judged by the fact that the final R was 0·095 for the reflexions $hk0$ and $\bar{1}k3$ common to both individuals whereas R was 0·137 for the F-values derived for other $hk3$ reflexions, the overall R-factor for all reflexions being 0·097. Small scaling errors indicate that the volume of the smaller twin crystal was in fact slightly less than one-fifth of that of the main individual, so that the common reflexions (which tend to equivalence through twinning) have been slightly over-corrected.

APPENDIX II

Decamethonium bromide

The bromine atoms were located from Patterson projections, and after attempts to solve the structure from the projections alone had failed, the chains were located from a 3-D Fourier synthesis. The refinement was carried out in exactly the same way as for the hexamethonium compound.

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The Crystal Structure of Bicyclohexylidene, $C_{12}H_{20}$

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Bicyclohexylidene crystallizes in the triclinic space group $P\bar{1}$ with the unit-cell dimensions $a=5{\cdot}32$, $b=6{\cdot}25$, $c=8{\cdot}36$ Å, $\alpha=107{\cdot}2°$, $\beta=79{\cdot}1°$, $\gamma=105{\cdot}6°$. $D_x=1{\cdot}073$ g.cm^{-3}, $D_m=1{\cdot}066$ g.cm^{-3}, $Z=1$.

The phase problem was solved from the Patterson function. The final anisotropic parameters for the C atoms and positional parameters of the H atoms were obtained by three-dimensional least-squares refinement. For the calculation of interatomic distances the atomic coordinates were corrected for rigid-body thermal libration of the molecule.

The molecule consist of two cyclohexane rings in the chair conformation with a center of symmetry at the mid-point of the double bond linking them. The six carbon atoms contiguous with the double bond are planar. The whole molecule has a non-crystallographic mirror plane perpendicular to this plane and passing through the center of symmetry and the center of the two cyclohexane rings.

The length of the C=C double bond is 1·332 Å and the single bonds in the ring vary between 1·509 Å and 1·533 Å with e.s.d. 0·004 Å. The mean length of the ten independent C–H bonds is 1·01 Å, with individual deviations from the mean averaging 0·03 Å. The shortest distance between hydrogen atoms belonging to neighbouring carbon atoms of the molecule is 1·944 Å. The angles in the ring vary between 110·4° and 111·9° but conform to the mirror-plane symmetry of the molecule. The intermolecular atomic radii for hydrogen and carbon are 1·27 Å and 1·87 Å, respectively.

Introduction

The compound $C_{12}H_{20}$ was first prepared synthetically by Criegee, Vogel & Horger (1952) by zinc powder dehalogenization of 1,1′-dibromobicyclohexyl. The molecule was shown to consist of two cyclohexane rings linked by a double bond. The conformation and detailed stereochemistry of this molecule are of interest because of the non-bonding hydrogen atom interactions which might distort the cyclohexane rings from the customary strain-free chair conformation. In fact, Tömösközi (1959) has proposed that the boat form would be the more stable for this reason.

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Experimental

The crystals used were prepared by one of the authors (M. L.) by the method described by Criegee, Vogel & Horger (1952). They are colorless and generally grow as thin needles elongated in the direction of the a axis. More rarely, rhomboid crystals with a few well-developed planes and sharp edges were obtained which were 1–2 mm in the c direction and smaller in the b direction and from which sections were cut for the structure analysis. The crystals (m. p. 55°C) sublime rather quickly at room temperature. It was therefore easy to get almost cylindrically shaped crystals with a cross-section of about 0·1 mm. These crystals were protected against further sublimation by covering them with a thin skin of polyvinyl acetate.

The unit-cell dimensions, determined from Stoe precession photographs around the a and c axes with Cu $K\alpha$ radiation and redetermined by single-crystal counter diffractometry are:

$a = 5{\cdot}32 \pm 0{\cdot}01$ Å $\quad \alpha = 107{\cdot}2 \pm 0{\cdot}1°$
$b = 6{\cdot}25 \pm 0{\cdot}01$ $\quad \beta = 79{\cdot}1 \pm 0{\cdot}1$
$c = 8{\cdot}36 \pm 0{\cdot}01$ $\quad \gamma = 105{\cdot}6 \pm 0{\cdot}1$

The presence of a center of symmetry was deduced from the intensity statistics (N_z function) of the three-dimensional reflections and the space group is therefore $P\bar{1}$, with $Z = 1$, $D_x = 1{\cdot}073$ g.cm^{-3} and D_m (by flotation) $= 1{\cdot}066$ g.cm^{-3}, molecular symmetry $\bar{1}$. The linear absorption coefficient μ for Cu $K\alpha$ radiation is 4·36 cm^{-1}. The diameter of the crystals was never greater than 0·1 mm for which $\mu . R \sim 0{\cdot}04$, and therefore absorption corrections were not made.

The intensities of the reflections, recorded with a Stoe integrating Weissenberg camera with the multiple-film technique, were measured with a Zeiss fast photometer. For the very weak reflections the photometry was not satisfactory and a visual comparison with a standard intensity scale was used. The intensities collected from different films were brought to a common scale by multiple-film scaling, and by interlayer scaling after applying the Lorentz-polarization factors. The absolute scale and approximate isotropic temperature factor were determined by a Wilson plot from the three-dimensional data. Of the 1282 reflections within the Cu $K\alpha$ sphere, the intensities of 858 were recorded, of which 129 were unobserved with $F_o = 0$.

Determination of structure

The first attempt to solve the phase problem was by the direct method based on the three-dimensional sign-correlation through the Sayre equation (Beurskens, 1964). This method gave a choice of 16 equally probable sign combinations, one of which proved later to be correct. The three-dimensional Patterson function was therefore computed and this showed eight well-defined vector peaks with the relative heights given in Table 1. Knowing that the molecule has two cyclohexane rings and a center of symmetry, with one molecule in the unit cell, the 1–6 vectors in Table 1 could be identified as the interatomic vectors of the cyclohexane rings (three sides and three diagonals), all of which have the multiplicity of four. With aid of these six vectors, one ring of the molecule could be constructed with its correct shape and orientation in the unit cell. Vector 7 corresponds to the vector between the mid-points of the cyclohexane rings of the molecule and is formed from the corresponding atoms in the two cyclohexane rings taken in pairs. Its weight is approximately 6:4 relative to vectors 1 to 6, which agrees well with the ratio of the corresponding vector peak heights. Vector 8 cannot be explained as a resolved interatomic vector. It coincides with the vector belonging to atoms 2 and 4′ and their symmetrical pair (Fig. 2), but its weight shows that it must include other superimposed vectors. This interpretation of the Patterson synthesis fixed both the relative position of the cyclohexane rings and the position of the whole molecule in the unit cell. The corresponding approximate atomic coordinates of the carbon atoms with isotropic temperature factors $B = 5{\cdot}0$ Å^{-2} gave a structure-factor agreement for all observed reflections of $R = 0{\cdot}41$. A three-dimensional Fourier synthesis, using 528 reflections for which $F_c > 0{\cdot}7$, then gave new carbon coordinates, which with an isotropic temperature factor $B = 4{\cdot}0$ Å^{-2} resulted in an R value of 0·30.

Table 1. *The coordinates and relative heights of the peaks in the three-dimensional Patterson function*

	Coordinates			Relative
Peak	x	y	z	height
1	−0·06	−0·12	0·14	62
2	0·12	0·24	0·06	66
3	0·16	0·335	−0·07	69
4	0·21	−0·11	0·22	62
5	0·25	0·05	0·09	57
6	0·35	0·30	0·15	67
7	0·40	0·27	0·43	103
8	0·50	0·49	0·49	52

For eleven strong reflections in the region of small $\sin\theta$ values, there was evidence of strong extinction and for these the $|F_o|$ values were replaced by the $|F_c|$ in the subsequent three-dimensional differential Fourier synthesis refinement. After three cycles with isotropic and four cycles with anisotropic temperature factors, the R values were 0·16 and 0·17 for the observed and all reflections, respectively.

The hydrogen atomic coordinates were generated geometrically from the positions of the carbon atoms, and with the same anisotropic temperature factors as the corresponding carbon atoms, their inclusion reduced the R values to 0·128 and 0·144 respectively.

The hydrogen atoms were also deduced from a three-dimensional difference Fourier synthesis, the projection of which is shown in Fig. 1, together with the two-dimensional projection of the electron density distribution of the structure. All reflections with $\sin\theta > 0{\cdot}7$

and for which there was high extinction were omitted. Well-defined maxima of approximately equal height at positions close to those predicted for the hydrogen atoms were obtained, together with two smaller spurious maxima of about one third the magnitude. The hydrogen positions deduced geometrically and observed on the difference Fourier map differed by less than 0·1 Å.

