

## Vibrational spectra of 1,6-dichlorohexane, *n*-chloro-, 2-chloro- and 3-chlorohexanes

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**Abstract.** Infrared spectra of 1,6-dichlorohexane have been recorded with an FT-IR spectrometer at ambient temperatures as neat liquid and at liquid N<sub>2</sub> temperatures for crystalline thin films. Raman spectra of the compound have been obtained using Ar<sup>+</sup> and Kr<sup>+</sup> lasers as sources of excitation at ambient temperatures. FT-IR and laser Raman spectra of *n*-chlorohexane, 2-chlorohexane and 3-chlorohexane molecules have also been recorded as neat liquids at ambient temperatures. Most of the observed bands of the liquid 1,6-dichlorohexane have been found to correspond to the molecular symmetry C<sub>2h</sub>. Crystal phase spectra have been assigned assuming this symmetry for the compound. Vibrational spectra of *n*-chloro-, 2-chloro- and 3-chlorohexanes have been assigned assuming C<sub>s</sub> as the highest symmetry in the planar configuration of each of these molecules which are found to exist in more than one conformation in the liquid phase.

**Keywords.** Vibrational spectra; infrared and Raman spectra; 1,6-dichlorohexane, *n*-chloro-, 2-chloro- and 3-chlorohexanes.

### 1. Introduction

Vibrational spectra of *n*-alkyl halides were taken by Brown and Sheppard (1954) to study molecular conformations and to understand the spectra of fully extended polymethylene chains. Such studies (Snyder and Schachtschneider 1963; Schachtschneider and Snyder 1963), along with the normal coordinate calculations of *n*-paraffins have led to a detailed analysis of the spectra of these compounds. Vibrational analysis of a series of *n*-alkylchlorides, ethyl through *n*-hexyl, have been made by Snyder and Schachtschneider (1969) by recording the infrared spectra by the vapour deposition method at liquid N<sub>2</sub> temperatures and carrying out normal coordinate calculations. Vibrational spectra of primary and secondary alkyl bromides have been studied by Crowder and co-workers (Crowder and Iwunze 1977; Crowder and Jalilian 1977) by recording the infrared and Raman spectra of these compounds at the ambient as well as liquid N<sub>2</sub> temperatures (infrared only), and carrying out normal coordinate calculations. These authors have analysed the spectra to determine the various conformations present in the neat liquid and also to determine the force fields. Vibrational spectra of 1,6-dichlorohexane and secondary chlorohexanes, however, are not available in literature.

The present communication reports briefly the results of the analyses of vibrational

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spectra of 1,6-dichlorohexane as well as *n*-chloro-, 2-chloro- and 3-chlorohexanes. 1,6-dichlorohexane was expected to possess a centre of symmetry in its most symmetric conformation in the liquid phase. So a study of its vibrational spectra was considered to be very interesting and valuable. The vibrational spectra of *n*-chloro-, 2-chloro- and 3-chlorohexanes were studied in comparison with those of their bromo counterparts. Changes in the spectra owing to the change in position of the halogen atom from the one to the two and three positions were also studied.

## 2. Experimental

1,6-dichlorohexane, *n*-chloro-, 2-chloro- and 3-chlorohexanes were obtained in pure form from M/s Tokyo Kasei Kogyo Co. Ltd., Tokyo (Japan) and used as such to record the infrared and Raman spectra. The infrared spectra were recorded as neat liquids with a Nicolet MX-1 FT-IR spectrometer at ambient temperatures. The infrared spectrum of 1,6-dichlorohexane was also recorded as a thin crystalline film by the vapour deposition method at liquid N<sub>2</sub> temperatures. Raman spectra were recorded on a Beckman model 700 spectrometer equipped with a Coherent Radiation model 54 Ar<sup>+</sup> laser and also on a Cary model 82 spectrometer using Kr<sup>+</sup> laser at ambient temperature. The measurements are accurate to within  $\pm 3 \text{ cm}^{-1}$  in case of infrared and  $\pm 5 \text{ cm}^{-1}$  for Raman bands.

