dium atoms have different coordinations. These are indicated in Fig. 1. Table 3 gives the sodium–oxygen distances below 3·52 Å. Since there are no distances in the range 3·13 to 3·52 Å, the number of oxygen atom neighbours at 3·13 Å or less are taken to be the coordination number of sodium. This number is 7, 6 and 8 for Na(1), Na(2) and Na(3) respectively. The coordinating oxygen atoms do not surround the cations in a symmetrical fashion. The sodium–oxygen distances of the coordinating oxygens range from 2·237 to 3·130 Å. In comparison *International Tables for X-ray Crystallography* (1962) gives for sixfold coordinated sodium, the range 2·25 to 2·78 Å derived from 16 determinations (The sodium–oxygen distances listed by Hyman, Perloff, Mauer & Block (1967) for sodium tetraborate cannot be reconciled with their parameter list.)

The present results reinforce the general impression that the cation sites in borate polymer networks depend strongly on the requirements of the polymer anion.

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The Conformation of Non-Aromatic Ring Compounds. LXXIV.* The Crystal and Molecular Structure of *cis*-2-Chloro-4-t-butylcyclohexanone

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Crystals of *cis*-2-chloro-4-t-butylcyclohexanone are triclinic. The space group is $P\bar{1}$ and $Z=2$. The unit-cell dimensions are $a=8{\cdot}294$, $b=10{\cdot}563$, $c=6{\cdot}366$ Å, $\alpha=100{\cdot}8$, $\beta=97{\cdot}4$ and $\gamma=79{\cdot}5°$. The structure has been solved by Patterson methods and direct methods and refined with a least-squares procedure using Mo $K\alpha$ data measured with a three-circle diffractometer. The final conventional R value is 3·7 %. The cyclohexane ring has a chair conformation with the chlorine atom and the t-Bu group in the equatorial positions. The staggering of the latter group with respect to the ring atoms is − 1·5°. The thermal motion has been analysed in terms of a rigid body. Statistical tests indicate that the rigid body model without methyl groups cannot be rejected on a 5 % significance level and suggest a librational motion of the t-Bu group.

Introduction

This investigation forms part of the studies in this laboratory concerning the geometry of t-Bu groups. Fraterman & Romers (1971) (hereafter referred to as FR) showed that the $C(CH_3)_3$ groups in the aromatic compound 2,6-di-t-butyl-4-(*p*-bromophenyl)imino-2,5-cyclohexadien-1-one have conspicuously high temperature factors and presumably suffer from restricted rotation (libration) about the central C–C bonds, or possibly show conformational disorder. This phenomenon has not been encountered in other aromatic compounds with t-Bu groups (consult FR for literature) but might be present in analogous cyclohexane derivatives. Altona & Sundaralingam (1970) carried out empirical force-field calculations of a number of substituted cyclohexane compounds. Their calculations indicate that the $C(CH_3)_3$ groups do not occupy staggered positions relative to the ring bonds. The angle of twist with respect to the ideally staggered position (± 17°) is concomitant with an asymmetrical distortion of the cyclohexane ring. A very low barrier (300 cal.mole^{-1}) separates the two equivalent forms and the authors suggest that in diffraction analysis the twisting phenomenon would reveal itself by unusual large temperature factors (disorder) of atoms of the t-Bu group and, to a less degree, of the ring atoms.

So far no published experimental evidence is avail-

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* Part LXXIII: Koer, de Kok & Romers (1972)