The final refinement of the parameters was made with the full-matrix anisotropic least-squares IBM 7090 program of Busing & Levy (1962), minimizing the function $\Phi=\sum_h w_h(F_{oh}-G|F_c|_h)^2$, where G is the scaling factor. The weighting factor of Cruickshank (1961*a*), $w=1/(a+b\,.\,F+c\,.\,F^2)$, was used with $a=0{\cdot}25$, $b=1$ and $c=0{\cdot}05$. The refinement of the carbon and hydrogen atoms was carried out in two separate stages. First the carbon coordinates and their temperature factors were refined using only observed reflections from which those of $\sin\theta<0{\cdot}333$ and $F_c>10{\cdot}0$ were omitted because of their high extinction. In the second step only the hydrogen coordinates were refined, omitting from the reflections also those with $\sin\theta>0{\cdot}7$. For both stages, the refinement converged in the first cycle, so that in the second cycle the shifts of the parameters were less than the estimated standard deviations. The final atomic parameters together with their estimated standard deviations are listed in Tables 2 and 3.

The structure factors calculated from the parameters of Table 2 and 3 are given in Table 4. The atomic scattering factors of carbon and hydrogen atoms were taken from *International Tables for X-ray Crystallography* (1962). For the hydrogen atoms the temperature

Table 2. *Final positional parameters of carbon and hydrogen atoms and (in parentheses) their estimated standard deviations*

For carbon atoms are also given (in parentheses below) the parameters corrected for libration of the molecule as a rigid body.

In the notation of hydrogen atoms the first digit refers to the corresponding carbon atoms. The two hydrogen atoms attached to a carbon atom are distinguished by the second digit.

Atom	*x*	*y*	*z*
C(1)	0·0309 (6)	0·0498 (5)	0·0779 (4)
	(0·0309)	(0·0499)	(0·0781)
C(2)	0·1438 (7)	0·3019 (5)	0·1463 (5)
	(0·1439)	(0·3030)	(0·1465)
C(3)	0·4002 (8)	0·3451 (6)	0·2194 (5)
	(0·4008)	(0·3460)	(0·2195)
C(4)	0·3643 (8)	0·2183 (7)	0·3539 (4)
	(0·3645)	(0·2184)	(0·3543)
C(5)	0·2563 (8)	−0·0365 (6)	0·2845 (4)
	(0·2564)	(−0·0372)	(0·2849)
C(6)	−0·0013 (7)	−0·0779 (6)	0·2129 (4)
	(−0·0018)	(−0·0786)	(0·2129)
H(21)	0·178 (10)	0·384 (9)	0·059 (6)
H(22)	−0·001 (10)	0·362 (9)	0·249 (6)
H(31)	0·536 (10)	0·273 (9)	0·132 (6)
H(32)	0·470 (11)	0·512 (9)	0·272 (7)
H(41)	0·540 (10)	0·251 (9)	0·395 (7)
H(42)	0·239 (10)	0·282 (9)	0·447 (6)
H(51)	0·394 (10)	−0·102 (9)	0·190 (6)
H(52)	0·215 (11)	−0·122 (9)	0·369 (7)
H(61)	−0·066 (10)	−0·240 (9)	0·163 (6)
H(62)	−0·136 (10)	−0·010 (9)	0·307 (6)

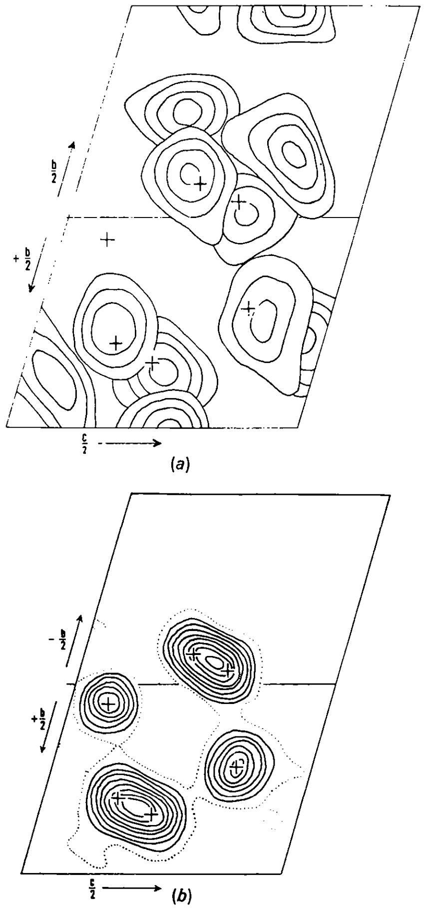

Fig. 1. (*a*) The projection of the hydrogen electron density peaks of the three-dimensional F_o-F_c synthesis on the plane (100). Magnified 2·5 as compared with (*b*). Contours are at arbitrary intervals. The contours of zero level are omitted. The crosses (+) are the carbon positions according to the data of Table 2. (*b*) The two-dimensional electron density projection on (100). Contours are at arbitrary intervals. The dotted contour is the zero level. The crosses (+) are the carbon positions according to the data of Table 2.

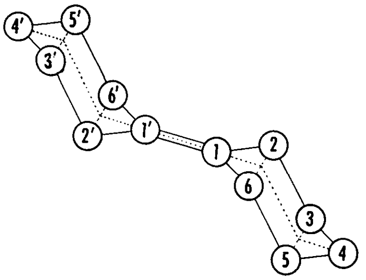

Fig. 2. The bicyclohexylidene molecule in schematic representation by only the carbon atoms. The dotted broken line shows the trace of the non-crystallographic mirror plane of the molecule and also the inclinations of the atomic planes of the chair conformation.

factors of the corresponding adjacent carbon atoms were used. The reliability index $R(hkl)$ for the observed reflections, without the eleven reflections of high extinction, is 0·116.

The geometry of the molecule

In the molecule of bicyclohexylidene the six carbon atoms associated with the central ethylene bond (6211′2′6′) are planar (Fig. 2). The equation for the best plane calculated for these six atoms is

$$0{\cdot}9496X - 0{\cdot}3288Y - 0{\cdot}0854Z = 0$$

and the deviations from this plane for the asymmetric atoms forming the plane are 0·0004 Å for C(6) and C(2), and 0·0020 Å for C(1). The molecule also has a non-crystallographic mirror plane normal to the plane described above and passing through the carbon atoms (411′4′) and the center of symmetry of the molecule (along the dotted line of Fig. 2). Since they are related in pairs by the center of symmetry, these four atoms are exactly in a plane, the equation of which is

$$0{\cdot}0384X + 0{\cdot}9201Y + 0{\cdot}4490Z = 0\,.$$

The other atoms of the molecule deviate from this plane by mean values of 0·023 Å for carbon and 0·048 Å for hydrogen. The symmetry of the molecule including the hydrogen atoms is therefore $2/m$, within the experimental error.

The molecule maintains the chair conformation associated with the free cyclohexane ring, with the four carbon atoms (2356) of the ring planar within a max-

Table 3. *Thermal parameters of the carbon atoms with their estimated standard deviations in parentheses*

The b_{ij} are given here as defined by: $T = \exp\{-10^{-4}(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + 2b_{12}hk + 2b_{13}hl + 2b_{33}kl)\}$

Atom	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
C(1)	295 (14)	185 (8)	128 (7)	4 (8)	−62 (8)	55 (6)
C(2)	386 (16)	218 (10)	169 (7)	13 (9)	−104 (9)	42 (7)
C(3)	408 (18)	266 (11)	158 (7)	−17 (10)	−93 (9)	28 (7)
C(4)	444 (18)	344 (13)	140 (7)	45 (11)	−116 (9)	43 (7)
C(5)	460 (19)	302 (12)	148 (7)	69 (11)	−94 (10)	75 (7)
C(6)	409 (17)	282 (11)	134 (6)	−1 (10)	−70 (8)	90 (7)

