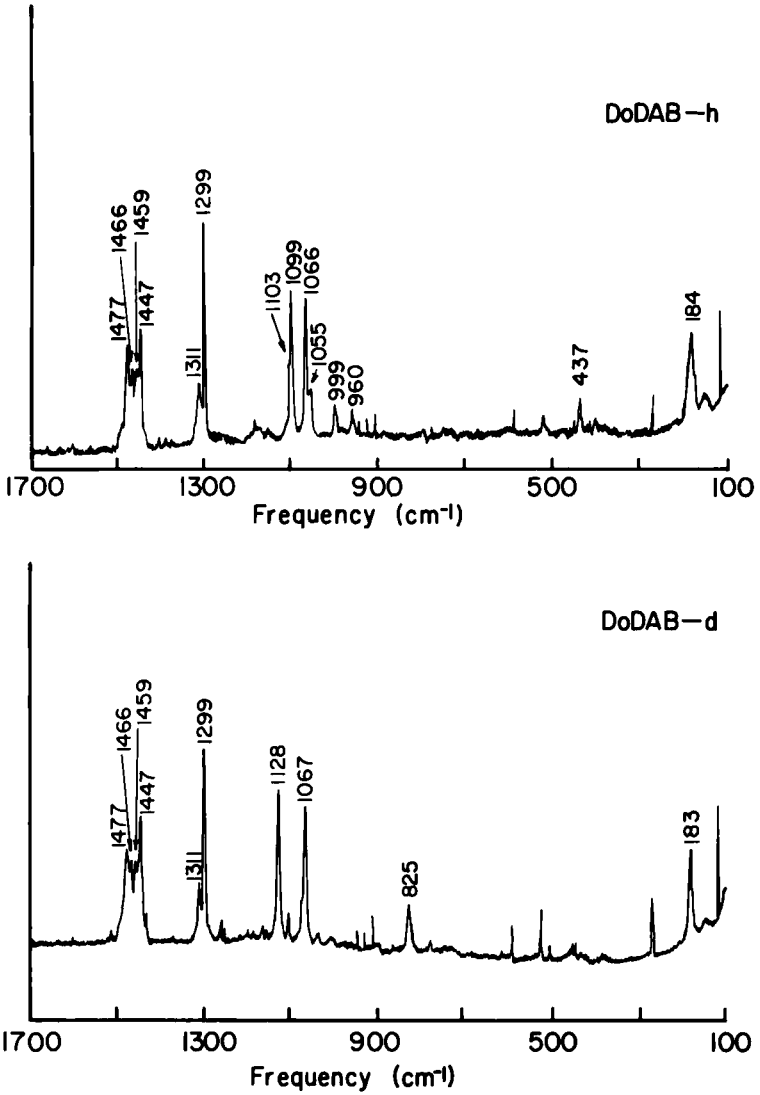

FIGURE 8 The Raman spectra of $C_{12}H_{24}N_2H_6Br_2 \cdot nH_2O$ (top) and $C_{12}H_{24}N_2D_6Br_2 \cdot nD_2O$ (bottom).

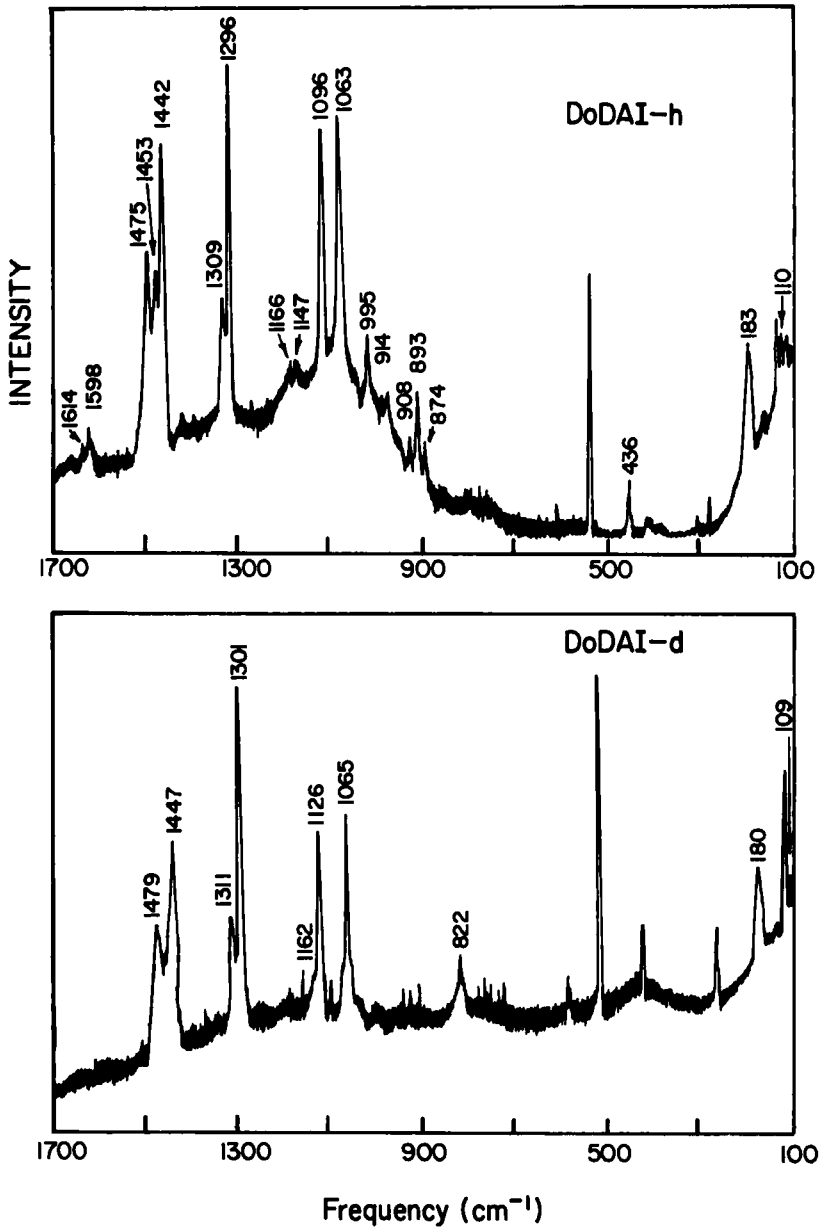

FIGURE 9 The Raman spectra of $C_{12}H_{24}N_2H_6I_2 \cdot nH_2O$ (top) and $C_{12}H_{24}N_2D_6I_2 \cdot nD_2O$ (bottom).

of 1-dodecylamine, all in both the protiated and N-deuterated forms. Several of the bands are common to all the spectra and have also been observed in the spectra of some monobasic cationic surfactants[5] as well as *n*-alkanes[4] and crystalline polyethylene.[15] These bands are listed in Table IV along with the assignments. One other band is common to the spectra of alkyl ammonium compounds but does not appear in the spectra of quaternary ammonium compounds. Thus, the band at $1315 \pm 5\ cm^{-1}$ is believed to arise from an as-yet-undetermined motion of the head groups, perhaps an N—H band.

