

force constants derived in this way are given in Table 6. A totally independent derivation of bond force constants is possible by means of vibrational spectroscopy. Infrared stretching-force constants can be calculated accurately by complete assignment of the whole spectrum of the compound or approximately from the position of the symmetric stretching frequency of the anion in the compound (preferably in the solid state, although force constants have proved to be characteristic of the bond and fairly independent of the compound). For the compounds under discussion, the infrared force constants are 5.48 (K_2CrO_4 ; Stammreich, Bassi & Sala, 1958), 10.26 (K_2SO_4 ; Rosenthal, 1934), 5.2 (K_2MnO_4 ; from the infrared results of Rocchiccioli, 1963), and 6.1 millidynes/ \AA (K_2SeO_4 ; from the infrared results of Tarte & Nizet, 1964). Comparison of the force constants derived in these two ways enables the effective charges upon the chemical species in the crystal lattice to be estimated. For potassium chromate, potassium sulphate and possibly potassium manganate, the charges upon A (=Cr, S, Mn respectively), K and O appear to be close to 2.0, 1.0 and -1.0. For the purposes of this calculation, the structure of potassium selenate needs to be redetermined. Force constants derived from the crystallographic results with these values for the charges are rather smaller than those derived from vibrational spectroscopy, but they do show the right trend in going from potassium chromate to potassium sulphate. Closer agreement can be achieved by increasing the charge separation within the anions. However, forces other than electrostatic are important in the solid state and at least the short-range repulsive forces should be included before accurate force constants can be expected. However, several approximations must be

made before these short-range forces can be estimated and much of the value of the electrostatic calculation detailed above would be obscured.

It should be noted that, whilst there are significant differences in the chromium-oxygen band lengths observed in potassium chromate, there will not necessarily be three observable infrared bands corresponding to the stretching modes. Since equation (2) appears to be valid, the absorption frequency for all three bonds would be expected to be nearly equal. The mere fact that a 'symmetric' stretching frequency is observable in the infrared spectrum is indicative of the fact that the anion is distorted in the solid state.

(iv) If this procedure is valid then any observed deviations must be assigned to specific causes. For example, in ammonium sulphate there should be extra forces due to hydrogen bonding, and calculations on this compound are in progress.

References

- CROMER, D. T. (1965). *Acta Cryst.* **18**, 17.
- CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104. *Intermolecular Forces and Packing in Crystals* (1970). *Trans. ACA*, **6**.
- KÁLMÁN, A., STEPHENS, J. S. & CRUICKSHANK, D. W. J. (1970). *Acta Cryst.* **B26**, 1451.
- PALENÍK, G. J. (1967). *Inorg. Chem.* **6**, 507.
- ROBINSON, M. T. (1958). *J. Phys. Chem.* **62**, 925.
- ROCCHICCIOLI, C. (1963). *C.R. Acad. Sci. Paris*, **256**, 1707.
- ROSENTHAL, J. E. (1934). *Phys. Rev.* **46**, 730.
- STAMMREICH, H., BASSI, D. & SALA, O. (1958). *Spectrochim. Acta*, **12**, 403.
- TARTE, P. & NIZET, G. (1964). *Spectrochim. Acta* **20**, 503.
- ZACHARIASEN, W. H. (1967). *Acta Cryst.* **23**, 558.
- ZACHARIASEN, W. H. & ZIEGLER, G. E. (1931). *Z. Kristallogr.* **80**, 164.

Acta Cryst. (1972). **B28**, 2852

The Crystal and Molecular Structure of *cis*-1,3,5-Trichlorocyclohexane

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The crystal and molecular structure of *cis*-1,3,5-trichlorocyclohexane, a cyclic form of polyvinyl chloride, has been determined from X-ray data by direct methods and confirmed by Patterson techniques. The structure was refined by block-diagonal least-squares methods using three-dimensional data. The unit cell is monoclinic, space group $C2/c$ with $a = 22.131$, $b = 8.803$, $c = 9.033 \text{ \AA}$, $\beta = 101.3^\circ$ and $Z = 8$. The final R value based on 1399 independent observed reflexions is 6.3 %.