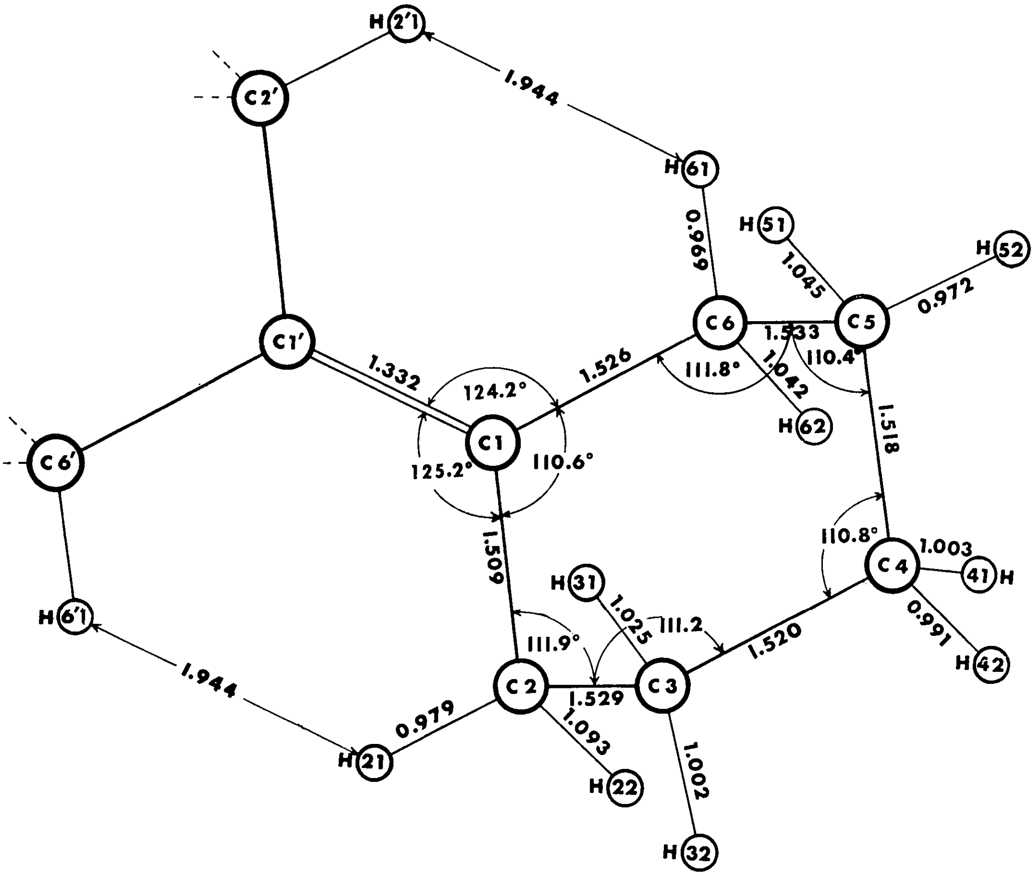

Fig. 3. The most significant atomic distances and bond angles in the molecule of bicyclohexylidene, given in the orthogonal projection on the plane (100).

Table 4. *Observed and calculated structure factors*

* Unobserved or omitted from final calculations owing to extinction.