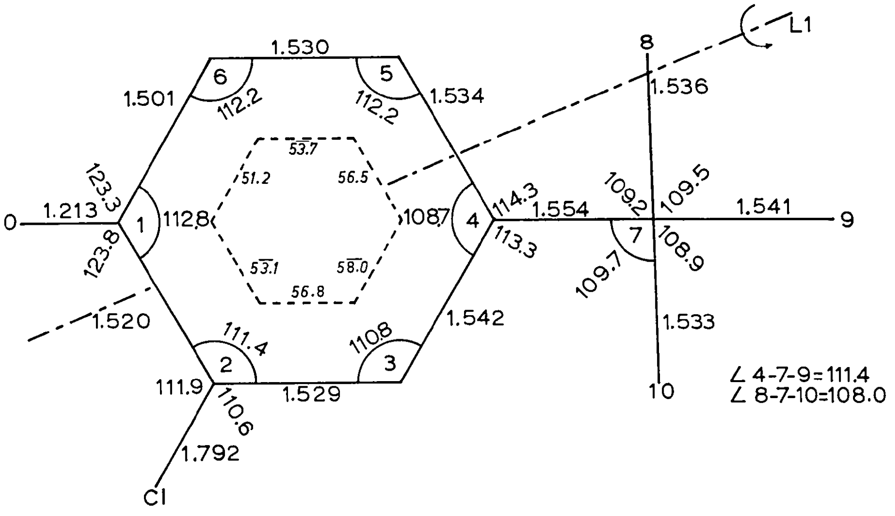

Fig. 1. Bond lengths (Å), valency and endocyclic torsional angles (decimal degrees) of CBC. The direction of largest libration axis L_1 is indicated in the figure.

able to support this view. The molecular structures of *cis*- and *trans*-4-t-butyl-1-chlorocyclohexane were investigated by means of gas-phase electron diffraction (Dallinga & Toneman, 1969). In all models considered constraints of C_s symmetry and staggered conformations were, however, maintained. The aim of the present X-ray diffraction investigation is to verify the above-mentioned prediction using accurate diffractometer measurements of the title compound (hereafter CBC).

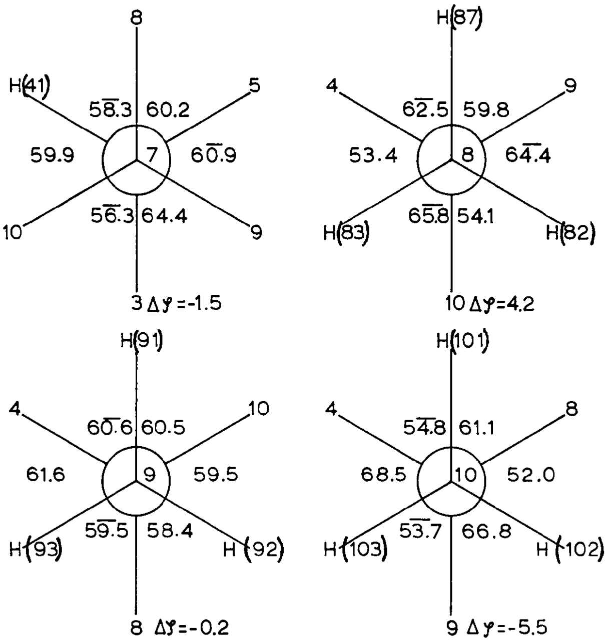

Fig. 2. Newman projections along the bonds C(7)–C(4)-C(8)–C(7), C(9)–C(7) and C(10)–C(7). The amount of 'off, staggering' indicates the average angle of twist (positive if clockwise, negative if counter-clockwise) required to restore ideal staggering.

Experimental

The compound CBC was prepared according to the description of Allinger, Allinger, Freiberg, Czaja & Le Bel (1960). Crystallization from a solution in heptane gave colourless needles elongated along a zone called [001]. Preliminary rotation and Weissenberg photographs indicated triclinic diffraction symmetry. The space group $P\bar{1}$ was established by the Patterson analysis and subsequent successful refinement of the centrosymmetric structure. Approximate values of a^*, b^*, c^*, α^*, and γ^* were obtained from crystals rotated about [100] and [001]. Precise values of the reciprocal lattice at 20°C (Table 1) were measured on a three-circle diffractometer using orientations in which c, a and a^* coincide with the φ axis of the instrument. Cu $K\alpha$ radiation ($\lambda = 1{\cdot}54178$ Å) was used during these calibrations.