Introduction

The crystal and molecular structure of *cis*-1,3,5-trichlorocyclohexane, a cyclic trimer model of polyvinyl chloride, was determined as part of a systematic study of the conformational structure of polyvinyl chloride model compounds (Obereigner, Doskočilová & Štokr, 1968). Colourless crystals, purified in the final stage

by vacuum sublimation, were prepared by Dr B. Oberigner of the Institute of Macromolecular Chemistry, ČSAV, Prague.

Experimental

The elementary cell parameters were derived by the least-squares technique from rotation and Weissen-

berg photographs calibrated with Si powder lines. Crystal data:

$C_6H_9Cl_3$, M.W. 187.50, m.p. 61–62°C.

Monoclinic:

$a=22.131$, $b=8.803$, $c=9.033$ Å

$\beta=101.3^\circ$, $V=1726$ Å 3 , $F(000)=768$

$\lambda(Mo\ K\alpha)=0.7107$ Å, $\mu(Mo\ K\alpha)=9.62$ cm $^{-1}$

$Z=8$, $D_c=1.44$, $D_m=1.42$ (1) g.cm $^{-3}$ by flotation in aqueous potassium iodide.

Absent reflexions: hkl when $h+k$ odd, $h0l$ when l odd. Space group Cc or $C2/c$. The latter was indicated by statistical tests and confirmed by the subsequent refinement.

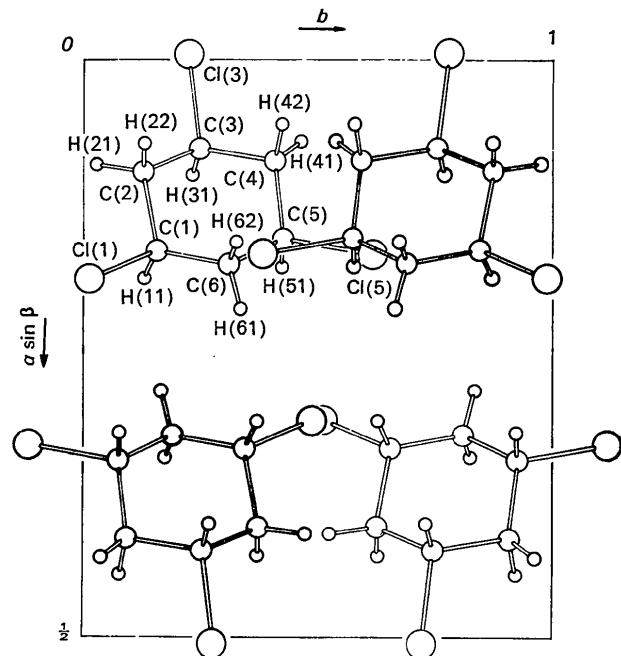


Fig. 1. The structure viewed along c .

Because of the slow evaporation of crystals in the atmosphere, they were sealed in Lindemann-glass tubes during the X-ray examination. Two independent sets of intensities were collected. The first one, kindly supplied by Professor G. D. Andreatti and Dr P. Sgarabotto (University of Parma) and provided by an automatic Siemens diffractometer on line with a Siemens 304/P computer, was used for the direct methods phase determination. The other set of intensities, measured with a manual Hilger & Watts diffractometer in our laboratory, was used for the Patterson synthesis and the final refinement of the structure. The latter set of intensities was measured by the ω -scan method, with a scintillation counter and normal beam arrangement. Zirconium-filtered Mo $K\alpha$ radiation was used, with pulse-height discrimination and Al attenuation filters. No corrections were made for absorption or extinction. Data were collected for 1803 reflexions, of which 1399 were observed and used for refinement. The absolute scale and approximate overall temperature factors were determined from all three-dimensional data by the Wilson method (Ahmed, Hall, Pippy & Huber, 1966). All the calculations were performed on an IBM 7040 computer.