H	K	L	F_o	F_c
0	0	1	8.7*	11.4
0	0	2	15.6*	-27.2
0	0	3	4.1	-3.4
0	0	4	2.0	-1.3
0	0	5	0.4	0.5
0	0	6	1.2	-1.2
0	0	7	3.9	-3.8
0	0	8	0.7	-0.5
0	0	9	0.6	0.5
0	1	0	11.9*	21.8
0	1	1	9.6*	-15.1
0	1	2	6.3	-6.9
0	1	3	1.9	-1.2
0	1	4	0.8	1.0
0	1	5	0.7	0.4
0	1	6	3.5	-3.4
0	1	7	1.1	-1.3
0	1	8	0.0*	-0.4
0	1	9	2.2	-2.0
0	-1	1	10.8*	22.0
0	-1	2	3.0	-3.3
0	-1	3	12.4	13.9
0	-1	4	2.0	1.5
0	-1	5	7.8	-8.0
0	-1	6	1.9	-1.5
0	-1	7	2.3	-2.3
0	-1	8	3.6	-3.2
0	-1	9	1.2	1.0
0	-1	10	1.3	1.2
0	2	0	0.0*	-0.1
0	2	1	13.4*	20.1
0	2	2	4.4	4.1
0	2	3	5.8	-6.0
0	2	4	0.0*	-0.3
0	2	5	2.5	-2.1
0	2	6	3.4	-2.9
0	2	7	0.5	0.0
0	2	8	1.7	-1.7
0	2	9	0.3	-0.7
0	-2	1	10.6	-13.7
0	-2	2	9.3	-9.5
0	-2	3	14.2	16.5
0	-2	4	15.3	18.6
0	-2	5	6.6	-6.5
0	-2	6	7.3	-7.3
0	-2	7	0.0*	-0.5
0	-2	8	3.4	-2.6
0	-2	9	1.0	-1.0
0	-2	10	0.3	0.6
0	3	0	10.9	13.6
0	3	1	11.8	14.8
0	3	2	7.5	-8.1
0	3	3	6.9	-7.2
0	3	4	0.0*	-0.5
0	3	5	3.8	-3.3
0	3	6	1.3	-1.3
0	3	7	1.1	0.8
0	-3	1	3.6	-3.5
0	-3	2	1.1	-1.4
0	-3	3	0.0*	0.4
0	-3	4	6.9	6.8
0	-3	5	2.4	1.9
0	-3	6	3.4	-3.2
0	-3	7	0.0*	-0.0
0	-3	8	0.9	-0.9
0	-3	9	1.8	-1.7
0	-3	10	0.0*	-0.3
0	4	0	2.9	2.5
0	4	1	1.0	-0.9
0	4	2	5.2	-5.2
0	4	3	1.1	-1.1
0	4	4	2.0	-1.8
0	4	5	2.7	-2.3
0	4	6	1.1	1.0
0	-4	1	1.9	-1.4
0	-4	2	9.1	9.2
0	-4	3	8.6	9.9
0	-4	4	4.0	-3.4
0	-4	5	4.7	-4.1
0	-4	6	0.0*	-0.5
0	-4	7	1.8	1.1
0	-4	8	0.0*	0.4
0	-4	9	1.5	-1.5
0	5	0	2.3	-2.0
0	5	1	0.8	0.4
0	5	2	1.5	0.7
0	5	3	1.3	-1.2
0	5	4	2.3	-2.0
0	-5	1	6.2	-5.9
0	-5	2	1.8	1.6
0	-5	3	10.1	12.3
0	-5	4	4.2	3.5
0	-5	5	3.5	-3.4
0	6	0	0.0*	-0.2
0	6	1	0.0*	0.2
0	6	2	0.4	-0.3
0	6	3	2.1	-1.8
0	-6	1	0.5	-0.5
0	-6	2	0.9	-0.8
0	-6	3	2.2	2.1
0	-6	4	2.2	2.0
0	-6	5	0.5	0.4
0	-6	6	3.2	3.1
0	-6	7	1.8	1.3
0	-6	8	2.1	-2.0
0	7	0	1.9	-2.0
0	7	1	0.0*	-0.9
0	7	2	0.4	-1.0
0	-7	1	1.1	0.7
0	-7	2	3.7	3.5
0	-7	3	1.4	1.1
0	-7	4	1.9	-1.6
0	-7	5	1.6	-0.9
0	-7	6	2.8	2.3
1	0	0	7.3*	10.3
1	0	1	9.1*	-11.3
1	0	2	4.3	4.2
1	0	3	6.3	-6.2
1	0	4	3.7	-3.0
1	0	5	1.4	-0.8
1	0	6	1.7	-1.6
1	0	7	0.4	-0.3
1	0	8	2.3	-2.1
1	0	9	1.8	-1.5
1	0	10	1.2	1.2
1	1	0	0.9	0.9
1	1	1	8.8	10.0
1	1	2	2.5	-2.4
1	1	3	3.5	-3.2
1	1	4	0.3	-0.3
1	1	5	1.9	-1.4
1	1	6	0.4	0.7
1	1	7	2.3	-2.4
1	1	8	5.5	-5.6
1	1	9	0.6	0.8
1	-1	1	5.2	-5.0
1	-1	2	5.7	5.8
1	-1	3	1.6	1.6
1	-1	4	4.2	-3.9
1	-1	5	2.1	1.9
1	-1	6	4.8	-4.7
1	-1	7	6.2	-6.2
1	-1	8	0.5	0.7
1	-1	9	2.9	2.5
1	-1	10	0.2	0.4
1	2	0	16.0	18.4
1	2	1	6.5	6.7
1	2	2	5.9	-6.1
1	2	3	2.0	1.5
1	2	4	3.6	-3.3
1	2	5	5.0	-5.2
1	2	6	0.0*	-0.4
1	2	7	2.1	-1.9
1	2	8	1.5	-1.1
1	2	9	2.6	2.6
1	-2	1	2.9	-2.3
1	-2	2	8.1	8.4
1	-2	3	12.7	15.8
1	-2	4	4.6	-3.8
1	-2	5	2.6	-1.9
1	-2	6	0.0*	0.1
1	-2	7	7.5	-7.8
1	-2	8	4.0	-3.6
1	-2	9	2.7	2.6
1	-2	10	0.3	0.5
1	3	0	9.0	9.4
1	3	1	4.7	-4.2
1	3	2	3.1	-2.6
1	3	3	0.5	-0.7
1	3	4	7.3	-7.9
1	3	5	3.7	-3.5
1	3	6	3.3	3.0
1	3	7	1.2	1.4
1	3	8	0.2	-0.0
1	-3	1	2.8	-2.7
1	-3	2	1.3	-0.9
1	-3	3	9.5	11.1
1	-3	4	6.9	6.0
1	-3	5	5.0	-4.6
1	-3	6	0.3	-0.3
1	-3	7	2.3	-1.9
1	-3	8	3.1	-2.7
1	-3	9	0.4	0.3
1	-3	10	1.8	-1.9
1	4	0	0.3	-0.2
1	4	1	1.8	-2.1
1	4	2	0.0*	-0.3
1	4	3	3.3	-3.1
1	4	4	3.4	-3.1
1	4	5	1.3	1.2
1	4	6	1.6	1.3
1	4	7	0.2	-0.2
1	-4	1	4.1	4.0
1	-4	2	5.6	5.3
1	-4	3	0.0*	-0.2
1	-4	4	2.4	2.1
1	-4	5	0.3	-0.3
1	-4	6	1.5	-1.6
1	-4	7	0.0*	-0.4
1	-4	8	0.0*	-0.2
1	-4	9	0.0*	0.5
1	-4	10	1.5	-1.6
1	5	0	0.0*	-1.3
1	5	1	0.0*	-0.9
1	5	2	0.0*	-0.5
1	5	3	0.0*	-0.2
1	5	4	0.0*	0.1
1	5	5	2.0	-2.0
1	-5	1	0.0*	0.3
1	-5	2	9.9	10.1
1	-5	3	5.0	4.3
1	-5	4	1.7	-1.5
1	-5	5	0.0*	0.7
1	-5	6	1.3	-1.3
1	-5	7	1.4	-1.3
1	-5	8	0.0*	-0.0
1	-5	9	0.7	-1.0
1	6	0	1.1	-1.2
1	6	1	0.4	-0.3
1	6	2	0.7	-0.7
1	6	3	0.4	-0.5
1	-6	1	3.8	-3.5
1	-6	2	3.7	3.0
1	-6	3	5.1	4.9
1	-6	4	0.5	0.7
1	-6	5	2.3	1.8
1	-6	6	1.4	1.1
1	-6	7	1.4	-1.1
1	-6	8	0.3	0.0
1	-7	0	0.0*	0.1
1	-7	1	1.7	1.3
1	-7	2	0.4	0.6
1	-7	3	0.0*	0.0
1	-7	4	0.3	-0.3
1	-7	5	1.4	1.5
1	-7	6	2.4	2.5
-1	0	1	19.4*	42.4
-1	0	2	2.8	2.5
-1	0	3	7.8	-7.9
-1	0	4	1.0	1.0
-1	0	5	3.2	-3.0
1	-1	0	19.3*	35.4
-1	1	1	14.0*	23.1
-1	1	2	13.6	-17.1
-1	-1	1	1.6	1.5
-1	-1	2	0.9	-0.5
-1	-1	3	2.9	2.9
-1	-1	4	13.5	13.5
-1	-1	5	0.4	0.7
-1	-1	6	9.0	-10.4
-1	2	0	2.4	2.3
-1	2	1	1.1	-0.9
-1	2	2	1.8	1.6
-1	-2	1	0.8	-0.6
-1	-2	2	4.6	-4.3
-1	-2	3	2.4	-1.8
-1	-2	4	7.0	7.2
-1	-2	5	7.5	7.5
-1	-2	6	4.4	-4.6
-1	-2	7	4.6	-4.5
-1	3	0	2.2	-2.0
-1	3	1	10.0	10.5
-1	3	2	9.4	9.7
-1	3	3	6.7	-6.7
-1	3	1	5.1	4.6
-1	-3	2	2.0	1.5
-1	-3	3	6.5	6.5
-1	-3	4	0.0*	0.4
-1	-3	5	2.3	-2.2
-1	-3	6	0.9	-1.1
-1	-3	7	0.0*	0.4
-1	-3	8	1.2	1.4
-1	4	0	1.3	0.8
-1	4	1	6.7	6.2
-1	4	2	1.8	-1.5
-1	4	3	8.2	-8.5
-1	-4	1	0.3	0.0
-1	-4	2	1.2	-0.9
-1	-4	3	9.3	9.4
-1	-4	4	7.5	7.7
-1	-4	5	4.3	-4.1
-1	-4	6	4.4	-4.1
-1	-4	7	0.0*	0.3
-1	-4	8	1.2	1.1
-1	5	0	4.5	-4.6
-1	5	1	1.3	-1.2