The boundary planes of a carefully selected crystal with dimensions $0{\cdot}36 \times 0{\cdot}24 \times 0{\cdot}30$ mm were measured with a binocular microscope. The crystal was mounted about [001] on a goniometer head of the crystal orienter. By means of zirconium-filtered Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å), employing the θ–2θ scan technique and using a scintillation counter equipped with pulse-height discriminator, 1468 reflexions were measured at 20°C in a range between 3 and 25°. An additional 421 reflexions having counts less than twice the standard deviation were considered as being non-observed. More details of the measurement technique have been given elsewhere (Portheine, Romers & Rutten, 1972). The reduction to structure factors was performed in the usual way by dividing by Lorentz, polarization and absorption factors. The latter factors were calculated according to a program designed by de Graaff (1972).

Table 1. *Crystallographic data of CBC*

The standard deviations are given in parentheses.

cis-2-Chloro-4-t-butylcyclohexanone, $C_{10}H_{17}OCl$; melting point 59°C; space group $P\bar{1}$:

Bravais-reduced lattice

$d(100) = 8{\cdot}1143\ (6)$ Å	$a = 8{\cdot}294\ (4)$ Å
$d(010) = 10{\cdot}2376\ (10)$	$b = 10{\cdot}563\ (5)$
$d(001) = 6{\cdot}2231\ (8)$	$c = 6{\cdot}366\ (3)$
$\alpha^* = 80{\cdot}35\ (2)°$	$\alpha = 100{\cdot}8\ (9)°$
$\beta^* = 84{\cdot}34\ (6)$	$\beta = 97{\cdot}4\ (8)$
$\gamma^* = 99{\cdot}37\ (4)$	$\gamma = 79{\cdot}5\ (7)$

d_{obs}(by flotation) = $1{\cdot}15$ g.cm^{-3}, $d_x = 1{\cdot}168$ g.cm^{-3}; $V = 536{\cdot}0$ Å^3; $Z = 2$; μ(Mo $K\alpha$) = $3{\cdot}1$ cm^{-1}

Structure refinement

Assuming the space group $P\bar{1}$, the structure was solved by Patterson methods as well as direct methods. Since the two methods gave the same results and were carried out as a routine procedure, no details will be given here. The refinement with anisotropic temperature parameters for the heavy atoms and isotropic B values for hydrogen atoms was carried out successfully and was stopped after several cycles attaining a minimum R value of 3·7% (observed reflexions only). The weighted value R_w (omitting non-observed reflexions)

Table 2. *Absolute values of observed and calculated structure factors* (FO *and* FC) *of CBC on* 100 *times absolute scale*

Phases of FC have been omitted. Reflexions with amplitudes below a certain threshold value have not contributed to the refinement and were assigned the threshold value and a negative sign. Zero FO values represent reflexions with very small θ values that could not be measured.