TABLE I. Structure determination and refinement

The structure was determined by the symbolic-addition procedure of Karle & Karle (1966) with the revised programs of Ahmed *et al.* (1966). A Fourier synthesis of 276 E values clearly showed the positions of all the chlorine and carbon atoms. As a check, a three-dimensional Patterson summation was performed and interpreted, giving an orientation of the molecule that was identical with that obtained from the E map. The R index ($\sum|F_o|-|F_c|/\sum|F_o|$, for observed reflexions only) for the trial structure was 33%. Refinement was by block-diagonal least squares, with the use of the pro-

Table 1. Final parameters and e.s.d.'s of non-hydrogen atoms

Temperature factor = $\exp [-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$.

B_t (Å 2) are the temperature factors corresponding to the principal radii of the thermal motion ellipsoids. The other values are all $\times 10^5$.

	x/a	y/b	z/c	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	B_1	B_2	B_3
C(1)	16366 17	15319 40	24846 39	279 7	1399 39	1358 38	-88 65	192 26	8 27	5.35	4.43	4.16
C(2)	9555 17	12735 39	18001 50	255 7	1310 40	2057 56	-194 76	72 32	-191 26	5.12	7.00	3.54
C(3)	7581 17	24665 44	6006 50	236 7	1525 42	1979 54	-378 81	-171 30	-96 27	4.94	7.69	3.44
C(4)	8617 17	40697 42	11915 52	252 7	1414 42	2139 60	18 81	76 33	4 28	4.58	7.22	4.38
C(5)	15395 16	42506 39	19031 40	264 7	1352 39	1527 42	-361 68	251 27	-119 26	4.88	5.29	3.78
C(6)	17538 17	31005 42	31297 41	283 8	1522 43	1448 42	-258 72	84 29	-152 29	5.94	5.04	3.87
Cl(1)	18836 6	1367 13	39401 14	384 3	1777 13	2066 15	1024 24	-31 10	-87 10	4.29	9.29	6.24
Cl(3)	-544 6	22356 17	-1812 24	304 3	2179 19	3926 36	184 40	-809 17	-203 11	6.80	16.16	3.83
Cl(5)	16700 6	61556 11	26210 15	382 3	1351 11	2327 16	-653 21	371 11	-252 8	7.23	7.73	3.72

gram of Ahmed *et al.* (1966). The quantity minimized is $\sum w(|F_o| - |F_c|)^2$. The nine hydrogen atoms were located from a difference map which was computed when the *R* index was 12 %. Almost all hydrogen atoms were found at or near the expected positions. During the computation procedure their parameters were also refined, with starting thermal motion characteristics assumed to be the mean isotropic values of the adjacent carbon atoms. The initial weighting scheme was chosen arbitrarily, but that used in the final stages was

$$w = (11/|F_o|)^2 \sin^2 \theta \quad \text{for } |F_o| > 11 \\ w = (|F_o|/11)^2 \sin^2 \theta \quad \text{for } |F_o| \leq 11.$$

This scheme was devised to remove the strong dependence of $w(|F_o| - |F_c|)^2$ on $|F_o|$ and θ . The scattering factor curves were taken from *International Tables for X-ray Crystallography* (1962). Refinement appeared to be complete when the *R* index was 6.4 %. None of the shifts indicated in the final cycle exceeded $\frac{1}{3}$ of the corresponding e.s.d. Three additional L.S. cycles of the reflexions with $\sin \theta/\lambda < 0.4 \text{ \AA}^{-1}$ with fixed parameters of Cl and C atoms were used for the further refinement of the hydrogen atoms. The shifts of the H parameters were reasonable and the *R* index from all reflexions decreased to 6.3 %. The final atomic parameters with their estimated standard deviations are given in Tables 1 and 2. A three-dimensional final difference synthesis reveals some peaks in the vicinity of the Cl(3) atom and at the mid-points of the *c*-glide symmetrically related Cl(1) and Cl(1'), and Cl(5) and Cl(5') atoms. These peaks are as high as 0.35 e. \AA^{-3} . Negative peaks (-0.3 e. \AA^{-3}) appear in the neighbourhood of the Cl(1) and Cl(5) atoms. Elsewhere, the residual electron density stays between the limits $\pm 0.2 \text{ e. \AA}^{-3}$. The e.s.d. of electron density is about 0.1 e. \AA^{-3} . However, the agreement between the observed and calculated structure factors (Table 3) leaves no doubt as to the essential correctness of the proposed structure.