A band at $1420\ cm^{-1}$ in the spectrum of crystalline polyethylene was assigned to $(b_{2g} + b_{3g})$, parallel to the chain, a CH_2 wag. A peak at 1420 ± 5 in 1,12-diaminododecane and dodecylamine hydrochloride is believed to be due to this vibration. In the dihydrohalide salts of 1,12-diaminododecane, however, a band of similar intensity appears in the region $1447 \pm 5\ cm^{-1}$.

The band which appears in the region $1090–1100\ cm^{-1}$ in the spectra of the protiated diamine and all its salts is shifted to $1120–30\ cm^{-1}$ in the deuterated compounds, the same position as a medium to strong band in the spectrum of dodecylamine hydrochloride. A band appearing at $1125 \pm 5\ cm^{-1}$ in the spectra monobasic surfactants,[5] *n*-alkanes,[4] and crystalline polyethylene[15] has been assigned to a skeletal deformation on the basis of a normal coordinate analysis.[15] In this analysis, however, head group and interchain effects between head groups were negligible, a situation which one does not obtain in the case of the diamino compounds. The shift upon deuteration shows that motion of the ammonium head group contributes to this band, and the observation that the position of this band is nearly independent of counterion (in fact, being insensitive to the presence of a counterion) suggests that hydrogen

TABLE IV

Raman bands common to the spectra of the solids DoDa, DoDax, and Dac†

Frequency (cm^{-1})	Assignment‡
1440–60	CH_2 deformation
1298	CH_2 twist
1168–75	CH_2 rock
1066	Skeletal deformation
184	Accordian made, characteristic of a 12-carbon chain

† *DoDa* = 1,12-diaminododecane, DoDax = 1,12-diaminododecane dihydrochloride (x = F, Cl, Br, I), Dac = 1-dodecylamine hydrochloride.

‡ From J. R. Nielsen and R. F. Holland, *J. Mol. Spec.*, **6**, 394 (1961).

bonding to a water molecule between head groups of neighboring chains is important. Determination of the exact contribution must await the results of a theoretical analysis.

The direction of the shift, from 1095 ± 5 to 1125 ± 5 cm^{-1}, is opposite to that expected solely on the basis of a change in reduced mass. However, the shift can be accounted for if one accepts the view that water acts as a weak base in the case of the dihydrochloride salt, in which the N—H . . . O grouping is not collinear. It is known that deuterium bonds to weak bases are often stronger than are hydrogen bonds.[16] An alternative view is that the increased length of the N—H bond upon deuteration reduces greatly the strength of the hydrogen bond to water, so that interchain forces are minimized.The compound would then vibrate at the same frequency as the monobasic surfactants and crystalline polyethylene.

Features not held in common The striking feature of the spectrum of the dihydrochloride salt of 1,12-diaminododecane (Figure 4) is the shift upon deuteration of the 1163 cm^{-1} band to 1127 cm^{-1}. Since such a shift was observed also in dodecylammonium chloride (Figure 5) but not in any of the other compounds studied, we believe the 1163 cm^{-1} vibration involves the chloride ion.

The band at 997 cm^{-1} in the spectrum of the dihydrochloride is believed, on the basis of the assignment in methyl ammonium chloride,[17] to be due to the C—N stretch. In the deuterated compound, a peak is found at 949 cm^{-1}, compared with 947 cm^{-1} for $CH_3\overset{+}{N}D_3\overset{-}{Cl}$. Most of the other salts show these bands also, though in some they are very weak, so that picking them out of the noise is not possible.

In the parent diamine, the C—N stretch is believed to be part of the 1064 cm^{-1} band, shifted to 1044 cm^{-1} in the deuterated compound. Bands of this type are known to result in Raman frequencies in the range 1060–1090 cm^{-1}.[18]

All the salts have weak bands in the range 900–1060 cm^{-1}, bands which shift upon deuteration. We believe motions involving the hydrogen bonds are responsible for these bands.

One feature of the spectrum of the dihydrofluoride (Figure 7) that is particularly noteworthy is the band at 200 cm^{-1}. Joesten and Schaad[19] pointed out that bands in this region can arise from motion of the B atom in an A—H . . . B grouping. This is a particularly appealing explanation in view of the small site of the fluoride ion and the likelihood of a small amount of motion about its equilibrium position in the lattice.

The Raman spectra confirm the X-ray crystallographic data, namely:

(a) Water molecules are bonded to the head groups of the bolaforms, and

(b) The methylene chain structure is normal for an all trans structure, and show similarities to monobasic surfactants, alkanes and crystalline polyethylene.

A layered type structure is obtained with fully extended hydrocarbon chains in an all trans structure.

It is tempting to carry over this structure to the aggregated bolaform in aqueous solution. One can envisage an extended structure of bolaform molecules with extended chains all packed in an overlaid fashion as shown in Figure 2 for the crystal structure. The exterior of this structure is covered with polar head groups which are in contact with the aqueous phase. The interior of the structure is hydrophobic containing the methylene groups. The ends of such a structure present hydrocarbon chains to the aqueous phases. To minimize this effect it might be possible to fold bolaform solvents to enclose the ends. U type structures, as suggested for bolaforms on water surfaces,[20] could be placed such that the hydrocarbon chains of the U shaped bolaform locate at the ends of the extended bolaform structure, with the polar heads toward the aqueous phase. Such structures, if they exist, would be roughly cylindrical.

Supplementary material available A listing of observed and calculated structure amplitudes (X 10) (9 pages). Ordering information is given on any current masthead page.