## 3. Results and discussion

1,6-dichlorohexane may exist in the neat liquid in four distinct conformations. The most symmetric among these has a planar *trans* configuration, in which both the chlorine atoms lie in the plane of the C-C skeleton, and belongs to the *C*<sub>2h</sub> point group. The other *trans* configuration is one in which the two chlorine atoms are not in the plane of the molecule and has the symmetry *C*<sub>i</sub>. In the third conformation, both the chlorine atoms are on one side of the molecular plane with *C*<sub>2</sub> symmetry for the molecule and the fourth conformation has a *C*<sub>1</sub> symmetry.

A close scrutiny of table 1 shows that twenty-two bands out of a total of thirty-three bands observed in the infrared spectra of the crystalline 1,6-dichlorohexane do not have their counterparts in the liquid-phase Raman spectrum of the compound. Most of the remaining eleven bands which show their counterparts (with an error upto  $\pm 5 \text{ cm}^{-1}$ ) are very weak. Fourteen bands out of a total of thirty-six Raman bands of the liquid compound do not have their counterparts in the infrared spectra of the liquid. Similarly fifteen bands out of a total of thirty-six infrared bands of the liquid sample do not have counterparts in the Raman spectrum.

The above observations suggest that there are several conformers of the compound in liquid phase. However, most prominent among these has either *C*<sub>2h</sub> or *C*<sub>i</sub> symmetry. If it had *C*<sub>i</sub> symmetry, then all the bands observed in the Raman spectra should be polarized. But we do find some depolarized Raman bands having no counterparts in the infrared spectra. This suggests that the most prominent conformer of the compound in the liquid phase has *C*<sub>2h</sub> symmetry. Since there are also such bands of the liquid which appear both in Raman as well as infrared spectra with comparable relative intensities, it is concluded that the last two conformers of symmetry *C*<sub>2</sub> and *C*<sub>1</sub> are

Table 1. Observed infrared and Raman bands of 1,6-dichlorohexane.

Liquids				Solid (crystalline)		
IR ( $\text{cm}^{-1}$ )	Relative intensity	Raman ( $\text{cm}^{-1}$ )	Relative intensity	Polarization	IR ( $\text{cm}^{-1}$ )	Relative intensity
2990	<i>vvs</i>	2995	<i>m</i>	D	1468	<i>vvs</i>
2960–2910( <i>b</i> )	<i>vvs</i>	2955	<i>vs</i>	P	1452	<i>vvs</i>
2865	<i>vvs</i>	2937	<i>vs</i>	P	1441	<i>s</i>
1462	<i>vvs</i>	2906	<i>vs</i>	P	1420	<i>w(sh)</i>
1445	<i>vvs</i>	2865	<i>s</i>	P	1393	<i>vw</i>
1432	<i>vvs(sh)</i>	1442	<i>s</i>	D	1373	<i>ms(sh)</i>
1374	<i>mw(sh)</i>	1432	<i>s(sh)</i>	D	1357	<i>vvs</i>
1354	<i>m(sh)</i>	1390	<i>vw</i>		1320	<i>vvw</i>
1346	<i>ms</i>	1345	<i>vw</i>		1290	<i>vvs</i>
1309	<i>vvs</i>	1318	<i>mw(sh)</i>		1255	<i>m</i>
1278	<i>vvs</i>	1305	<i>m</i>	D	1242	<i>w(sh)</i>
1238	<i>ms</i>	1240	<i>vvw</i>		1204	<i>vs</i>
1178	<i>vvw</i>	1157	<i>vvw</i>		1185	<i>vw(sh)</i>
1158	<i>vvw</i>	1130	<i>vvw</i>		1160	<i>vvw</i>
1083	<i>w(sh)</i>	1081	<i>mw(sh)</i>	P(?)	1152	<i>vvw</i>
1059	<i>m(sh)</i>	1063	<i>m</i>	P(?)	1133	<i>vvw</i>
1038	<i>ms</i>	1027	<i>vw</i>		1123	<i>w</i>
1019	<i>m</i>	930	<i>vw</i>		1103	<i>m</i>
985	<i>mw</i>	908	<i>vw</i>		1049	<i>vvs</i>
950	<i>m(sh)</i>	902	<i>vw(sh)</i>		1002	<i>s</i>
932	<i>m(sh)</i>	860	<i>vw</i>		960	<i>mw</i>
904	<i>s</i>	812	<i>w</i>	P	898	<i>vw</i>
868	<i>m</i>	775	<i>w</i>	P	889	<i>vw</i>
855	<i>m</i>	755	<i>w(sh)</i>	P	874	<i>m</i>
840	<i>w</i>	745	<i>vw(sh)</i>		860	<i>vw</i>
815	<i>m</i>	731	<i>mw(sh)</i>	P(?)	802	<i>w(sh)</i>
780	<i>w(sh)</i>	724	<i>ms</i>	P	777	<i>vs</i>
730	<i>vvs</i>	715	<i>ms(sh)</i>	P	768	<i>mw(sh)</i>
651	<i>vvs</i>	651	<i>vvs</i>	P	730	<i>vvs</i>
565	<i>vvw</i>	535	<i>vvw</i>		700	<i>m</i>
540	<i>vw</i>	498	<i>vvw</i>		682	<i>w</i>
503	<i>mw</i>	445	<i>w</i>		668	<i>mw</i>
480	<i>w</i>	404	<i>vvw</i>		651	<i>w</i>
455	<i>mw</i>	345	<i>w(sh)</i>	P		
440	<i>vvw</i>	334	<i>w</i>			
420	<i>m</i>	235	<i>m</i>	P		