Table 2. Final parameters and e.s.d.'s of hydrogen atoms

Fractional coordinates are $\times 10^4$.

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>B</i> (\AA^2)
H(11)	1884	1346	1723	4.76
	32	65	74	1.58
H(21)	893	340	1276	2.61
	25	52	61	1.20
H(22)	660	1223	2568	1.65
	25	47	58	1.22
H(31)	972	2320	-212	2.14
	23	52	51	1.10
H(41)	709	4759	188	6.45
	37	69	92	1.71
H(42)	567	4189	1976	2.62
	24	55	62	1.22
H(51)	1767	4213	1218	2.79
	24	57	58	1.29
H(61)	2196	3291	3616	4.92
	31	66	72	1.51
H(62)	1517	3333	3922	3.94
	27	55	61	1.32

Description of the structure and discussion

A projection of the structure along the *c* axis is shown in Fig. 1. The shortest intermolecular contacts are: C(1')-Cl(1), 3.66; C(5')-H(62), 3.43; Cl(1)-Cl(5''), 3.70; Cl(1')-H(11), 2.83; H(42)-H(42''), 2.85 Å, where a one, two or three-prime superscript to the arabic numerals means an atom related by a *c* glide, a translation by one unit cell in the $-\mathbf{b}$ direction or a twofold rotation axis. All the intermolecular C-C distances are longer than 4 Å. The thermal motion ellipsoids (program written by Huml & Hlavatá, 1971) of the independent atoms are illustrated in Fig. 2. The molecular geometry of the non-hydrogen atoms is summarized in Fig. 3 and Tables 4 and 5. Valence bonds and angles involving hydrogen atoms are given

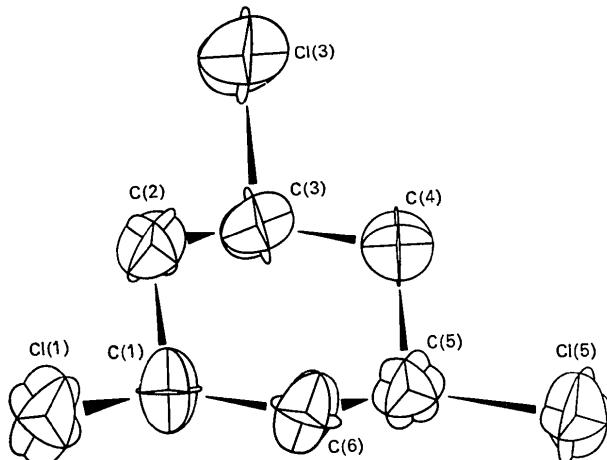


Fig. 2. Thermal-motion ellipsoids of 50 % probability.
Projection along c^* .