-1	5	2	0.7	-1.0
-1	-5	1	0.0*	0.1
-1	-5	2	2.0	-1.3
-1	-5	3	2.4	2.1
-1	-5	4	6.2	6.3
-1	-5	5	0.9	1.1
-1	-5	6	0.0*	-0.8
-1	-5	7	0.5	1.1
-1	-5	8	0.6	-1.0
-1	6	0	2.6	-2.7
-1	6	1	0.3	0.6
-1	6	2	1.8	1.8
-1	-6	1	0.0*	-0.5
-1	-6	2	2.7	2.1
-1	-6	3	1.6	1.4
-1	-6	4	0.0*	0.2
-1	-6	5	0.0*	-0.4
-1	-6	6	0.8	1.0
-1	-6	7	3.4	3.3
-1	-7	0	0.0*	-0.3
-1	-7	1	1.2	-0.9
-1	-7	2	1.2	1.1
-1	-7	3	2.4	2.7
-1	-7	4	0.0*	0.6
-1	-7	5	0.1	-0.7
2	0	0	6.6	7.5
2	0	1	17.3	15.8
2	0	2	10.2	-11.0
2	0	3	12.2	-14.5
2	0	4	0.0*	0.5
2	0	5	4.3	4.6
2	0	6	2.2	1.8
2	0	7	5.4	-6.0
2	0	8	4.5	-4.7
2	0	9	2.4	2.4
2	0	10	1.2	1.3
2	1	0	4.3	4.7
2	1	1	0.0*	0.2
2	1	2	0.0*	0.0
2	1	3	1.8	1.8
2	1	4	2.4	-2.3
2	1	5	0.0*	0.4
2	1	6	2.3	-2.5
2	1	7	6.2	-6.9
2	1	8	0.0*	0.2
2	1	9	4.7	3.9
2	-1	1	13.1	17.0
2	-1	2	8.0	7.6
2	-1	3	13.0	-12.8
2	-1	4	2.1	-1.8
2	-1	5	1.7	-1.6
2	-1	6	3.5	-3.9
2	-1	7	0.0*	-0.3
2	-1	8	0.4	-0.1
2	-1	9	0.3	0.2
2	-1	10	0.0*	-0.3
2	2	0	[illegible]	[illegible]
2	2	1	2.6	2.9
2	2	2	8.9	8.8
2	2	3	5.5	-5.4
2	2	4	10.4	-11.7
2	2	5	1.2	-1.0
2	2	6	1.4	0.9
2	2	7	0.3	-0.5
2	2	8	1.5	1.2
2	-2	1	2.8	2.6
2	-2	2	6.0	5.8
2	-2	3	0.0*	0.0
2	-2	4	7.9	7.2
2	-2	5	3.7	3.3
2	-2	6	11.7	-12.7
2	-2	7	7.4	-7.0
2	-2	8	3.9	3.6
2	-2	9	2.6	2.1
2	-2	10	0.0*	-0.5
2	3	0	0.4	-0.4
2	3	1	2.5	1.8
2	3	2	0.3	-0.2
2	3	3	9.8	-10.0
2	3	4	5.4	-4.6
2	3	5	4.3	3.8
2	3	6	2.6	2.0
2	3	7	0.0*	-0.5
2	-3	1	1.3	0.9
2	-3	2	2.1	1.7
2	-3	3	2.5	1.9
2	-3	4	2.5	2.3
2	-3	5	7.1	6.6
2	-3	6	4.0	-3.7
2	-3	7	8.5	-8.3
2	-3	8	0.0*	0.0
2	-3	9	0.0*	0.0
2	-3	10	2.7	-1.9
2	4	0	4.3	-4.5
2	4	1	2.1	-2.0
2	4	2	1.4	-0.8
2	4	3	0.9	-0.3
2	4	4	0.3	0.2
2	4	5	1.6	-1.3
2	4	6	0.0*	-0.7
2	-4	1	3.3	3.0
2	-4	2	0.6	0.6
2	-4	3	4.2	3.9
2	-4	4	1.8	1.4
2	-4	5	2.4	-2.1
2	-4	6	0.5	-0.9
2	-4	8	2.1	1.6
2	-4	9	1.7	-1.2
2	-4	10	4.9	-4.7
2	5	0	2.9	-3.1
2	5	1	0.0*	0.3
2	5	2	2.4	1.9
2	5	3	0.2	0.2
2	5	4	5.4	-4.0
2	5	5	3.0	-2.8
2	-5	1	4.8	4.8
2	-5	2	2.6	2.1
2	-5	3	2.7	2.3
2	-5	4	4.6	4.7
2	-5	5	2.4	-2.3
2	-5	6	4.8	-4.7
2	-5	7	0.0*	-0.1
2	-5	8	2.0	1.7
2	-5	9	0.0*	0.0
2	-5	10	2.3	-2.6
2	6	0	0.0*	0.3
2	6	1	2.1	-1.5
2	6	2	1.7	-1.3
2	-6	1	2.1	1.9
2	-6	2	4.0	3.6
2	-6	3	1.8	1.2
2	-6	4	3.5	3.4
2	-6	5	2.0	1.7
2	-6	6	2.2	-2.2
2	-6	7	0.0*	-0.1
2	-6	8	0.5	-0.4
2	-6	9	2.4	-2.6
2	-7	1	0.9	-0.5
2	-7	2	1.4	1.2
2	-7	3	0.8	0.9
2	-7	4	1.1	1.0
2	-7	5	1.5	1.5
2	-7	6	0.2	0.4
2	-7	7	2.0	1.5
-2	0	1	2.7	2.4
-2	0	2	8.6	8.0
-2	0	3	0.9	0.6
-2	0	4	0.0*	0.7
-2	0	5	3.0	2.2
-2	1	0	1.4	1.2
-2	1	1	11.6	13.8
-2	1	2	10.6	9.4
-2	-1	1	1.4	1.1
-2	-1	2	2.3	1.7
-2	-1	3	1.5	0.8
-2	-1	4	2.4	2.1
-2	-1	5	6.5	6.5
-2	-1	6	0.0*	-1.0
-2	2	0	3.5	3.3
-2	2	1	8.0	7.6
-2	2	2	2.6	-2.4
-2	-2	1	3.3	2.5
-2	-2	2	1.0	0.9
-2	-2	3	4.0	3.5
-2	-2	4	1.7	1.4
-2	-2	5	0.0*	0.5
-2	-2	6	1.2	0.7
-2	-2	7	1.8	-1.7
-2	3	0	2.3	1.7
-2	3	1	0.2	0.2
[illegible]	[illegible]	2	3.3	[illegible]
-2	-3	1	4.7	4.2
-2	-3	2	2.2	1.7
-2	-3	3	3.0	2.6
-2	-3	4	5.8	5.1
-2	-3	5	0.0*	-0.6
-2	-3	6	4.4	-3.8
-2	-3	7	0.9	-0.9
-2	4	0	2.1	2.0
-2	4	1	1.6	1.3
-2	4	2	5.3	5.2
-2	-4	1	1.4	1.3
-2	-4	2	4.5	4.2
-2	-4	3	1.7	1.3
-2	-4	4	4.9	4.0
-2	-4	5	3.4	2.9
-2	-4	6	2.6	-2.2
-2	-4	7	1.8	-1.3
-2	5	0	0.0*	-0.0
-2	5	1	1.6	-1.4
-2	5	2	0.0*	-0.2
-2	-5	1	2.9	-2.5
-2	-5	2	2.7	2.4
-2	-5	3	2.6	2.1
-2	-5	4	1.3	1.1
-2	-5	5	1.8	1.7
-2	-5	6	0.0*	0.3
-2	-5	7	1.2	0.9
-2	6	0	3.2	-3.3
-2	6	1	2.2	-2.3
-2	6	2	0.3	0.3
-2	-6	1	0.3	0.3
-2	-6	2	0.0*	0.3
-2	-6	3	2.2	1.3
-2	-6	4	0.4	0.7
-2	7	0	0.4	-0.8
-2	7	1	0.2	-0.1
3	0	0	6.2	6.8
3	0	1	3.0	-3.0
3	0	2	7.9	-7.5
3	0	3	1.0	0.5
3	0	4	3.8	4.1
3	0	5	2.8	2.8
3	0	6	5.4	-6.6
3	0	7	5.4	-6.2
3	0	8	2.1	2.1
3	0	9	1.1	1.0
3	1	0	0.8	0.6
3	1	1	7.5	7.4
3	1	2	5.9	6.2
3	1	3	6.4	-6.6
3	1	4	4.4	-4.2
3	1	5	0.6	-0.8
3	1	6	4.3	-4.6
3	1	7	0.0*	-0.2
3	1	8	1.9	1.8
3	1	9	0.0*	-0.4
3	-1	1	9.2	9.5
3	-1	2	13.9	-14.2
3	-1	3	6.4	-7.8
3	-1	4	2.3	2.5
3	-1	5	1.8	1.9
3	-1	6	0.0*	-0.3
3	-1	7	4.1	-4.6
3	-1	8	1.3	-1.2
3	-1	9	1.2	1.1
3	-1	10	0.5	-0.7
3	2	0	3.8	3.8
3	2	1	8.6	9.1
3	2	2	4.4	-4.2
3	2	3	11.7	-13.6
3	2	4	2.0	-1.5
3	2	5	2.7	2.4
3	2	6	0.0*	-0.0
3	2	7	0.0*	0.2
3	2	8	0.0*	-0.6
3	-2	1	7.6	9.0
3	-2	2	0.6	0.3
3	-2	3	1.3	1.1
3	-2	4	3.6	3.5
3	-2	5	7.6	-8.5
3	-2	6	5.8	-6.0
3	-2	7	2.0	1.7
3	-2	8	0.6	0.6
3	-2	9	0.3	0.1
3	3	0	1.7	-1.6
3	3	1	0.0*	-0.3
3	3	2	5.0	-5.1
3	3	3	2.6	-2.4
3	3	4	1.6	1.4
3	3	5	0.0*	-0.2
3	3	6	0.2	-0.1
3	-3	1	0.4	-0.5
3	-3	2	1.6	-1.2
3	-3	3	5.6	4.6
3	-3	4	11.1	11.0
3	-3	5	2.7	-2.4
3	-3	6	11.0	-12.4
3	-3	7	2.1	-1.6
3	-3	8	1.6	1.6
3	-3	9	1.0	-1.0
3	-3	10	0.2	0.3
3	4	0	3.2	-3.3
3	4	1	1.7	1.3
3	4	2	3.0	2.4
3	4	3	0.0*	-0.6
3	4	4	3.8	-4.0
3	4	5	1.5	-1.2
3	-4	1	0.3	0.2
3	-4	2	0.0*	-0.1
3	-4	3	1.3	1.0
3	-4	4	3.3	2.7
3	-4	5	2.6	2.2
3	-4	6	3.6	-3.2