H	K	L	FO	FC
-9	-6	0	308	267
-9	-5	0	597	542
-9	-4	0	196	142
-8	-8	0	277	313
-8	-7	0	249	184
-8	-6	0	272	291
-8	-1	0	640	590
-8	0	0	503	559
-8	1	0	266	227
-8	3	0	720	672
-8	4	0	778	748
-7	-7	0	232	218
-7	-6	0	328	329
-7	-5	0	269	297
-7	-4	0	1856	1869
-7	-3	0	422	407
-7	-2	0	653	665
-7	0	0	182	230
-7	2	0	819	817
-7	3	0	214	193
-7	5	0	332	298
-7	6	0	260	278
-6	-9	0	247	282
-6	-8	0	1029	1018
-6	-6	0	284	267
-6	-5	0	2345	2347
-6	-4	0	2678	2702
-6	-2	0	1255	1210
-6	-1	0	535	527
-6	0	0	1019	1012
-6	1	0	555	496
-6	2	0	167	139
-6	3	0	531	543
-6	4	0	440	448
-6	5	0	869	913
-6	6	0	316	288
-6	7	0	284	267
-6	8	0	1191	1141
-5	-9	0	2042	2040
-5	-8	0	1634	1685
-5	-7	0	288	265
-5	-6	0	609	626
-5	-5	0	286	355
-5	-4	0	1007	947
-5	-3	0	1116	1106
-5	-2	0	416	361
-5	-1	0	1648	1639
-5	0	0	1131	1232
-5	1	0	2066	2003
-5	2	0	1249	1257
-5	3	0	1458	1427
-5	4	0	4664	4704
-5	5	0	970	1025
-5	6	0	1225	1235
-5	7	0	541	555
-5	8	0	247	291
-5	9	0	393	335
-4	-9	0	790	825
-4	-8	0	271	310
-4	-7	0	1637	1640
-4	-6	0	675	657
-4	-5	0	1484	1553
-4	-4	0	383	419
-4	-3	0	2273	2151
-4	-2	0	2349	2377
-4	-1	0	202	205
-4	0	0	6718	6420
-4	1	0	277	341
-4	2	0	2482	2428
-4	3	0	1777	1864
-4	4	0	2480	2539
-4	5	0	439	424
-4	6	0	1266	1302
-4	8	0	205	241
-4	9	0	842	901
-4	10	0	733	768
-3	-12	0	567	583
-3	-11	0	439	446
-3	-10	0	260	208
-3	-9	0	654	560
-3	-8	0	638	647
-3	-7	0	921	925
-3	-5	0	1674	1705
-3	-4	0	3140	3061
-3	-3	0	2362	2430
-3	-2	0	1049	1048
-3	-1	0	8610	8397
-3	0	0	8004	7912
-3	1	0	2691	2734
-3	2	0	561	545
-3	3	0	850	876
-3	4	0	3123	3097
-3	5	0	963	998
-3	6	0	649	698
-3	7	0	1009	1007
-3	8	0	1341	1324
-3	9	0	746	652
-2	-12	0	223	290
-2	-10	0	574	555
-2	-9	0	1127	1123
-2	-8	0	3348	3344
-2	-6	0	1060	1069
-2	-5	0	2246	2326