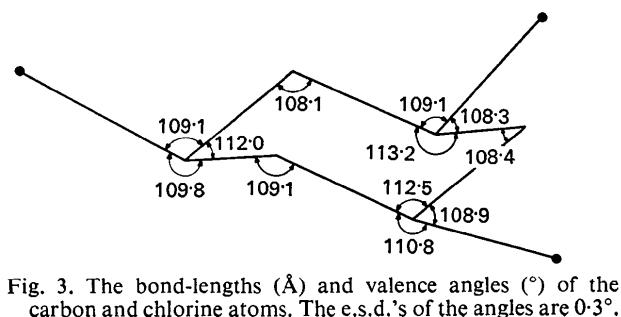
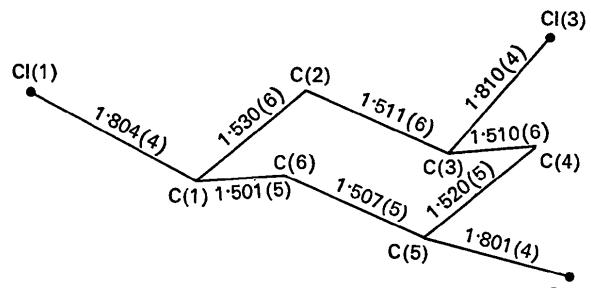


Fig. 3. The bond-lengths (\AA) and valence angles ($^\circ$) of the carbon and chlorine atoms. The e.s.d.'s of the angles are 0.3° .

Table 3. Observed and calculated structure factors

Unobserved reflexions are indicated by an asterisk ($|F_{\text{th}}|$ in place of $|F_0|$).

L	F_0	F_C	L	F_0	F_C	L	F_0	F_C	L	F_0	F_C	L	F_0	F_C	L	F_0	F_C	L	F_0	F_C	L	F_0	F_C				
H	O	K	O	0	76	57	2	146	136	6	399	368	-5	58	-62	6	125	-116	-2	13	-21	H	10	K	4		
2	651	-491	0	2	130	-45	9	167	157	7	256	-256	-8	95	-26	7	173	-179	-3	65	-58	H	12	K	3		
4	437	-435	-2	125	129	6	27	-23	8	158	-162	-1	16	16	2	10	55	-45	-5	35	-32	H	11	K	1		
10	44	-12	34	H	1	x	1	6	158	147	2	130	-130	-1	16	16	2	10	55	-45	-5	35	-32	H	12	K	7
4	2	K	0	H	1	x	1	6	158	147	-1	205	-279	0	61	-62	5	173	282	-1	67	-143	H	12	K	1	
0	266	-266	0	1	61	-767	2	186	80	6	491	-499	4	181	-176	6	125	-127	0	42	45	H	20	K	5		
2	189	176	2	1376	1398	-1	212	153	5	269	270	-1	167	-167	7	173	-179	-3	65	-58	0	113	117	H	6	K	6
4	244	-244	3	150	149	5	237	125	3	216	194	5	119	-119	8	173	-179	-3	65	-58	0	113	117	H	6	K	6
4	247	246	4	210	198	-5	237	125	3	216	194	5	119	-119	8	173	-179	-3	65	-58	0	113	117	H	6	K	6
1	274	-211	6	148	148	5	255	260	-3	30	35	7	61	-61	10	13	91	8	130	91	8	149	-143	H	12	K	7
1	1883	1883	7	278	274	-7	37	29	8	130	129	-1	10	81	7	13	91	8	149	-143	0	123	118	H	6	K	7
2	279	279	8	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	481	480	9	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	280	280	10	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	281	280	11	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	282	281	12	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	283	282	13	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	284	283	14	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	285	284	15	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	286	285	16	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	287	286	17	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	288	287	18	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	289	288	19	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	290	289	20	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	291	290	21	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	292	291	22	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	293	292	23	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	294	293	24	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	295	294	25	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	296	295	26	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	297	296	27	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	298	297	28	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	299	298	29	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	300	299	30	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	301	300	31	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	302	301	32	82	-67	-9	13	29	-1	10	81	-1	10	81	-1	10	81	-1	10	81	-1	10	81	H	6	K	7
4	303	302	33	82	-67																						

Table 3 (cont.)