*b* = broad band; *v* = very; *w* = weak; *s* = strong; *m* = medium; *sh* = shoulder; P = polarized; D = depolarized

also present in the liquid in some proportion. Symmetry coordinates for the  $C_{2k}$  configuration of the molecule have been constructed to identify the normal mode descriptions and to facilitate assignment of observed fundamental frequencies to these modes.

Concentrating on the spectral range corresponding to C–Cl stretch vibration we find that while four bands at 731, 724, 715 and 651  $\text{cm}^{-1}$  have been observed in the Raman spectra, only two very strong infrared bands at 730 and 651  $\text{cm}^{-1}$  appear for the liquid. Of the four Raman bands, the one at 731  $\text{cm}^{-1}$  is weak while the rest are

of good intensities. In the solid phase infrared spectra five bands at 730, 700, 682, 668 and 651  $\text{cm}^{-1}$  have been observed. Of these the first is very strong, the second has medium intensity and the rest are weak. This shows that a situation exists in the solid phase as well which is similar to that in the liquid phase of the compound where all the probable conformations are present. This suggests that the thin film of the compound whose infrared spectra were obtained at liquid  $\text{N}_2$  temperatures was partly in the amorphous form, although most of it was in the crystalline form with the molecular conformation of  $C_{2h}$  symmetry. Most of the bands of the spectra can thus be explained in terms of the various modes of vibration of this conformation of the compound as given in table 2. Extensive use of the literature available on normal coordinate analysis of *n*-alkanes, primary and secondary halogenated alkanes and related compounds has been made in the assignment of bands of the vibrational