-2	-4	0	1000	1034
-2	-3	0	1812	1834
-2	-2	0	4182	4217
-2	-1	0	763	227
-2	0	0	3962	4039
-2	1	0	2359	2328
-2	2	0	2505	2495
-2	3	0	4461	4573
-2	4	0	4120	4094
-2	5	0	3643	3646
-2	6	0	2167	2158
-2	7	0	1537	1543
-2	8	0	1051	1150
-2	10	0	779	819
-2	11	0	871	952
-1	-12	0	300	297
-1	-11	0	671	721
-1	-10	0	924	893
-1	-9	0	1596	1627
-1	-8	0	2046	2064
-1	-7	0	1742	1729
-1	-6	0	3404	3348
-1	-5	0	315	243
-1	-4	0	1504	1547
-1	-3	0	2444	2446
-1	-2	0	7396	7270
-1	2	0	4397	4337
-1	3	0	362	403
-1	4	0	10850	10639
-1	5	0	3656	3596
-1	6	0	1976	1980
-1	7	0	667	693
-1	8	0	978	985
-1	9	0	1398	1364
-1	10	0	731	787
-1	11	0	232	230
0	-12	0	1070	1022
0	-11	0	764	748
0	-10	0	505	553
0	-9	0	2214	2207
0	-7	0	214	353
0	-6	0	1140	1157
0	-5	0	4201	4038
0	-4	0	2386	2416
0	-3	0	1896	1894
0	-2	0	208	250
2	7	0	179	187
2	11	0	192	167
6	7	0	172	49
9	3	0	209	191
-9	-4	1	701	632
-9	-2	1	293	267
-9	-1	1	442	445
-9	0	1	447	416
-8	-6	1	662	654
-8	-5	1	300	217
-8	-4	1	298	372
-8	-1	1	378	458
-8	0	1	519	537
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-8	2	1	217	154
-8	3	1	215	123
-8	4	1	196	92
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-7	-7	1	280	331
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-7	2	1	307	357
-7	4	1	684	628
-7	5	1	398	334
-6	-11	1	287	287
-6	-9	1	1039	1020
-6	-8	1	1210	1181
-6	-7	1	473	460
-6	-6	1	404	397
-6	-5	1	1643	1697
-6	-4	1	2691	2663
-6	-3	1	1085	1045
-6	-2	1	1885	1883
-6	-1	1	587	580
-6	0	1	324	208
-6	1	1	530	475
-6	2	1	1365	1386
-6	3	1	1044	1086
-6	4	1	1332	1328
-6	6	1	954	991
-6	7	1	322	323
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-5	0	1	623	704
-5	1	1	1394	1421
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-5	3	1	405	491
-5	4	1	1052	1173