L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	L	FO	FC	
16	42	K ₁	8	12	85	K ₂	23	139	-15	9	119	-10	1	139	19	1	139	25	3	129	7	-2	25	-41
5	69	-89	0	209	-173	0	-3	61	49	2	37	35	-1	71	-66	5	139	-59	1	139	19	1	139	19
5	76	89	0	246	-135	0	-2	22	76	4	16	K ₂	0	129	1	-1	139	-67	0	152	131	-2	119	127
-1	139	-137	2	168	98	0	12	128	K ₂	1	139	-10	1	139	25	1	139	-107	1	139	19	1	139	19
-1	305	305	0	299	76	0	12	128	K ₂	2	139	-10	1	139	-10	1	139	-107	1	139	19	1	139	19
-5	41	-89	0	119	-119	0	139	149	2	22	67	1	81	76	1	139	-107	1	139	19	1	139	19	
-5	161	281	0	119	122	1	59	59	2	22	297	1	67	-78	2	139	25	1	139	-107	1	139	19	
-5	57	35	0	12	128	1	2	139	-107	1	139	-10	1	139	-107	1	139	-107	1	139	19	1	139	19
16	62	K ₁	0	-2	139	-107	0	139	149	1	139	-107	1	139	25	1	139	-107	1	139	19	1	139	19
16	62	K ₁	0	-3	89	-102	0	139	149	1	139	-107	1	139	25	1	139	-107	1	139	19	1	139	19
0	162	137	0	-4	69	-85	1	139	149	1	139	-107	1	139	25	1	139	-107	1	139	19	1	139	19
1	57	63	0	-6	37	35	1	139	149	1	139	-107	1	139	25	1	139	-107	1	139	19	1	139	19
1	57	-29	0	10	48	0	1	139	149	1	139	-107	1	139	25	1	139	-107	1	139	19	1	139	19
1	129	-124	0	10	48	0	1	139	149	1	139	-107	1	139	25	1	139	-107	1	139	19	1	139	19
0	51	98	0	10	48	0	1	139	149	1	139	-107	1	139	25	1	139	-107	1	139	19	1	139	19
0	91	-110	0	100	85	0	1	139	149	1	139	-107	1	139	25	1	139	-107	1	139	19	1	139	19
0	105	-176	0	176	-76	0	0	139	-107	1	139	149	1	139	-107	1	139	19	1	139	19	1	139	19
0	172	-179	0	174	-190	0	2	139	149	1	139	-107	1	139	25	1	139	-107	1	139	19	1	139	19
-2	26	53	0	98	-100	0	1	139	149	1	139	-107	1	139	25	1	139	-107	1	139	19	1	139	19
1	75	53	0	129	25	0	1	139	149	1	139	-107	1	139	25	1	139	-107	1	139	19	1	139	19
1	48	-76	0	129	-143	0	1	139	149	1	139	-107	1	139	25	1	139	-107	1	139	19	1	139	19

In Tables 6 and 7. Calculation of final bond lengths and angles confirm that the atomic positions are consistent with reasonable expectations. The mean C-C bond length of 1.513 Å and the mean C-Cl bond length of 1.805 Å are in good agreement with the values published elsewhere for cyclohexane compounds (Vloten, Kruissink, Strijk & Bijvoet, 1950). Bond angles show deviations from an ideal tetrahedral angle of 109.5° within the limits +3.7, -1.3°. All C-H distances lie within the range 0.87–1.09 Å. The mean value of the C-H bond is 0.99 Å. Bond angles involving hydrogen atoms lie within the range 102.5–115.7°. The molecule in the crystal shows slight deviation from the threefold symmetry of the molecule in vacuum, e.g. the Cl-Cl distances differ by 0.037 Å; however, the corresponding e.s.d. is only 0.002 Å.