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Obsd and calcd. structure amplitudes (× 10) $C_{12}H_{24}N_2H_6Cl_2 \cdot H_2O$

H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL
	K = 0**			17	2	32	32	−7	6	54	57	12	8	49	51
2	0	92	84	18	2	47	46	−6	6	200	211	13	8	42	38
3	0	212	212	19	2	38	37	−5	6	266	279	14	8	32	26
4	0	407	408	21	2	17	10	−4	6	225	230	15	8	29	32
5	0	390	405	−20	4	54	45	−3	6	36	37	17	8	28	29
6	0	296	310	−19	4	33	33	−2	6	340	350	18	8	32	32
7	0	159	157	−18	4	36	39	−1	6	255	255	−16	10	43	36
8	0	112	114	−17	4	31	30	0	6	431	443	−15	10	148	142
9	0	626	647	−15	4	78	74	1	6	307	314	−14	10	34	31
10	0	323	327	−14	4	109	110	2	6	119	117	−13	10	108	102
11	0	169	170	−13	4	259	272	3	6	97	107	−12	10	57	58
12	0	19	19	−12	4	47	46	4	6	274	276	−10	10	74	72
13	0	86	87	−11	4	12	15	5	6	302	302	−9	10	131	131
14	0	126	129	−10	4	127	127	6	6	96	95	−8	10	114	116
15	0	113	115	−9	4	194	209	7	6	251	239	−6	10	176	179
16	0	45	39	−8	4	172	185	8	6	19	27	−5	10	42	41
17	0	48	53	−7	4	26	30	9	6	203	201	−4	10	170	172
18	0	22	14	−6	4	233	236	10	6	157	160	−3	10	118	118
19	0	86	88	−5	4	514	515	11	6	98	97	−2	10	37	40
20	0	57	52	−4	4	26	32	12	6	23	24	−1	10	38	38
21	0	23	23	−3	4	627	625	13	6	74	73	0	10	111	111
−20	2	19	26	−2	4	244	233	14	6	116	117	1	10	113	111
−18	2	26	19	−1	4	71	78	15	6	56	55	2	10	53	53
−17	2	53	52	0	4	320	328	16	6	32	39	4	10	120	117
−16	2	142	143	1	4	387	400	18	6	37	35	5	10	101	95
−14	2	58	61	2	4	176	182	19	6	31	32	6	10	88	90
−13	2	14	6	3	4	216	227	−18	8	61	53	7	10	40	39
−12	2	82	84	4	4	445	474	−17	8	77	75	9	10	36	29
−11	2	102	107	5	4	87	77	−16	8	130	126	10	10	21	16
−10	2	67	70	6	4	401	423	−15	8	48	50	11	10	29	30
−8	2	80	87	7	4	226	244	−13	8	61	60	12	10	54	57
−7	2	39	30	8	4	84	91	−12	8	81	83	13	10	48	50
−6	2	896	891	9	4	48	53	−11	8	50	46	14	10	20	19

Obsd and calcd. structure amplitudes (× 10) $C_{12}H_{24}N_2H_6Cl_2 \cdot H_2O$

H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL
−5	2	374	376	10	4	136	146	−10	8	32	36	15	10	30	28
−4	2	81	86	11	4	109	113	−9	8	87	92	16	10	20	12
−3	2	122	120	13	4	74	75	−8	8	21	28	−13	12	28	32
−2	2	218	207	14	4	22	21	−7	8	211	221	−12	12	131	129
−1	2	116	107	15	4	74	74	−6	8	150	154	−10	12	73	71
0	2	64	62	16	4	58	55	−5	8	78	75	−9	12	53	55
1	2	13	9	17	4	35	32	−3	8	98	102	−8	12	26	25
2	2	679	764	20	4	19	15	−2	8	122	120	−6	12	64	62
4	2	517	538	−19	6	170	164	−1	8	91	88	−5	12	116	114
5	2	271	294	−18	6	23	27	0	8	31	30	−4	12	73	77
6	2	42	47	−17	6	32	27	1	8	138	137	−3	12	94	95
7	2	102	104	−16	6	63	61	2	8	112	116	−2	12	40	36
8	2	103	109	−15	6	84	83	3	8	151	154	−1	12	95	97
9	2	68	70	−14	6	75	75	4	8	109	117	0	12	81	84
10	2	75	78	−13	6	28	27	5	8	62	66	1	12	45	48
11	2	87	87	−12	6	39	39	6	8	15	17	3	12	43	42
12	2	180	179	−11	6	79	67	7	8	38	41	4	12	69	69
13	2	161	160	−10	6	314	358	8	8	113	117	5	12	48	47
14	2	116	117	−9	6	211	220	9	8	106	108	6	12	33	35
15	2	54	53	−8	6	99	101	10	8	106	106	7	12	36	34
8	12	46	41	8	1	114	117	−10	3	279	280	7	4	14	14
9	12	54	51	9	1	31	30	−9	3	299	301	8	4	103	107
10	12	32	36	10	1	118	127	−8	3	254	256	10	4	59	57
−7	14	35	36	11	1	111	116	−7	3	286	290	11	4	98	98
−2	14	31	33	12	1	71	77	−6	3	104	112	12	4	117	117
−1	14	49	52	13	1	28	28	−5	3	207	220	13	4	67	63
0	14	47	43	14	1	26	30	−4	3	198	200	14	4	26	28
1	14	31	24	15	1	39	36	−3	3	141	135	15	4	72	74
2	14	40	36	16	1	41	43	−2	3	195	185	17	4	41	41
3	14	25	25	17	1	93	94	−1	3	516	495	18	4	22	17
	K = 1**			−21	2	28	28	0	3	725	715	20	4	26	23
0	0	33	42	−17	2	57	56	1	3	716	720	−19	5	52	49
1	0	120	118	−16	2	59	60	2	3	96	97	−17	5	34	35

2	0	610	576	−15	2	21	32	3	3	21	18	−16	5	62	66
3	0	211	190	−14	2	48	54	4	3	251	256	−15	5	64	60
4	0	245	231	−13	2	163	168	5	3	390	403	−14	5	21	20
5	0	223	215	−12	2	124	128	6	3	49	50	−13	5	54	48
6	0	108	109	−11	2	112	114	8	3	146	149	−12	5	84	85
7	0	17	12	−10	2	86	88	9	3	222	226	−11	5	68	69
8	0	66	65	−9	2	94	101	10	3	201	208	−10	5	122	129
9	0	16	17	−8	2	385	386	11	3	128	136	−9	5	74	73
10	0	16	17	−7	2	208	215	12	3	37	40	−8	5	76	80
11	0	174	180	−6	2	135	136	13	3	70	72	−7	5	151	157
12	0	26	26	−5	2	65	63	14	3	145	144	−6	5	196	200
13	0	48	51	−4	2	177	170	16	3	18	14	−5	5	339	352
14	0	39	37	−3	2	317	307	17	3	27	27	−4	5	128	134
16	0	83	83	−2	2	413	394	18	3	34	31	−3	5	74	79
17	0	17	14	−1	2	444	416	20	3	53	52	−2	5	100	106
20	0	20	20	0	2	511	472	−20	4	42	46	−1	5	32	35
−20	1	31	25	1	2	638	627	−19	4	47	45	0	5	11	12
−19	1	35	32	2	2	422	421	−18	4	56	54	1	5	58	60
−18	1	24	24	3	2	335	334	−17	4	35	37	2	5	111	116
−14	1	175	177	4	2	95	95	−14	4	52	50	3	5	189	196
−13	1	157	159	5	2	198	196	−13	4	67	66	4	5	349	358
−12	1	98	101	6	2	139	140	−12	4	29	30	5	5	41	49
−10	1	217	224	7	2	222	225	−11	4	164	170	6	5	63	69
−9	1	19	19	8	2	224	229	−10	4	113	114	7	5	17	23
−8	1	87	92	9	2	139	145	−10	4	113	114	8	5	177	182
−7	1	56	58	11	2	121	123	−9	4	148	148	11	5	27	28
−6	1	76	75	12	2	101	107	−8	4	143	144	12	5	65	60
−5	1	341	327	14	2	80	81	−7	4	103	107	13	5	66	65
−4	1	527	493	16	2	51	53	−6	4	320	320	14	5	49	51
−3	1	576	534	17	2	50	50	−5	4	207	207	15	5	45	46
−2	1	355	325	19	2	35	33	−4	4	186	191	17	5	54	54
−1	1	501	478	−20	3	55	53	−3	4	204	206	−18	6	46	41
0	1	155	145	−19	3	69	72	−2	4	231	222	−17	6	32	35
1	1	311	276	−18	3	48	49	0	4	133	131	−16	6	43	41
2	1	281	266	−16	3	55	53	1	4	174	172	−15	6	66	64
3	1	432	425	−15	3	77	77	2	4	192	195	−14	6	44	46
4	1	16	17	−14	3	61	61	3	4	450	457	−12	6	36	45