Table 4 (*cont.*)

H	K	L	F_o	F_c	H	K	L	F_o	F_c	H	K	L	F_o	F_c	H	K	L	F_o	F_c	H	K	L	F_o	F_c
3	-4	7	0.9	-1.0	-3	3	1	3.2	3.0	4	-1	9	1.5	-1.3	-4	1	0	4.9	5.7	5	0	0	3.6	3.5
3	-4	8	0.0*	0.0	-3	3	2	0.0*	0.3	4	2	0	4.1	3.6	-4	1	1	7.1	7.9	5	1	0	2.7	3.1
3	-4	9	3.5	-3.9	-3	-3	1	0.3	-0.2	4	2	1	1.8	-1.4	-4	1	2	0.0*	0.2	5	-1	1	0.3	0.2
3	-4	10	0.9	-1.1	-3	-3	2	6.0	5.5	4	2	2	7.2	-6.4	-4	1	3	0.0*	0.0	5	2	0	0.0*	-0.2
3	5	0	0.2	0.2	-3	-3	3	3.1	3.1	4	2	3	1.3	-1.0	-4	1	4	0.0*	0.4	5	-2	1	2.3	-2.1
3	5	1	0.9	0.7	-3	-3	4	0.5	0.6	4	2	4	0.9	-0.7	-4	1	5	1.4	-0.9	5	-2	2	0.3	0.3
3	5	2	0.0*	-0.1	-3	-3	5	1.9	1.3	4	2	5	1.8	-1.5	-4	-1	1	0.0*	-0.0	5	3	0	0.7	-0.4
3	5	3	3.5	-3.4	-3	-3	6	0.6	-0.7	4	2	6	1.6	1.1	-4	-1	2	2.6	-2.0	5	-3	1	0.3	0.4
3	-5	1	1.3	1.0	-3	-3	7	1.4	-1.3	4	2	7	0.7	0.5	-4	-1	3	0.0*	-0.2	5	-3	2	2.6	2.5
3	-5	2	2.6	2.2	-3	4	0	3.7	3.8	4	-2	1	0.7	0.9	-4	-1	4	3.3	3.2	5	-4	1	0.0*	-0.3
3	-5	3	5.2	4.9	-3	4	1	2.3	2.2	4	-2	2	4.2	-3.5	-4	-1	5	2.2	1.8	5	-4	2	3.0	2.8
3	-5	4	0.8	-0.8	-3	4	2	0.0*	-0.7	4	-2	3	1.3	0.9	-4	-1	6	1.4	-1.3	5	-5	1	0.3	0.0
3	-5	5	4.6	-4.5	-3	-4	1	5.2	-5.2	4	-2	4	3.1	-2.5	-4	-1	7	0.7	-0.7	5	-5	2	0.3	0.5
3	-5	6	0.8	-0.8	-3	-4	2	2.5	2.3	4	-2	5	2.3	-1.8	-4	2	0	8.2	10.3	5	-6	1	1.7	1.9
3	-5	7	2.6	2.4	-3	-4	3	5.3	5.2	4	-2	6	0.7	-0.5	-4	2	1	3.8	3.7	5	-6	2	1.9	2.0
3	-5	8	1.9	1.5	-3	-4	4	1.5	1.3	4	-2	7	3.1	-2.8	-4	2	2	3.3	-2.8	-5	0	1	0.3	0.8
3	-5	9	3.1	-3.0	-3	-4	5	0.3	0.7	4	-2	8	0.0*	0.1	-4	2	3	0.3	-0.8	-5	0	2	1.7	-1.0
3	-6	1	0.4	0.6	-3	-4	6	0.0*	0.6	4	-2	9	0.9	0.8	-4	2	4	0.0*	-0.2	-5	1	0	0.8	-0.9
3	-6	2	0.6	0.6	-3	-4	7	0.0*	-0.5	4	3	0	0.8	-0.7	-4	2	5	0.0*	-0.0	-5	1	1	2.1	2.0
3	-6	3	5.2	5.1	-3	5	0	2.7	2.6	4	3	1	0.0*	-0.6	-4	-2	1	1.6	1.1	-5	1	2	2.6	2.1
3	-6	4	3.5	3.4	-3	5	1	0.3	-0.3	4	3	2	0.0*	0.0	-4	-2	2	2.4	1.9	-5	-1	1	2.8	2.7
3	-6	5	3.2	-3.2	-3	5	2	0.0*	-0.6	4	3	3	1.4	-1.0	-4	-2	3	2.8	2.4	-5	-1	2	0.0*	0.2
3	-6	6	2.1	-1.9	-3	-5	1	1.0	-1.0	4	3	4	2.5	-2.4	-4	-2	4	0.0*	0.3	-5	2	0	1.8	1.2
3	-6	7	0.0*	0.6	-3	-5	2	0.3	-0.6	4	3	5	0.0*	0.3	-4	-2	5	0.0*	0.1	-5	2	1	6.2	6.3
3	-6	8	0.0*	-0.6	-3	-5	3	1.4	1.5	4	-3	1	0.7	0.7	-4	-2	6	0.0*	-0.2	-5	2	2	2.1	1.6
3	-7	1	0.2	0.5	-3	-5	4	1.1	1.0	4	-3	2	2.2	2.1	-4	3	0	1.8	1.4	-5	-2	1	0.0*	0.7
3	-7	2	0.0*	0.3	-3	6	0	0.3	0.5	4	-3	3	7.2	6.8	-4	3	1	0.3	0.1	-5	-2	2	0.3	0.0
3	-7	3	1.4	1.3	-3	6	1	1.4	-1.4	4	-3	4	0.2	-0.1	-4	3	2	0.6	0.6	-5	3	0	2.6	2.5
3	-7	4	2.1	2.2	-3	-6	1	0.5	1.2	4	-3	5	7.7	-7.4	-4	3	3	0.3	-0.2	-5	3	1	2.8	2.4
3	-7	5	0.0*	0.3	-3	-6	2	2.2	2.3	4	-3	6	2.0	-1.5	-4	3	4	0.0*	-0.4	-5	3	2	1.0	-0.9
3	-7	6	1.2	1.0	-3	7	0	0.4	-0.6	4	-3	7	0.9	-0.7	-4	3	5	0.0*	-0.1	-5	-3	1	0.0*	-0.3
3	-7	7	1.4	1.4	4	0	0	1.0	0.6	4	-3	8	2.0	-1.9	-4	-3	1	3.5	-3.0	-5	-3	2	1.4	-1.1
-3	0	1	3.3	3.1	4	0	1	2.5	2.0	4	-3	9	1.7	1.2	-4	-3	2	0.3	0.2	-5	4	0	1.7	-0.6
-3	0	2	1.4	1.2	4	0	2	1.3	1.0	4	4	0	0.5	0.5	-4	-3	3	5.9	5.4	-5	4	1	0.0*	0.1
-3	0	3	3.0	3.1	4	0	3	2.8	-1.6	4	4	1	1.5	1.1	-4	-3	4	2.6	2.7	-5	4	2	1.4	1.4
-3	0	4	0.0*	0.2	4	0	4	0.0*	0.6	4	4	2	0.3	-0.5	-4	-3	5	1.2	-1.0	-5	5	0	0.0*	-0.2
-3	0	5	0.3	0.4	4	0	5	3.1	-2.9	4	4	3	3.4	-2.8	-4	-3	6	0.0*	-0.6	-5	5	1	1.3	1.6
-3	1	0	12.6	14.9	4	0	6	3.7	-3.7	4	4	4	0.7	-1.0	-4	4	0	0.0*	0.0	-5	5	2	1.9	1.7
-3	1	1	1.3	1.2	4	0	7	0.4	0.6	4	-4	1	2.5	-2.4	-4	4	1	3.9	3.9	-5	6	0	0.0*	0.0
-3	1	2	2.6	1.9	4	0	8	2.1	-1.6	4	-4	2	0.0*	-0.4	-4	4	2	2.0	1.7	6	0	0	0.5	0.8
-3	1	3	2.6	1.9	4	1	0	6.4	6.3	4	-4	5	4.0	-3.2	-4	4	3	1.7	-1.5	6	1	0	0.2	-0.0
-3	-1	1	1.8	-1.8	4	1	1	6.2	5.8	4	-4	6	3.5	-3.2	-4	4	4	0.0*	-0.5	6	-3	1	1.2	1.4
-3	-1	2	0.3	-0.3	4	1	2	6.0	-5.4	4	-4	7	0.0*	-0.1	-4	-4	1	1.6	-1.4	6	-4	1	0.8	0.9
-3	-1	3	3.3	3.7	4	1	3	6.0	-5.1	4	-4	8	2.4	-2.6	-4	-4	2	2.3	-2.0	6	-4	2	1.4	1.3
-3	-1	4	2.7	2.7	4	1	4	0.0*	-0.2	4	5	0	0.3	-1.3	-4	-4	3	1.6	1.7	6	-5	2	1.1	0.8
-3	-1	5	0.3	-0.5	4	1	5	3.3	-2.9	4	-5	1	0.3	0.4	-4	-4	4	3.7	2.7	-6	0	1	1.4	1.3
-3	-1	6	0.6	0.5	4	1	6	0.0*	-0.5	4	-5	2	2.0	2.0	-4	-4	5	0.3	0.5	-6	0	2	0.0*	0.3
-3	2	0	4.0	4.0	4	1	7	0.0*	0.3	4	-6	1	0.0*	-0.0	-4	5	0	1.7	1.6	-6	1	0	1.7	1.3
-3	2	1	0.5	0.3	4	1	8	1.0	-0.9	4	-6	2	4.2	4.2	-4	5	1	2.3	2.6	-6	1	1	0.6	0.5
-3	2	2	4.5	3.1	4	-1	1	6.2	-6.1	4	-7	1	0.0*	-1.0	-4	5	2	0.8	-0.9	-6	1	2	0.0*	0.5
-3	-2	1	1.0	0.7	4	-1	2	4.1	-4.1	4	-7	2	1.0	1.7	-4	5	3	1.4	-1.4	-6	-1	1	0.5	1.0
-3	-2	2	0.9	0.8	4	-1	3	1.3	1.0	-4	0	1	0.6	0.4	-4	5	4	0.5	-0.3	-6	2	0	1.5	0.6
-3	-2	3	1.1	1.1	4	-1	4	0.6	1.1	-4	0	2	0.0*	-0.5	-4	-5	1	0.1	0.2	-6	2	1	0.3	0.7
-3	-2	4	3.1	2.8	4	-1	5	0.3	0.4	-4	0	3	2.4	2.2	-4	6	0	0.2	-0.3	-6	2	2	1.8	1.9
-3	-2	5	0.8	0.7	4	-1	6	4.3	-4.2	-4	0	4	3.8	3.7	-4	6	1	0.0*	-0.0	-6	3	0	0.5	0.4
-3	-2	6	1.8	-1.4	4	-1	7	1.8	-1.7	-4	0	5	0.5	-0.5	-4	6	2	0.8	-0.7	-6	3	1	1.5	1.7
-3	3	0	1.5	1.4	4	-1	8	0.0*	0.4	-4	0	6	1.8	-1.5	-4	7	0	0.0*	-0.5	-6	3	2	1.4	[illegible]
																				-6	4	0	0.2	0.4
																				-6	4	1	0.4	0.5
																				-6	5	0	0.1	0.2