-5	5	1	1052	1041
-5	6	1	299	269
-5	7	1	417	396
-5	8	1	730	719
-5	9	1	930	908
-4	-12	1	467	411
-4	-11	1	328	376
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-4	-9	1	929	940
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-3	-6	1	173	120
-3	-5	1	992	840
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-3	2	1	1751	1646
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-3	4	1	276	290
-3	6	1	1201	1177
-3	7	1	611	618
-3	8	1	264	293
-3	9	1	564	580
-3	10	1	603	716
-2	-12	1	194	230
-2	-11	1	312	330
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0	-10	1	1678	1681
0	-9	1	410	446
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0	-5	1	1492	1491
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0	4	1	2795	2590
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0	6	1	1653	1604
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0	9	1	637	558
0	10	1	1196	1238
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1	-4	1	1461	1449
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2	-5	1	2431	2453
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2	0	1	2941	2878
2	1	1	2568	2471
2	2	1	1788	1757
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4	-7	1	1293	1272
4	-5	1	816	808
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4	6	1	1163	1113
4	7	1	208	180
4	8	1	614	646
4	9	1	666	686
4	10	1	256	230
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5	-8	1	727	713
5	-7	1	212	249
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5	-5	1	698	684
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5	-3	1	2392	2411
5	-2	1	783	763
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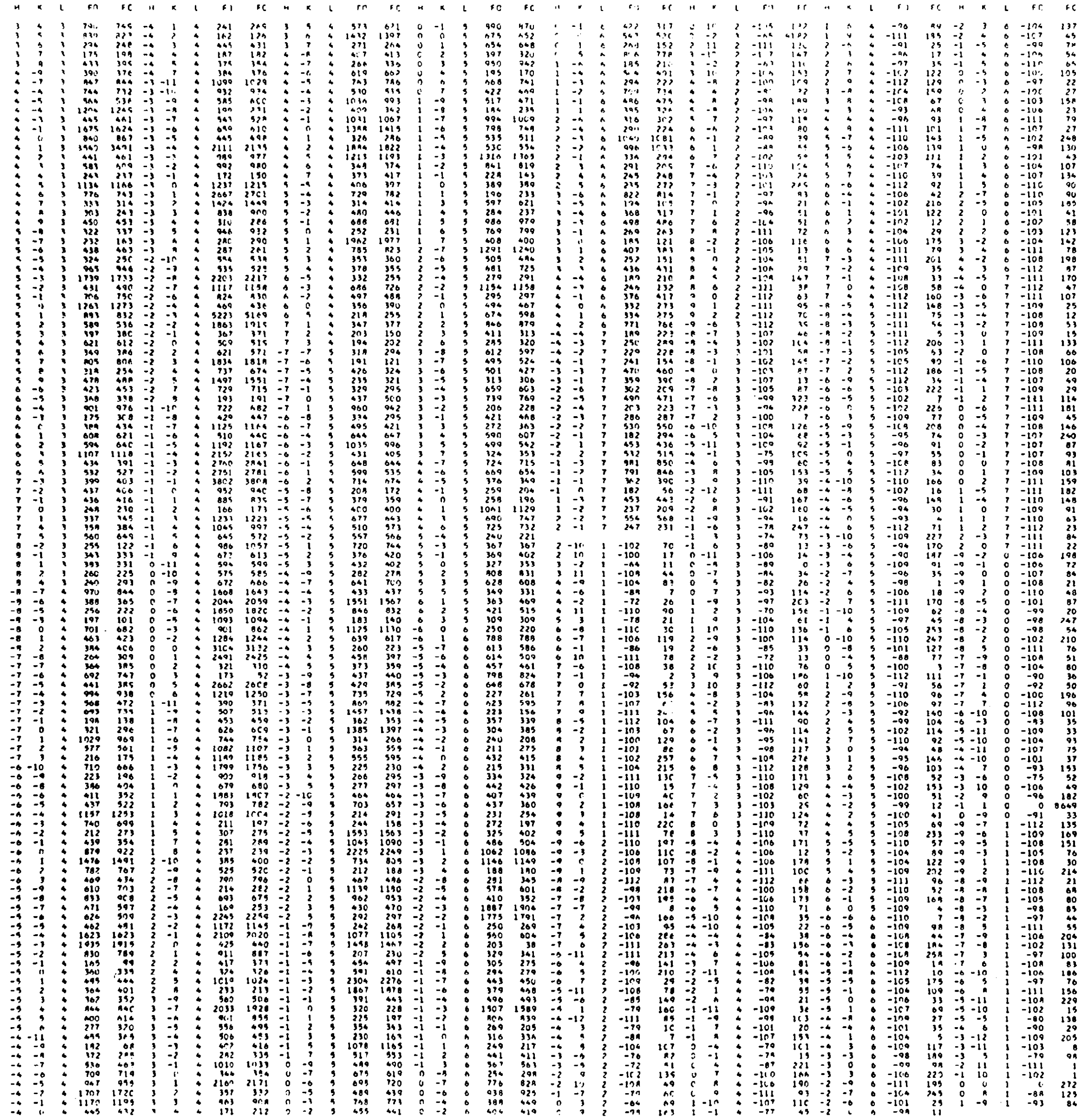
Table 2 (*cont.*)