Obsd and calcd. structure amplitudes (× 10) $C_{12}H_{24}N_2H_6Cl_2 \cdot H_2O$

H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL
5	1	320	321	−13	3	15	18	4	4	33	36	−11	6	95	96
6	1	356	368	−12	3	90	95	5	4	156	163	−10	6	67	70
7	1	257	262	−11	3	171	172	6	4	259	264	−9	6	77	81
−8	6	110	114	15	7	32	31	6	9	31	29	−1	12	87	87
−7	6	106	108	16	7	26	23	7	9	56	54	2	12	23	18
−6	6	163	165	−18	8	57	56	8	9	83	79	3	12	27	26
−5	6	126	128	−17	8	57	58	9	9	38	34	4	12	50	49
−4	6	34	37	−16	8	56	55	10	9	86	89	8	12	25	26
−3	6	132	135	−15	8	48	46	11	9	24	24	−10	13	41	38
−2	6	131	135	−13	8	48	48	14	9	49	48	−8	13	59	58
−1	6	129	127	−12	8	85	84	15	9	22	18	−6	13	36	41
0	6	88	88	−11	8	95	93	−16	10	57	54	−5	13	60	60
1	6	63	62	−10	8	57	57	−14	10	33	36	−4	13	63	59
2	6	53	52	−9	8	22	28	−13	10	81	79	−3	13	41	44
3	6	129	130	−8	8	133	137	−12	10	22	25	−1	13	32	30
4	6	93	98	−7	8	171	174	−10	10	30	29	1	13	56	58
5	6	23	13	−6	8	22	27	−9	10	38	38	2	13	18	15
6	6	142	139	−5	8	74	76	−8	10	42	43	4	13	33	29
7	6	16	15	−4	8	63	64	−7	10	62	65	5	13	64	65
8	6	38	40	−3	8	156	156	−6	10	30	31	6	13	23	17
9	6	125	129	−2	8	183	188	−5	10	40	42	9	13	19	4
12	6	24	25	−1	8	35	36	−4	10	137	136	−7	14	51	50
13	6	17	16	0	8	70	71	−2	10	41	41	−6	14	19	19
15	6	16	22	1	8	79	81	0	10	51	51	−4	14	33	33
16	6	22	28	2	8	131	128	1	10	105	104	−3	14	51	51
18	6	29	30	3	8	100	103	2	10	27	25	−2	14	30	25
−18	7	41	38	4	8	74	73	3	10	57	57	2	14	49	49
−17	7	50	48	6	8	107	103	5	10	51	50	3	14	22	20
−16	7	81	78	7	8	122	119	6	10	34	35	5	14	21	8
−15	7	38	38	9	8	47	47	7	10	45	44	7	14	28	30
−14	7	82	77	11	8	18	15	8	10	27	31	−2	15	32	24
−13	7	96	96	12	8	69	70	9	10	22	18	−1	15	28	31
−12	7	69	71	13	8	27	30	10	10	59	57	0	15	35	34

												K = 2**			
−10	7	75	71	15	8	26	23	12	10	18	20	0	0	242	226
−9	7	143	144	16	8	29	27	15	10	22	15	1	0	208	191
−8	7	180	185	−17	9	37	31	−14	11	27	30	2	0	116	104
−7	7	120	126	−15	9	31	31	−11	11	66	63	3	0	30	26
−6	7	61	67	−14	9	93	91	−10	11	34	30	4	0	133	111
−5	7	172	176	−13	9	40	36	−9	11	41	42	5	0	234	218
−4	7	185	187	−12	9	32	31	−8	11	51	54	6	0	60	65
−3	7	107	106	−11	9	71	70	−7	11	60	58	7	0	22	22
−2	7	210	211	−10	9	103	102	−6	11	47	48	8	0	64	64
−1	7	64	67	−9	9	91	95	−2	11	106	106	9	0	73	74
0	7	139	137	−8	9	48	49	1	11	54	56	10	0	73	70
1	7	164	166	−6	9	70	68	2	11	91	89	11	0	70	70
2	7	59	68	−5	9	210	210	4	11	38	41	12	0	64	65
3	7	55	51	−4	9	18	13	5	11	20	21	13	0	130	134
4	7	136	138	−3	9	13	11	7	11	23	28	14	0	95	98
5	7	124	129	−2	9	113	115	8	11	34	34	15	0	25	23
6	7	73	74	−1	9	156	158	9	11	23	23	20	0	20	19
7	7	151	149	0	9	74	78	11	11	36	32	−20	1	33	39
9	7	27	24	1	9	148	148	13	11	19	21	−19	1	35	30
10	7	17	19	2	9	33	34	−10	12	70	68	−18	1	78	78
11	7	93	88	3	9	42	45	−8	12	19	21	−17	1	74	72
13	7	24	23	4	9	120	119	−7	12	52	54	−16	1	35	36
14	7	30	30	5	9	97	98	−6	12	49	45	11	5	69	73
−15	1	18	14	2	2	114	109	−13	4	50	47	12	5	119	117
−14	1	82	79	3	2	148	144	−12	4	46	45	13	5	71	72
−13	1	21	18	4	2	136	131	−11	4	43	44	14	5	25	24
−12	1	104	109	5	2	84	84	−10	4	44	47	15	5	34	30
−11	1	215	220	6	2	136	120	−9	4	224	222	16	5	55	55
−10	1	58	58	7	2	100	92	−8	4	208	209	17	5	38	34
−9	1	244	244	8	2	68	67	−7	4	45	45	19	5	27	23
−8	1	320	323	10	2	13	10	−6	4	24	26	−19	6	18	10
−7	1	288	283	11	2	26	29	−5	4	74	75	−17	6	17	12
−6	1	123	121	12	2	26	26	−4	4	98	96	−16	6	17	21
−5	1	123	123	13	2	17	16	−3	4	122	116	−15	6	59	54
−4	1	10	21	14	2	39	36	−2	4	139	131	−14	6	41	39
−3	1	528	501	15	2	79	81	−1	4	119	109	−11	6	26	27
−2	1	491	455	16	2	74	76	0	4	212	200				