**Table 2.** Assignment of bands of 1,6-dichloronhexane assuming  $C_{2h}$  symmetry.

Symmetry species	Modes of vibration	Raman ( $\text{cm}^{-1}$ )	Symmetry species	Modes of vibration	IR ( $\text{cm}^{-1}$ )
<i>A<sub>g</sub></i>	CH <sub>2</sub> X(sym st)	2955	<i>B<sub>u</sub></i>	CH <sub>2</sub> X(sym st)	2960–2910( <i>b</i> )
	CH <sub>2</sub> (sym st)	2937		CH <sub>2</sub> (sym st)	2960–2910( <i>b</i> )
	CH <sub>2</sub> (sym st)	2906		CH <sub>2</sub> (sym st)	—
	CH <sub>2</sub> (sym def)	1390		CH <sub>2</sub> (asym def)	1462
	CH <sub>2</sub> (sym def)	—		CH <sub>2</sub> (asym def)	1374
	CH <sub>2</sub> (wag)	1318			1354
	CH <sub>2</sub> (wag)	1305		CH <sub>2</sub> (wag)	1309
	C–C(st)	1027		CH <sub>2</sub> (wag)	1278
	C–C(st)	908		C–C(st)	1038
	C–C(st)	860			1019
	C–X(sym st)	724		C–C(st)	985
	C–C–C(def)	535			950
	CH <sub>2</sub> X(sym def)	498		C–X(asym st)	730
	CH <sub>2</sub> X(wag)	415		C–C–C(def)	565
	C–C–C(def)	415			540
		404		CH <sub>2</sub> X(asym def)	503
	C–C–X(sym def)	245		C–C–C(def)	480
		234		CH <sub>2</sub> X(wag)	455
	CH <sub>2</sub> X(asym st)	—		C–C–X(asym def)	—
<i>B<sub>g</sub></i>	CH <sub>2</sub> (asym st)	2995	<i>A<sub>u</sub></i>	CH <sub>2</sub> (asym st)	2990
	CH <sub>2</sub> (asym st)	2995		CH <sub>2</sub> (asym st)	2990
	CH <sub>2</sub> (tw)	1130		CH <sub>2</sub> X(asym st)	2960–2910( <i>b</i> )
	CH <sub>2</sub> (tw)	1063		CH <sub>2</sub> (tw)	1238
		1027		CH <sub>2</sub> (tw)	1178
	CH <sub>2</sub> (rock)	775		CH <sub>2</sub> (rock)	868
		755			855
	CH <sub>2</sub> (rock)	745			840
	CH <sub>2</sub> X(tw)	404		CH <sub>2</sub> (rock)	780
	CH <sub>2</sub> X(rock)	345		CH <sub>2</sub> X(tw)	440
	CH <sub>2</sub> X–(τ)	—		CH <sub>2</sub> X(rock)	420
	CH <sub>2</sub> –(τ)	—		CH <sub>2</sub> X–(τ)	—
				CH <sub>2</sub> –(τ)	—
				CH <sub>2</sub> –(τ)	—

st = stretch, def = deformation, asym = asymmetric, wag = wagging, tw = twisting, τ = torsion.

Table 3. Assignment of bands of *n*-chlorohexane.