Table 4. Bond lengths and e.s.d.'s of non-hydrogen atoms and Cl-Cl distances

C(1)-C(2)	1.530 (6) Å
C(1)-C(6)	1.501 (5)
C(1)-Cl(1)	1.804 (4)
C(2)-C(3)	1.511 (6)
C(3)-C(4)	1.510 (6)
C(3)-Cl(3)	1.810 (4)
C(4)-C(5)	1.520 (5)
C(5)-C(6)	1.507 (5)
C(5)-Cl(5)	1.801 (4)
Cl(1)-Cl(3)	5.423 (2)
Cl(1)-Cl(5)	5.431 (2)
Cl(3)-Cl(5)	5.394 (2)

Table 5. Bond angles and e.s.d.'s of non-hydrogen atoms

C(2)-C(1)-C(6)	112.0 (3)°
C(2)-C(1)-Cl(1)	109.1 (3)
C(6)-C(1)-Cl(1)	109.8 (3)
C(1)-C(2)-C(3)	108.1 (3)
C(2)-C(3)-C(4)	113.2 (3)
C(2)-C(3)-Cl(3)	109.1 (3)
C(4)-C(3)-Cl(3)	108.3 (3)
C(3)-C(4)-C(5)	108.4 (3)
C(4)-C(5)-C(6)	112.5 (3)
C(4)-C(5)-Cl(5)	108.9 (3)
C(6)-C(5)-Cl(5)	110.8 (3)
C(1)-C(6)-C(5)	109.1 (3)

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Table 6. Bond lengths and e.s.d.'s of hydrogen atoms

C(1)-H(11)	0.97 (7) Å
C(2)-H(21)	0.95 (5)
C(2)-H(22)	1.04 (5)
C(3)-H(31)	0.96 (5)
C(4)-H(41)	1.09 (8)
C(4)-H(42)	1.06 (5)
C(5)-H(51)	0.87 (5)
C(6)-H(61)	1.01 (7)
C(6)-H(62)	0.99 (6)

Table 7. Bond angles and e.s.d.'s involving hydrogen atoms

C(2)-C(1)-H(11)	110 (4)°
C(6)-C(1)-H(11)	111 (4)
Cl(1)-C(1)-H(11)	106 (4)
C(1)-C(2)-H(21)	112 (3)
C(1)-C(2)-H(22)	116 (3)
C(3)-C(2)-H(21)	105 (3)
C(3)-C(2)-H(22)	112 (3)
H(21)-C(2)-H(22)	104 (4)
C(2)-C(3)-H(31)	110 (3)
C(4)-C(3)-H(31)	109 (3)
Cl(3)-C(3)-H(31)	107 (3)
C(3)-C(4)-H(41)	103 (4)
C(3)-C(4)-H(42)	105 (3)
C(5)-C(4)-H(41)	114 (4)
C(5)-C(4)-H(42)	113 (3)
H(41)-C(4)-H(42)	112 (5)
C(4)-C(5)-H(51)	111 (3)
C(6)-C(5)-H(51)	111 (3)
Cl(5)-C(5)-H(51)	103 (3)
C(1)-C(6)-H(61)	114 (4)
C(1)-C(6)-H(62)	113 (3)
C(5)-C(6)-H(61)	110 (4)
C(5)-C(6)-H(62)	105 (3)
H(61)-C(6)-H(62)	105 (5)

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References

- AHMED, F. R., HALL, S. R., PIPPY, M. E. & HUBER, C. P. (1966). N.R.C. Crystallographic Programs for the IBM/360 System, *World List of Crystallographic Computer Programs*, 2nd ed.
- HUML, K. & HLAVATÁ, D. (1971). *Czech. J. Phys. A* **21**, 419. *International Tables for X-ray Crystallography* (1962). Vol. III. Birmingham: Kynoch Press.
- KARLE, J. & KARLE, I. S. (1966). *Acta Cryst.* **21**, 849.
- OBEREIGNER, B., DOSKOČILOVÁ, D. & ŠTOKR, J. (1968). *Tetrahedron Letters*, **31**, 3511.
- VLOTEN, G. W. VAN, KRUISSINK, C. A., STRIJK, B. & BIJVOET, J. M. (1950). *Acta Cryst.* **3**, 139.