Obsd and calcd. structure amplitudes (× 10) $C_{12}H_{24}N_2H_6Cl_2 \cdot H_2O$

H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL
−1	1	165	155	17	2	34	32	1	4	190	174	−10	6	36	37
0	1	206	192	−20	3	37	38	2	4	27	25	−9	6	38	39
1	1	482	448	−18	3	29	28	3	4	40	37	−8	6	52	52
2	1	547	508	−16	3	43	38	4	4	67	65	−7	6	142	144
3	1	394	379	−15	3	94	93	5	4	75	76	−6	6	227	229
4	1	187	173	−14	3	97	96	6	4	89	90	−5	6	130	136
5	1	192	172	−13	3	38	39	7	4	55	59	−4	6	20	21
6	1	336	340	−12	3	61	62	9	4	79	80	−3	6	21	16
7	1	210	206	−11	3	211	213	10	4	49	49	−2	6	56	55
8	1	170	164	−9	3	45	42	11	4	13	13	−1	6	57	58
9	1	20	16	−8	3	113	107	18	4	47	50	0	6	73	75
10	1	99	97	−7	3	52	54	19	4	27	29	1	6	107	106
11	1	171	170	−6	3	223	220	−18	5	38	35	2	6	125	121
12	1	205	206	−5	3	305	301	−17	5	106	104	3	6	113	111
13	1	167	168	−4	3	242	240	−16	5	86	86	4	6	62	59
14	1	67	71	−2	3	382	360	−14	5	19	15	5	6	24	26
15	1	97	101	−1	3	18	22	−13	5	81	80	7	6	23	21
16	1	39	39	0	3	77	88	−12	5	158	153	8	6	30	29
17	1	54	53	1	3	168	160	−11	5	189	187	9	6	48	46
18	1	25	25	2	3	15	15	−10	5	151	149	10	6	41	40
−20	2	37	36	3	3	142	145	−9	5	23	16	12	6	41	42
−19	2	20	21	4	3	219	226	−8	5	366	369	13	6	20	21
−17	2	27	27	5	3	204	206	−7	5	254	261	−18	7	34	35
−16	2	29	28	6	3	113	111	−6	5	50	50	−17	7	37	39
−14	2	14	12	7	3	83	74	−5	5	68	63	−16	7	27	30
−12	2	93	94	8	3	105	104	−4	5	91	92	−14	7	77	74
−11	2	198	204	9	3	132	127	−3	5	258	258	−13	7	114	110
−10	2	103	107	10	3	71	74	−2	5	365	365	−12	7	32	31
−9	2	29	30	11	3	31	33	−1	5	370	372	−10	7	50	49
−8	2	71	69	12	3	15	21	0	5	181	181	−9	7	94	92
−7	2	101	98	13	3	36	36	1	5	268	271	−8	7	118	116
−6	2	122	117	14	3	29	24	2	5	345	338	−7	7	144	144
−5	2	121	119	15	3	42	37	3	5	171	172	−6	7	183	185
−4	2	75	71	16	3	85	86	4	5	143	151	−5	7	139	141

−3	2	217	200	18	3	44	44	6	5	143	139	−4	7	258	262
−2	2	338	299	−18	4	38	38	7	5	198	199	−3	7	98	94
−1	2	33	27	−17	4	42	45	8	5	176	178	−2	7	72	72
0	2	21	21	−16	4	17	10	9	5	77	79	−1	7	23	21
1	2	76	71	−14	4	34	33	10	5	21	22	0	7	99	97
1	7	138	135	3	9	108	104	2	12	60	63	1	1	201	200
2	7	190	190	4	9	58	56	3	12	18	18	2	1	215	205
3	7	206	209	5	9	19	22	6	12	18	10	3	1	108	103
5	7	154	156	6	9	68	68	11	12	21	18	4	1	163	165
6	7	122	116	8	9	59	59	−9	13	31	35	5	1	56	51
7	7	103	105	9	9	50	49	−8	13	43	41	6	1	73	81
8	7	38	41	10	9	32	30	−7	13	19	16	7	1	24	14
9	7	24	23	11	9	16	3	−4	13	48	44	9	1	34	33
10	7	70	64	12	9	18	21	−3	13	39	40	10	1	83	85
11	7	86	88	13	9	28	18	−2	13	24	25	11	1	100	100
12	7	65	63	14	9	24	18	0	13	58	52	12	1	41	43
13	7	34	31	−13	10	19	23	1	13	66	68	13	1	63	60
15	7	35	39	−12	10	19	19	5	13	19	22	14	1	37	37
16	7	40	38	−10	10	36	35	6	13	26	26	16	1	51	52
17	7	26	28	−9	10	45	43	7	13	30	30	19	1	21	18
−16	8	20	12	−5	10	43	41	9	13	19	18	−17	2	48	47
−15	8	18	6	−4	10	48	50	−6	14	18	12	−16	2	52	56
−13	8	36	34	−3	10	18	21	3	14	20	24	−15	2	37	39
−12	8	55	54	−2	10	31	32	4	14	29	23	−13	2	43	37
−11	8	24	25	−1	10	113	117	5	14	20	21	−12	2	205	201
−9	8	27	27	0	10	67	70		**K = 3****			−11	2	134	131
−8	8	42	41	4	10	31	29	0	0	127	123	−10	2	173	180
−7	8	38	37	5	10	34	35	1	0	84	83	−9	2	94	96
−4	8	157	159	8	10	46	49	3	0	148	141	−8	2	133	139
−3	8	149	150	9	10	16	13	4	0	320	313	−7	2	173	169
−2	8	54	57	−13	11	54	54	5	0	270	267	−6	2	159	152
0	8	16	17	−12	11	78	73	6	0	121	107	−5	2	69	61
1	8	45	45	−11	11	69	64	7	0	42	45	−4	2	87	95
2	8	29	31	−9	11	29	25	8	0	40	39	−3	2	166	153
4	8	24	27	−8	11	59	61	9	0	74	72	−2	2	326	325
5	8	98	96	−7	11	113	110	10	0	49	49	−1	2	531	514
6	8	42	41	−6	11	72	72	11	0	26	24	0	2	23	18