Present work (liquid)		Previous work (solid)		Assignment	Present work (liquid)		Previous work (solid)		Assignment
IR (cm <sup>-1</sup> )	Raman (cm <sup>-1</sup> )	<i>Trans</i> (cm <sup>-1</sup> )	<i>Gauche</i> (cm <sup>-1</sup> )		IR (cm <sup>-1</sup> )	Raman (cm <sup>-1</sup> )	<i>Trans</i> (cm <sup>-1</sup> )	<i>Gauche</i> (cm <sup>-1</sup> )	
2995	2995	3002	3002	CH <sub>2</sub> Cl (asym st)	994	990	—	998	C-C (st)
—	2988	—	—		985	—	—	—	
2960	2957	2961*	2965*	CH <sub>3</sub> (asym st)	970	965	—	—	C-C (st)
2940	2938	—	—	CH <sub>2</sub> (asym st)	952	—	—	954	
2920	—	2922*	2922*	CH <sub>2</sub> (asym st)	932	—	—	—	CH <sub>3</sub> (rock)
2910	2912	2912*	2912	CH <sub>2</sub> (asym st)	915	—	—	—	
—	2904	—	—	CH <sub>2</sub> (asym st)	904	—	—	—	C-C (st)
—	2877	2877*	2877*	CH <sub>3</sub> (sym st)	—	890	891	890	
2865	2865	2864*	—	CH <sub>2</sub> (sym st)	868	—	—	866	CH <sub>2</sub> Cl (rock)
2860	—	2858*	2858*	CH <sub>2</sub> (sym st)	—	862	—	—	
—	2855	2853*	2850*	CH <sub>2</sub> (sym st)	856	—	—	—	CH <sub>2</sub> Cl (rock)
1462	—	—	1461	CH <sub>2</sub> (def)	840	845	—	—	
—	1455	1456	—		—	—	821	—	820
—	1447	1449	1449	CH <sub>3</sub> (asym def)	815	—	—	—	CH <sub>2</sub> (rock)
1445	1445	1444	1445	CH <sub>3</sub> (asym def)	803	807	—	—	
—	1442	—	1439	CH <sub>2</sub> (def)	780	—	790	—	CH <sub>2</sub> (rock)
1432	1436	1436	—	CH <sub>2</sub> Cl (wag)	—	756	—	763	
1372	1375	1380	1380	CH <sub>3</sub> (sym def)	735	—	735	—	CH <sub>2</sub> (rock)
1352	—	—	—	CH <sub>2</sub> (wag)	730	—	727	—	
1346	1345	—	—		—	725	728	722	—
1310	—	—	1310	CH <sub>2</sub> (tw)	651	651	—	653	C-Cl (st)
—	1305	1306	1302	CH <sub>2</sub> (wag)	610	—	—	—	
1277	—	1273	—	CH <sub>2</sub> (tw)	601	—	—	—	C-Cl (st)
—	1267	—	1267	CH <sub>2</sub> (tw)	565	—	—	—	
1238	—	1242	—	CH <sub>2</sub> (wag)	—	557	—	—	C-C-C (def)
1202	1206	—	1211	CH <sub>2</sub> (wag)	538	—	—	—	
—	—	—	—	C-C (st)	—	530	—	—	C-C-C (def)
1185	1180	1189	1188	CH <sub>3</sub> (rock)	512	—	—	—	
1178	1173	—	—	C-C (st)	502	—	—	494	C-C-C (def)
1157	—	—	—	CH <sub>2</sub> (tw)	482	490	—	—	
1116	1116	1120	1114	CH <sub>2</sub> Cl (tw)	473	475	—	—	C-C-C (def)
—	1100	1094	1098		—	455	454	453	
1082	—	—	—	C-C (st)	440	—	—	—	C-C-C (def)
—	1076	—	—		—	416	—	415	
—	1065	1062	1062	C-C (st)	—	385	—	387	C-C-C (def)
1056	—	—	1056		—	—	370	—	
1036	—	1032	—	C-C (st)	—	340	—	344	C-C-Cl (def)
—	1023	—	—		—	—	290	—	
1018	—	—	1014	C-C (st)	—	265	266*	—	C-C-Cl (def)
—	1002	1001	—		—	—	—	—	

st = stretch, def = deformation, asym = asymmetric, wag = wagging, tw = twisting; \*calculated values

spectra of the present compound. Normal coordinate calculations are in progress and will be published elsewhere.

There are five probable conformations for *n*-chlorohexane in the neat liquid. In only one of these is the Cl atom coplanar with the six carbon atoms of the molecule. While the planar conformation belongs to *C<sub>s</sub>* symmetry, all the rest possess *C<sub>1</sub>* symmetry.

**Table 4.** Assignment and correlation of bands of 2-chloro- and 3-chloro-hexanes (liquids) with their bromo counterparts.