imum deviation of 0·003 Å. The angles between the planes of the carbon atoms (126), (2356) and (345) are 130·6° and 128·9°. The principal interatomic distances and bond angles of the molecule were calculated for the atomic positions corrected for thermal libration of the molecule as rigid body (as described below). These data are given in Table 5 and Fig. 3. The uncorrected values of the C–C bond lengths are also given to show the effect of thermal libration.

The thermal motion analysis

The root mean square amplitudes and the vectors of the principal axes of the vibration ellipsoids for the individual carbon atoms were calculated from their anisotropic thermal parameters given in Table 3. From these data the distances of the ends of the principal axes to the two planes of the molecule, the non-crystallographic mirror plane and the plane formed by the carbon atoms (6211′2′6), respectively, were calculated and are given in Table 6. This confirmed that within experimental errors the ellipsoids of thermal motion conform to the non-crystallographic molecular mirror plane.

The thermal motion has also been analyzed as rigid-body molecular vibration on the basis of Cruickshank's (1961*b*) procedure using Gantzel, Coulter & Trueblood's (1961) program. The calculations are referred to an orthogonalized system of axes, as specified in *International Tables* (1959). The U tensors of the thermal vibration of the individual atoms calculated from rigid-body thermal motion (U_{calc}) are compared with the thermal parameters from the structure analysis (U_{obs}) in Table 7. The good agreement supports the rigid-body assumption. The translational **T** and librational **ω** tensors are given in Table 8, together with their standard deviations in parentheses. The **T** and **ω** tensor ellipsoids are compared with the vibrational ellipsoids of the individual atoms in Table 9, where the principal axes of the librational ellipsoids are represented by their unit vectors. The angles of the principal axes to the axis of the molecule defined by the center of symmetry of the molecule and the center of gravity of the triangle formed by the carbon atoms (345) are also given. These show that both the translational and librational ellipsoids are so oriented that the two longer principal axes are always parallel to the non-crystallographic molecular mirror plane and the longest one is parallel also to the axis of the molecule defined above. The latter axis is that around which there is the greatest libration and the next greatest one is around the other principal axis being parallel to the non-crystallographic

Table 5. *The bond lengths, bond angles and non-bonding hydrogen distances in the bicyclohexylidene molecule, with their estimated standard deviations*

The distances are corrected for thermal libration. The uncorrected bond lengths are also given in parentheses.

Atoms	l	$\sigma(l)$	Atoms	Angle	$\sigma(°)$
C(1)–C(1′)	1·332 Å (1·329)	0·003 Å	C(1)–C(2)–C(3)	111·9°	0·3°
C(1)–C(2)	1·509 (1·503)	0·003	C(2)–C(3)–C(4)	111·2	0·3
C(2)–C(3)	1·529 (1·527)	0·004	C(3)–C(4)–C(5)	110·8	0·3
C(3)–C(4)	1·520 (1·515)	0·004	C(4)–C(5)–C(6)	110·4	0·3
C(4)–C(5)	1·518 (1·514)	0·004	C(5)–C(6)–C(1)	111·8	0·3
C(5)–C(6)	1·533 (1·530)	0·004	C(6)–C(1)–C(2)	110·6	0·3
C(6)–C(1)	1·526 (1·520)	0·003			
H(21)–C(2)	0·979	0·038	H(21)–C(2)–H(22)	112·1	4·3
H(22)–C(2)	1·093	0·038			
H(31)–C(3)	1·025	0·040	H(31)–C(3)–H(32)	112·3	4·5
H(32)–C(3)	1·002	0·040			
H(41)–C(4)	1·003	0·039	H(41)–C(4)–H(42)	109·7	4·5
H(42)–C(4)	0·991	0·039			
H(51)–C(5)	1·045	0·040	H(51)–C(5)–H(52)	109·2	4·6
H(52)–C(5)	0·972	0·040			
H(61)–C(6)	0·969	0·038	H(61)–C(6)–H(62)	111·8	4·4
H(62)–C(6)	1·042	0·038			
H(21)–H(6′1)	1·944	0·076			

Mean values

C–C	1·523 Å
C=H	1·332
C–H	1·012
C–C–C	111·05°

Table 6. *The principal axes of the ellipsoids of thermal vibration for the carbon atoms given by their lengths and vectors, referred to the crystal axes in* Å

The mid-points of the ellipsoids are at the origin .The last two columns give the distances of the end points of the principal axes from the non-crystallographic symmetry plane of the molecule D_1 and the plane formed by the carbon atoms (1, 2, 6), D_2, respectively.

Atom	i	$(u^2)^{\frac{1}{2}}$ (Å)	x (Å)	y (Å)	z (Å)	D_1 (Å)	D_2 (Å)
C(1)	1	0·168	0·105	0·165	0·016	0·149	0·044
	2	0·178	−0·082	0·027	−0·130	0·080	−0·075
	3	0·224	−0·157	0·092	0·168	0·003	−0·194
C(2)	1	0·180	0·122	0·150	0·096	0·099	0·058
	2	0·207	−0·057	0·137	−0·088	0·164	−0·091
	3	0·258	−0·193	0·043	0·208	−0·062	−0·215
C(3)	1	0·178	0·116	0·141	0·108	0·085	0·054
	2	0·237	0·057	0·127	−0·177	0·199	0·028
	3	0·261	0·205	−0·129	−0·114	−0·060	0·247
C(4)	1	0·173	0·092	0·056	0·144	−0·010	0·057
	2	0·247	−0·091	−0·239	0·047	−0·244	−0·012
	3	0·264	0·217	−0·089	−0·163	−0·000	0·249
C(5)	1	0·180	0·065	−0·032	0·141	−0·090	0·060
	2	0·227	−0·107	−0·237	−0·066	−0·192	−0·018
	3	0·261	0·225	−0·052	−0·167	0·036	0·245
C(6)	1	0·176	−0·001	−0·064	0·146	−0·125	0·008
	2	0·205	−0·160	−0·170	−0·068	−0·132	−0·090
	3	0·262	0·182	−0·151	−0·146	−0·066	0·235

Table 7. *The observed and calculated components of the U tensors of the individual carbon atoms referred to the orthogonalized coordinates axes* (in 10^{-2}Å^2)

	U_{11}		U_{22}		U_{33}		U_{12}		U_{13}		U_{23}	
Atom	obs.	calc.	obs.	calc.	obs.	calc.	obs.	calc.	obs.	calc.	obs.	calc.
C(1)	3·93	4·23	2·96	3·22	4·09	3·88	−0·36	−0·34	−0·89	−0·79	0·24	0·23
C(2)	4·95	5·26	3·84	3·55	5·40	4·91	−0·31	−0·29	−1·44	−1·30	−0·43	−0·36
C(3)	5·71	5·47	4·88	4·73	5·02	5·33	−1·18	−1·08	−1·19	−1·51	−0·67	−0·46
C(4)	5·52	5·23	6·08	6·14	4·46	4·59	−0·31	−0·36	−1·89	−1·57	−0·16	−0·32
C(5)	5·60	5·71	4·90	5·17	4·71	4·88	0·36	0·18	−1·59	−1·86	0·55	0·60
C(6)	5·60	5·40	4·29	4·14	4·26	4·34	−0·67	−0·59	−1·27	−1·27	1·07	0·91

molecular mirror plane. The atomic coordinates corrected for this thermal libration, using Cruickshank's (1961*b*) procedure of calculation, with the peak shape parameter $q^2=0{\cdot}13$, are given in Table 2.