Obsd and calcd. structure amplitudes (× 10) $C_{12}H_{24}N_2H_6Cl_2 \cdot H_2O$

H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL
7	8	14	16	−4	11	76	76	12	0	82	84	1	2	108	101
12	8	18	12	−3	11	137	134	13	0	55	60	2	2	228	221
13	8	25	14	−2	11	109	108	14	0	30	27	3	2	244	239
15	8	22	19	0	11	21	24	15	0	109	104	4	2	124	119
−16	9	50	47	2	11	108	105	16	0	36	32	5	2	83	80
−15	9	47	47	3	11	89	86	18	0	20	8	6	2	23	31
−14	9	23	24	4	11	47	49	−19	1	22	22	7	2	210	207
−12	9	20	22	5	11	22	22	−17	1	18	16	8	2	222	229
−11	9	69	67	6	11	83	83	−13	1	103	104	9	2	126	118
−10	9	104	104	7	11	67	69	−12	1	27	25	10	2	15	20
−9	9	21	24	8	11	20	15	−11	1	127	131	11	2	68	68
−8	9	42	44	9	11	20	25	−9	1	63	65	12	2	104	105
−7	9	108	109	11	11	29	27	−8	1	112	113	13	2	63	61
−6	9	125	122	12	11	30	31	−7	1	104	102	15	2	18	19
−5	9	65	61	13	11	24	24	−6	1	30	28	16	2	54	49
−3	9	69	71	−10	12	17	14	−5	1	83	89	17	2	19	17
−2	9	47	45	−7	12	36	32	−4	1	87	76	18	2	51	48
−1	9	178	178	−6	12	17	14	−3	1	151	146	19	2	23	17
0	9	78	76	−1	12	28	34	−2	1	236	214	−19	3	46	47
1	9	16	14	0	12	27	28	−1	1	31	32	−18	3	23	21
2	9	73	71	1	12	36	36	0	1	114	112	−16	3	47	47
−15	3	51	56	10	4	57	56	3	6	111	111	2	8	125	122
−14	3	54	55	11	4	123	120	5	6	156	161	3	8	132	131
−13	3	36	38	12	4	100	97	6	6	62	63	4	8	70	68
−11	3	41	38	13	4	60	58	8	6	33	33	5	8	33	32
−10	3	134	131	15	4	53	54	9	6	74	74	6	8	117	116
−9	3	165	167	16	4	56	56	10	6	78	77	7	8	38	36
−8	3	83	88	18	4	22	18	11	6	45	41	8	8	87	86
−7	3	57	57	−18	5	30	28	14	6	30	28	9	8	40	39
−6	3	115	115	−17	5	38	35	16	6	23	19	11	8	20	17
−5	3	160	159	−16	5	22	22	17	6	17	12	12	8	44	41
−4	3	163	162	−15	5	17	13	−17	7	21	21	13	8	64	61
−3	3	133	129	−13	5	28	30	−15	7	69	67	15	8	22	20
−1	3	99	90	−12	5	54	56	−13	7	28	27	−15	9	24	24

0	3	313	297	−11	5	54	58	−11	7	18	18	−14	9	31	32
1	3	80	77	−10	5	28	31	−10	7	52	50	−13	9	52	52
2	3	20	5	−9	5	44	42	−9	7	79	81	−12	9	42	44
3	3	80	71	−8	5	87	86	−8	7	93	89	−10	9	58	55
4	3	138	131	−6	5	153	159	−7	7	48	46	−7	9	29	27
5	3	173	173	−5	5	31	35	−6	7	119	122	−6	9	48	52
6	3	177	178	−4	5	47	47	−5	7	14	11	−5	9	83	89
7	3	58	54	−3	5	94	90	−4	7	17	20	−4	9	114	114
9	3	80	84	−2	5	101	95	−3	7	111	112	−3	9	56	61
10	3	41	38	−1	5	55	53	−2	7	20	19	−2	9	43	44
11	3	18	17	0	5	37	44	−1	7	58	58	0	9	78	77
13	3	32	33	1	5	69	65	0	7	109	110	1	9	19	15
14	3	69	72	2	5	36	39	1	7	123	122	3	9	42	43
15	3	65	67	3	5	183	186	2	7	48	47	4	9	85	86
16	3	26	25	4	5	15	21	3	7	33	35	5	9	68	69
19	3	26	27	5	5	21	17	4	7	23	18	7	9	30	29
−18	4	75	71	6	5	44	44	5	7	33	34	8	9	16	20
−17	4	53	52	7	5	43	39	6	7	102	102	9	9	47	45
−14	4	44	44	8	5	41	40	8	7	23	21	10	9	27	21
−13	4	58	60	9	5	99	98	9	7	49	52	12	9	20	16
−12	4	44	45	12	5	37	35	10	7	45	42	13	9	32	27
−10	4	28	29	13	5	17	14	12	7	31	32	−13	10	43	41
−9	4	163	158	18	5	35	35	15	7	20	19	−12	10	38	39
−8	4	128	123	−17	6	49	46	−16	8	51	48	−11	10	58	56
−7	4	261	266	−15	6	76	73	−15	8	24	21	−10	10	18	16
−6	4	29	31	−14	6	72	72	−14	8	64	60	−9	10	25	28
−5	4	68	70	−12	6	51	44	−12	8	79	77	−8	10	72	70
−4	4	137	141	−11	6	86	86	−11	8	124	118	−7	10	47	47
−3	4	150	152	−10	6	83	85	−10	8	49	53	−5	10	21	17
−2	4	104	103	−9	6	20	21	−9	8	16	14	−4	10	75	75
0	4	70	63	−8	6	84	80	−8	8	96	95	−3	10	98	101
1	4	115	112	−7	6	21	24	−7	8	148	147	0	10	95	96
2	4	394	392	−6	6	166	164	−6	8	107	108	1	10	41	39
3	4	142	135	−5	6	74	65	−5	8	55	51	2	10	64	66
4	4	27	29	−4	6	169	170	−4	8	32	31	3	10	43	45
5	4	89	87	−3	6	11	9	−3	8	177	178	5	10	25	27
6	4	167	165	−2	6	89	90	−2	8	97	89	6	10	50	53
7	4	143	140	−1	6	137	134	−1	8	149	147	7	10	57	56