2-Chlorohexane		2-Bromohexane*		3-Chlorohexane		3-Bromohexane*		Assignments
Infrared (cm <sup>-1</sup> )	Raman (cm <sup>-1</sup> )	Infrared (cm <sup>-1</sup> )	Raman (cm <sup>-1</sup> )	Infrared (cm <sup>-1</sup> )	Raman (cm <sup>-1</sup> )	Infrared (cm <sup>-1</sup> )	Raman (cm <sup>-1</sup> )	
1466	—	1466	—	—	—	—	—	CH <sub>2</sub> (def)
1460	—	1461	1457	1460	—	1461	—	CH <sub>2</sub> (def)
1445	1448	1454	—	—	1450	1455	1454	CH <sub>2</sub> (def)
								CH <sub>3</sub> (asym def)
1432	—	1428	—	1437	1440	1437	1441	CH <sub>2</sub> (asym def)
1378	1381	1380	1386	1381	1377	1381	1382	CH <sub>3</sub> (sym def)
1346	—	1346	—	1345	1340	1346	—	CH <sub>3</sub> (sym def)
1304	1304	1301	—	1307	1306	1310	1308	CH <sub>2</sub> (wag)
—	—	—	—	—	1300	1300	—	CH <sub>2</sub> (wag)
1293	1291	1291	—	1287	1287	1282	—	CH <sub>2</sub> (wag)
1262	1265	1260	—	1261	1260	1259	1260	} CH <sub>2</sub> (tw)
1246	—	1253	—	1255	—	1240	—	
—	1240	1239	1245	—	—	1232	1235	} CH <sub>2</sub> (tw)
1205	1208	1195	1201	1217	1217	1196	1197	
1160	—	—	—	—	—	—	—	} CH <sub>2</sub> (tw)
1151	1151	1149	1155	1150	—	—	—	
1144	—	—	—	1143	1143	1143	1142	
1127	1128	1131	1136	1136	—	—	—	} C-C (st)
—	1120	—	—	1116	1117	1117	1120	
1091	1091	1090	1096	1091	1091	1081	1084	C-C (st)
1065	1068	1066	1075	1063	1060	1055	1048	C-C (st)
—	1017	1019	1029	1027	1029	1028	1029	C-C (st)
985	982	1000	1007	1005	1001	1001	—	CH-X (def)
973	974	981	986	980	980	980	980	CH <sub>3</sub> (rock)
915	—	915	—	905	902	903	898	CH <sub>3</sub> (rock)
905	—	905	907	895	895	889	—	C-C (st)
896	894	900	—	888	—	—	—	} CH <sub>2</sub> (rock)
881	—	886	881	878	876	873	872	
—	872	—	—	867	—	—	—	} CH <sub>2</sub> (rock)
828	826	829	837	818	—	829	832	
811	813	810	818	818	—	—	—	} CH <sub>2</sub> (rock)
793	790	—	—	808	805	791	792	
745	745	731	—	742	—	747	742	CH <sub>2</sub> (rock)
673	672	579	586	660	660	590	—	} C-X (st)
—	—	—	—	636	633	—	—	
615	612	—	—	607	602	—	538	} C-X (st)
555	—	—	—	578	—	—	—	
550	548	540	548	534	533	—	—	X-C-H (def)
—	—	508	—	512	—	495	496	C-C-C (def)
485	487	475	—	—	—	—	—	CH X (rock)
465	465	455	—	445	—	—	—	} C-C-C (def)
440	440	—	437	445	438	430	432	
426	427	415	421	416	415	—	—	} C-C-C (def)
—	393	—	402	—	391	398	403	
—	375	—	—	—	367	—	—	} C-C-C (def)
—	365	359	363	—	351	352	—	
—	338	347	347	—	324	—	343	
—	294	295	295	—	306	285	283	} C-X (def)
—	262	—	222	—	—	—	227	

st = stretch, def = deformation, asym = asymmetric, wag = wagging, rock = rocking and tw = twisting, X = Cl, Br; \*data from the literature

Each of the 2-chloro- and 3-chlorohexanes may exist in six conformations in liquid phase and in none of these is the Cl atom co-planar with the carbon atoms of the molecule. Thus all these conformations belong to the  $C_1$  point group. Symmetry, therefore, is hardly of any significance in assignment of bands in these molecules.

Snyder and Schachtschneider (1969) have shown that while the spectrum of the unannealed deposit of *n*-chlorohexane indicates the presence of *trans* and *gauche* conformations, the well-annealed sample consists of the *trans* form only. A comparison of these data as well as those of the *n*-chlorohexane liquid obtained in the present studies has been made in table 3. This shows the presence of additional conformations of *n*-chlorohexane in the liquid phase as many normal modes could be associated with several frequencies. Generous use of the results of normal coordinate calculations available in the literature for this molecule has been made in the assignment of bands as shown in table 3.

Tentative assignments and correlation of bands of 2-chloro- and 3-chlorohexanes have been given in table 4. Extensive use of the results of normal coordinate calculations for their bromo counterparts available in literature has been made. A comparison of the assigned bands of these compounds has also been given in table 4. Normal coordinate calculations are in progress and will appear elsewhere.

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