Discussion of the structure

In the molecule of bicyclohexylidene the six carbon atoms associated with the central double bond are strictly planar, with a mean observed deviation of 0·001 Å. The deviations in atomic distances and angles from the molecular mirror plane perpendicular to the plane of the ethylene bond and passing through the carbon atoms (411′4′) are also insignificant, being on average 1σ of the C–H bond lengths and C–C–C bond angles, respectively.

The cyclohexane rings have the chair conformation with some flattening relative to the ideal strain-free configuration. The dihedral angles between the three planes of the carbon atoms in the ring are 49·4° and 51·1°, as compared with 60° for the ideal configuration. This distortion has recently been discussed by Wohl (1964). There is a corresponding increase in the C–C–C bond angles from the ideal tetrahedral to a mean value of 111·1°. This agrees very closely with the 111·55 ± 0·15° determined for cyclohexane by electron diffraction (Davis & Hassel, 1963). The central double bond length is equal to that reported in ethylene (1·334 Å) by Bartell (1957) within 0·002 Å, which is less than 1σ.

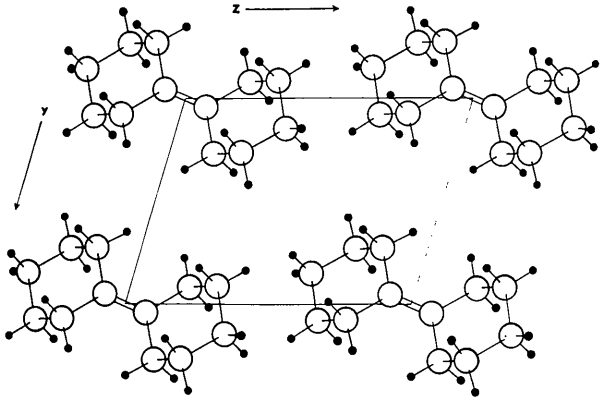

Fig. 4. The orthogonal projection of the structure of bicyclohexylidene on (100). Open circles, carbon; black dots, hydrogen atoms.

The mean C–C bond length in the cyclohexane rings of 1·523 Å agrees within 1σ with the value reported by Davis & Hassel (1963) for cyclohexane itself. The bond lengths appear to vary in a systematic manner, as shown in Fig. 3, with differences close to 3σ. Statistical tests on these variations were therefore made by the procedure of Cruickshank (1949). These results show that the C(1)–C(2) and C(1)–C(6) bonds are not significantly shorter than the mean of the other four, contrary to the quantum mechanical prediction that sp^2–sp^3C–C bonds should be shorter than sp^3–sp^3C–C bonds (Dewar & Schmeising, 1960). However, it is interesting to note that the difference of 0·013 Å between the mean of C(2)–C(3) and C(5)–C(6) bonds and the mean of the other four is at the 99·5% significance level, but there have been no theoretical predictions of such an observation.

The C–H bond lengths vary over a range of 1·5σ (±0·03 Å) with a mean value of 1·01 Å in good agreement with those from neutron diffraction structure analyses (Hamilton, 1962). The H–C–H bond angles have a mean value of 111·0°.

The shortest non-bonding H–H distances in the molecule are within the methylene groups with a mean value of 1·63 Å, and between the methylene groups on different rings at 1·94 Å. Although the latter is significantly shorter than the hydrogen van der Waals diameter (2·34 Å), the cyclohexane rings maintain their chair conformation and do not adopt the boat form, as was predicted by Tömösközi (1959).

The packing of the molecules in the structure is illustrated in Fig. 4. It conforms to the concept of molecular close-packing of Kitaigorodskii (1961). Since the external surface of the molecules is almost entirely hydrogen atoms, the intermolecular distances give a useful mean value for hydrogen van der Waals radius.

Table 8. *The translational* **T** (10^{-2}Å^2) *and librational* **ω** (10^{-2}rad^2) *tensors of the* $C_{12}H_{20}$ *molecule as rigid body referred to orthogonalized axes*

The e.s.d. given in parentheses refer to the last two digits.

$$\mathbf{T}=\begin{pmatrix} 4{\cdot}18\ (18) & -0{\cdot}34\ (13) & -0{\cdot}77\ (15) \\ & 3{\cdot}08\ (16) & 0{\cdot}27\ (12) \\ & & 3{\cdot}87\ (14) \end{pmatrix} \qquad \boldsymbol{\omega}=\begin{pmatrix} 0{\cdot}48\ (07) & 0{\cdot}05\ (03) & 0{\cdot}27\ (07) \\ & 0{\cdot}15\ (03) & 0{\cdot}10\ (03) \\ & & 0{\cdot}54\ (09) \end{pmatrix}$$

Table 9. *The translational and librational tensors of the thermal motion of the molecule as a rigid body*

The data are presented as in Table 8 with the inclusion of another column listing the angles of the principal axes to the molecular axis (see text). For ω the unit vectors of the principal axes are given.

Tensor	*i*	$(u^2)^{\frac{1}{2}}$ (Å)	*x* (Å)	*y* (Å)	*z* (Å)	D_1 (Å)	D_2 (Å)	Angle (°)
T	1	0·222	0·170	−0·091	−0·144	−0·013	0·204	90·3
	2	0·180	0·094	0·034	0·148	−0·031	0·066	10·1
	3	0·173	0·083	0·168	−0·023	0·168	0·026	79·8
ω	1	0·089	−0·600	−0·386	−0·786	−0·025	−0·375	178·6
	2	0·050	−0·770	0·416	0·636	0·068	−0·922	90·5
	3	0·036	−0·376	−0·913	0·300	−0·989	−0·082	88·7

Table 10. *The shortest intermolecular* H–H *and* C–C *distances for every atom in the molecule*

The atoms in the first column belong to the reference molecule at (x, y, z) corresponding to the data in Table 2.

From atom	To atom	In molecule at			Distance	
H(21)	H(61)	x	$1+y$	z	2·810 Å	Mean: 2·656 Å Min: 2·531
H(22)	H(31)	$-1+x$	y	z	2·689	
H(31)	H(51)	$1-x$	$-y$	$-z$	2·576	
H(32)	H(41)	$-1-x$	$1-y$	$1-z$	2·735	
H(41)	H(22)	$1+x$	y	z	2·531	
H(42)	H(52)	$-x$	$-y$	$1-z$	2·713	
H(51)	H(62)	$1+x$	y	z	2·710	
H(52)	H(62)	$-x$	$-y$	$1-z$	2·560	
H(61)	H(32)	$-1+x$	$-1+y$	z	2·684	
H(62)	H(41)	$-1+x$	y	z	2·548	
C(1)	C(4)	$-1+x$	y	z	4·063	Mean: 3·900 Å Min: 3·734
C(2)	C(5)	x	$1+y$	z	3·849	
C(3)	C(2)	$1+x$	y	z	3·957	
C(4)	C(4)	$1-x$	$1-y$	$1-z$	3·734	
C(5)	C(2)	x	$-1+y$	z	3·849	
C(6)	C(5)	$-1+x$	y	z	3·948	

The shorter intermolecular H–H and C–C distances are given in Table 10 with every H and C atom given at least once. The shortest H–H distance is 1·27 Å, and the mean value of the ten shortest H–H distances is 1·33 Å. This is considerably greater than the hydrogen van der Waals radius of 1·17 Å determined by Kitaigorodskii (1961). However, this value was based on the assumption of a C–H bond length of 1·08 Å, whereas in the present structure the mean C–H bond length was determined to be 1·01 Å. Similarly, a methylene van der Waals radius of 1·87 Å as the minimum and 1·96 Å as a mean value was derived. These also are a little longer than the usually accepted value of 1·80 Å.

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