Obsd and calcd. structure amplitudes (× 10) $C_{12}H_{24}N_2H_6Cl_2 \cdot H_2O$

H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL
8	4	28	23	0	6	102	99	0	8	55	54	8	10	18	19
9	4	39	39	2	6	70	71	1	8	47	48	9	10	48	45
10	10	18	14	−18	1	34	33	−15	3	74	68	−14	5	18	12
11	10	35	33	−15	1	22	17	−14	3	21	28	−13	5	31	31
12	10	21	20	−13	1	16	11	−13	3	19	16	−12	5	76	75
13	10	19	6	−10	1	30	27	−11	3	20	22	−11	5	87	82
−12	11	20	10	−8	1	87	90	−10	3	26	26	−8	5	17	20
−10	11	45	40	−7	1	131	133	−9	3	18	11	−7	5	40	40
−7	11	27	31	−6	1	29	31	−8	3	17	17	−6	5	34	36
−4	11	20	23	−4	1	22	20	−7	3	57	62	−4	5	47	57
−2	11	23	15	−3	1	25	27	−6	3	44	32	−3	5	54	50
−1	11	69	74	−1	1	40	44	−5	3	63	62	−2	5	43	38
1	11	38	33	0	1	103	99	−4	3	139	147	−1	5	131	134
3	11	28	22	1	1	158	159	−2	3	21	20	0	5	45	42
8	11	26	30	2	1	249	247	−1	3	40	42	2	5	33	33
10	11	21	18	3	1	29	28	0	3	50	46	3	5	77	75
−10	12	35	32	4	1	16	20	2	3	122	121	4	5	63	64
−9	12	43	43	5	1	21	17	3	3	93	89	5	5	122	123
−8	12	54	52	6	1	37	36	5	3	191	194	7	5	24	26
−7	12	51	52	9	1	70	72	6	3	22	21	8	5	93	96
−6	12	34	36	10	1	59	63	7	3	25	25	9	5	34	30
−5	12	40	39	11	1	98	99	8	3	15	13	10	5	25	27
−2	12	18	16	12	1	29	26	9	3	37	41	12	5	37	37
−1	12	32	32	13	1	24	23	11	3	43	45	13	5	31	28
0	12	57	56	17	1	20	15	12	3	42	45	14	5	30	35
3	12	65	60	−17	2	44	43	14	3	40	43	−16	6	52	46
4	12	34	27	−15	2	30	33	15	3	31	20	−15	6	54	54
5	12	18	16	−13	2	17	20	−14	4	106	103	−14	6	48	47
−7	13	22	14	−12	2	57	60	−13	4	45	49	−13	6	28	28
−6	13	26	24	−11	2	81	82	−12	4	42	46	−12	6	45	40
−4	13	27	25	−10	2	61	63	−11	4	45	44	−11	6	101	99
−2	13	21	14	−9	2	17	23	−10	4	89	87	−10	6	85	87
−1	13	30	34	−7	2	100	106	−9	4	122	126	−9	6	115	114
0	13	23	24	−6	2	84	86	−8	4	123	124	−8	6	21	18

2	13	28	29	−5	2	140	142	−7	4	66	60	−7	6	59	60
4	13	31	25	−4	2	47	42	−6	4	32	40	−6	6	128	129
	K = 4**			−3	2	133	130	−5	4	101	91	−5	6	153	160
0	0	199	200	−2	2	156	158	−4	4	161	155	−4	6	130	132
1	0	278	280	−1	2	124	123	−3	4	72	65	−3	6	35	28
2	0	65	62	0	2	64	62	−2	4	92	95	−2	6	43	44
3	0	129	127	1	2	136	133	−1	4	69	68	−1	6	160	160
4	0	182	181	2	2	119	114	0	4	181	178	0	6	98	92
5	0	191	192	3	2	59	56	1	4	209	208	1	6	119	117
6	0	155	155	4	2	249	246	2	4	131	128	2	6	22	19
7	0	104	107	5	2	25	23	3	4	44	43	3	6	87	83
8	0	112	107	6	2	62	61	4	4	25	20	4	6	167	165
9	0	119	115	7	2	104	100	5	4	137	135	5	6	166	164
10	0	157	162	8	2	94	92	6	4	19	15	7	6	19	25
11	0	44	43	9	2	65	60	7	4	130	131	8	6	98	95
12	0	15	14	10	2	100	102	9	4	67	63	9	6	30	24
13	0	60	60	12	2	22	21	10	4	98	99	10	6	77	76
14	0	80	82	13	2	85	84	11	4	70	70	11	6	41	43
15	0	70	68	14	2	44	42	13	4	18	18	13	6	50	52
16	0	30	29	15	2	17	10	14	4	23	24	14	6	64	63
18	0	39	36	17	2	26	24	16	4	22	28	15	6	26	19
−13	7	17	11	2	9	16	20	−5	1	66	60	2	3	55	54
−12	7	18	6	3	9	43	43	−4	1	120	119	3	3	139	136
−11	7	53	49	5	9	26	29	−3	1	127	129	4	3	149	148
−10	7	48	44	10	9	42	44	−2	1	75	72	5	3	78	77
−9	7	24	27	−11	10	19	12	−1	1	36	34	6	3	75	79
−8	7	80	77	−10	10	57	57	0	1	170	168	8	3	39	35
−7	7	27	25	−9	10	84	85	1	1	96	99	9	3	73	72
−6	7	18	18	−8	10	59	65	2	1	42	46	10	3	84	82
−4	7	26	21	−7	10	26	28	3	1	88	87	11	3	55	54
−3	7	20	20	−5	10	71	72	5	1	60	61	13	3	82	81
−2	7	23	28	−4	10	40	41	6	1	96	100	14	3	27	27
−1	7	35	37	−3	10	47	49	7	1	87	87	15	3	38	35
0	7	43	45	−1	10	42	46	8	1	34	30	−12	4	92	94
2	7	65	68	0	10	72	73	9	1	30	33	−10	4	18	19
3	7	32	33	1	10	68	69	10	1	101	101	−9	4	18	19
4	7	23	25	2	10	43	43	12	1	42	47	−6	4	16	9
5	7	16	11	4	10	23	21	15	1	21	16	−5	4	41	40

Obsd and calcd. structure amplitudes (× 10) $C_{12}H_{24}N_2H_6Cl_2 \cdot H_2O$

H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL
6	7	30	30	5	10	64	61	−15	2	49	48	−4	4	83	88
7	7	74	80	6	10	32	30	−14	2	40	40	−3	4	94	92
8	7	45	49	9	10	25	19	−13	2	18	20	−2	4	35	35
9	7	27	25	−7	11	26	30	−7	2	124	127	−1	4	14	6
11	7	33	31	−6	11	54	52	−6	2	52	48	0	4	21	13
−13	8	41	46	−5	11	37	37	−5	2	15	7	4	4	43	45
−12	8	54	54	−2	11	32	36	−3	2	17	18	5	4	111	112
−11	8	21	29	0	11	18	17	−2	2	23	23	7	4	39	42
−10	8	27	18	7	11	18	12	−1	2	20	18	8	4	20	22
−9	8	41	37	−6	12	51	51	1	2	18	20	11	4	18	14
−8	8	52	51	−5	12	77	75	2	2	161	162	12	4	23	26
−7	8	33	32	−2	12	43	43	4	2	65	65	14	4	24	30
−6	8	69	69	−1	12	35	31	5	2	16	20	−13	5	38	36
−4	8	37	37	0	12	36	36	6	2	18	10	−12	5	76	76
−3	8	71	70	3	12	37	31	7	2	23	25	−11	5	30	24
−2	8	82	81	4	12	44	43	8	2	40	36	−10	5	52	51
−1	8	66	71	5	12	35	35	9	2	17	17	−8	5	34	33
2	8	77	78		**K = 5****			10	2	35	36	−7	5	59	59
3	8	30	30	0	0	54	54	11	2	43	47	−6	5	61	63
4	8	37	37	1	0	103	98	13	2	42	42	−5	5	46	45
6	8	15	9	2	0	29	28	14	2	23	17	−4	5	20	19
7	8	47	47	4	0	15	12	−15	3	59	55	−3	5	150	149
8	8	74	78	5	0	19	10	−13	3	86	83	−2	5	45	53
9	8	17	13	7	0	40	37	−11	3	60	58	−1	5	73	74
10	8	27	22	9	0	18	19	−10	3	91	93	0	5	39	35
11	8	42	38	10	0	75	76	−9	3	93	93	1	5	18	12
−11	9	26	21	11	0	19	13	−8	3	51	50	2	5	40	40
−10	9	36	37	−15	1	39	34	−7	3	20	25	3	5	55	54
−9	9	19	19	−14	1	60	58	−6	3	178	179	4	5	68	70
−8	9	71	72	−13	1	58	56	−5	3	63	70	5	5	78	77
−5	9	61	59	−12	1	34	36	−4	3	127	126	6	5	36	39
−4	9	24	25	−10	1	64	60	−3	3	58	56	7	5	85	86
−3	9	20	10	−9	1	133	131	−2	3	31	29	8	5	44	45
−1	9	27	25	−8	1	15	16	−1	3	104	106	9	5	40	41

0	9	18	12	−7	1	92	91	0	3	145	149	13	5	28	24
1	9	17	20	−6	1	16	13	1	3	140	137	−13	6	19	13
−12	6	36	31	−5	9	44	48	−6	2	43	44	−1	5	70	74
−10	6	45	43	−4	9	46	47	−5	2	26	27	0	5	43	38
−9	6	61	62	−2	9	31	33	−4	2	63	62	1	5	29	31
−8	6	18	17	−1	9	49	57	−2	2	19	24	2	5	77	78
−6	6	22	21	0	9	47	52	−1	2	25	32	3	5	79	83
−4	6	27	24	1	9	24	23	0	2	43	49	4	5	31	31
−3	6	68	65	2	9	43	43	2	2	25	29	6	5	25	23
−2	6	22	26	3	9	59	61	3	2	37	41	7	5	46	46
−1	6	20	25	4	9	59	56	4	2	37	37	8	5	32	36
0	6	50	50	5	9	51	54	5	2	31	32	−8	6	73	70
1	6	22	25	6	9	27	31	7	2	34	32	−7	6	34	33
4	6	17	15	8	9	33	35	8	2	51	53	−6	6	48	47
6	6	28	27	−5	10	59	63	−11	3	17	1	−5	6	54	55
7	6	29	33	−3	10	21	17	−10	3	40	43	−4	6	20	20
8	6	48	54	2	10	33	33	−9	3	51	51	−3	6	20	21
10	6	21	12	4	10	21	19	−8	3	22	22	−1	6	18	10
−12	7	24	31	−1	11	21	23	−7	3	31	26	0	6	21	23
−10	7	49	49		**K = 6****			−6	3	30	32	1	6	80	84
−9	7	135	137	0	0	54	56	−5	3	55	60	3	6	31	30
−8	7	17	15	1	0	43	47	−4	3	37	36	4	6	38	41
−7	7	34	30	2	0	27	25	−3	3	49	49	6	6	20	18
−6	7	22	19	3	0	37	42	−1	3	61	68	−7	7	65	63
−5	7	59	61	4	0	58	56	0	3	67	71	−6	7	32	34
−4	7	73	76	5	0	48	49	1	3	33	29	−5	7	19	19
−3	7	58	56	7	0	36	37	2	3	23	25	−4	7	39	43
−2	7	18	19	9	0	25	25	4	3	33	38	−3	7	81	87
0	7	128	134	10	0	40	37	5	3	32	31	1	7	32	32
1	7	72	76	11	0	60	56	6	3	23	21	2	7	69	68
2	7	55	59	12	0	30	32	8	3	18	22	5	7	37	33
4	7	40	40	−12	1	52	55	9	3	32	27	6	7	54	53
5	7	54	56	−10	1	33	36	10	3	48	53	−3	8	27	25
6	7	34	35	−9	1	47	50	−10	4	72	77	−2	8	59	59
7	7	18	14	−8	1	68	70	−9	4	38	39	4	8	41	43
8	7	25	29	−7	1	42	43	−8	4	20	21		**K = 7****		
10	7	59	52	−4	1	86	93	−7	4	25	26	0	0	49	51
11	7	28	30	−3	1	102	104	−6	4	43	43	1	0	45	44

Obsd and calcd. structure amplitudes (× 10) $C_{12}H_{24}N_2H_6Cl_2 \cdot H_2O$

H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL	H	L	FOBS	FCAL
−9	8	39	40	−2	1	69	71	−5	4	19	15	5	0	54	60
−8	8	71	71	−1	1	53	54	−4	4	37	39	−5	1	19	14
−7	8	20	24	0	1	18	26	−3	4	52	54	−4	1	43	40
−6	8	35	35	1	1	65	67	−2	4	71	66	−3	1	57	59
−5	8	17	15	2	1	74	77	−1	4	52	57	4	1	19	18
−3	8	31	23	3	1	53	62	0	4	39	39	5	1	49	48
−2	8	37	41	5	1	35	38	1	4	33	35	−5	2	30	31
−1	8	18	16	6	1	75	77	3	4	27	27	−3	2	27	29
0	8	65	66	7	1	99	104	5	4	23	21	−2	2	85	95
1	8	28	21	8	1	19	16	7	4	26	23	−1	2	33	34
2	8	24	20	9	1	19	21	−10	5	44	39	1	2	26	30
3	8	27	29	10	1	19	9	−9	5	70	70	2	2	56	60
7	8	24	29	11	1	42	41	−8	5	34	37	3	2	58	64
9	8	27	24	12	1	53	57	−7	5	66	71	4	2	40	48
−9	9	40	38	−9	2	61	57	−6	5	84	88	−1	3	68	68
−8	9	30	28	−8	2	33	34	−3	5	41	40	0	3	24	25
−6	9	114	115	−7	2	47	46	−2	5	77	76				