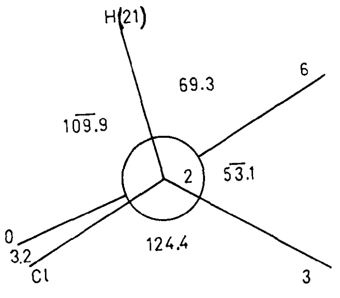


Fig. 3. Newman projection along C(2)–C(1).

is 3·9% and the conventional R value including 421 non-observed reflexions is 7·1%. The scattering values for chlorine, oxygen and carbon were taken from *International Tables for X-ray Crystallography* (1962). Stewart, Davidson & Simpson's (1965) scattering values for hydrogen were incorporated during the final stages of the refinement. The calculated and observed structure factors are listed in Table 2.

The positional parameters of the heavy atoms are listed in Table 3. The coordinates and isotropic B values of the hydrogen atoms are to be found in Table 4. The 'observed' U_{ij} values resulting from the least-squares refinement and the 'calculated' U_{ij} values (see next section) are listed in Table 5. Since the refinement was performed with 3×3 and 6×6 block matrices for the heavy atoms, the estimated standard deviations are too low, probably by 30%.

Table 3. *Fractional coordinates* ($\times 10^4$) *and standard deviations of the non-hydrogen atoms in CBC*

	x	y	z
Cl	3226 (6)	3746 (5)	2843(6)
O	−314 (13)	4647 (13)	2364 (12)
C(1)	427 (17)	5555 (17)	2934 (15)
C(2)	2272 (17)	5410 (16)	3390 (16)
C(3)	2990 (15)	6246 (16)	2160 (16)
C(4)	2217 (15)	7685 (15)	2680 (14)
C(5)	362 (16)	7798 (16)	2129 (17)
C(6)	−395 (17)	6947 (19)	3291 (19)
C(7)	3028 (16)	8608 (16)	1669 (16)
C(8)	2289 (21)	10025 (18)	2460 (21)
C(9)	2777 (20)	8325 (21)	−785 (18)
C(10)	4866 (19)	8440 (20)	2379 (21)

Table 4. *Fractional coordinates* ($\times 10^3$) *and B values* (Å^2) *of the hydrogen atoms in CBC with standard deviations*

	x	y	z	B
H(21)	250 (14)	565 (14)	492 (14)	4·67 (33)
H(31)	280 (14)	592 (14)	59 (14)	4·89 (34)
H(32)	412 (14)	614 (14)	250 (14)	4·94 (34)
H(41)	240 (13)	791 (13)	422 (13)	4·24 (32)
H(51)	15 (15)	756 (15)	56 (14)	5·19 (35)
H(52)	−14 (16)	867 (15)	248 (15)	6·00 (39)
H(61)	−153 (17)	699 (17)	286 (17)	7·04 (43)
H(62)	−30 (17)	724 (17)	484 (17)	6·96 (42)
H(81)	114 (17)	1020 (17)	203 (18)	7·55 (45)
H(82)	287 (20)	1059 (20)	198 (19)	8·94 (52)
H(83)	234(18)	1023 (18)	403 (18)	8·00 (47)
H(91)	325 (18)	744 (18)	−135 (17)	7·98 (47)
H(92)	328 (16)	892 (16)	−137 (17)	7·19 (45)
H(93)	165 (17)	846 (17)	−129 (16)	7·06 (43)
H(101)	509 (17)	857 (18)	396 (18)	7·88 (47)
H(102)	532 (18)	907 (17)	197 (17)	7·52 (46)
H(103)	541 (17)	759 (17)	176 (16)	7·24 (44)

Thermal motion

The thermal motion of the molecule was analysed according to the method described by Schomaker & Trueblood (1968). Omitting the hydrogen atoms in all calculations different groups of atoms contributing to the assumed rigid body were inspected. The model without the methyl carbon atoms gave the best fit between observed and calculated U_{ij} values (Table 5) and is slightly better than the rigid body composed of all heavy atoms. Inspection of these models with Hamilton's ratio test (1965) on a significance level of 5% (Table 6) indicates that the 12-atom rigid body can be rejected. On the other hand, the 9-atom rigid body cannot be rejected in favour of the zero hypothesis (*i.e.* no constraints on thermal parameters).

Table 6. *Numerical data for the* R_w *ratio tests*

The symbols are explained in Hamilton (1965). Significance level $\alpha = 0{\cdot}05$.

	Model I No constraints on thermal parameters	Model II Fixed U_{ij}'s for 12 heavy atoms	Model III Fixed U_{ij}'s for 9 heavy atoms
R	0·0373	0·0491	0·0439
R_w	0·0385	0·0525	0·0462
n	1468	1468	1468
m	177	105	123
b	0	72	54

$R_w(\mathrm{II})/R_w(\mathrm{I}) = 1{\cdot}364$ $\qquad R_w(\mathrm{III})/R_w(\mathrm{I}) = 1{\cdot}200$

$R_{b,\,n-m,\alpha} = R_{72,\,1291,\,0\cdot05} = 1{\cdot}332$ $\qquad R_{54,\,1291,\,0\cdot05} = 1{\cdot}240$

The test, of course, does not give a verdict upon the validity of the libration effect. For the 9-atom model the U_{ij} values were also calculated for the methyl carbon atoms (Table 5). Since a number of calculated U_{ij} values of these atoms are larger than the corresponding observed ones riding motion of the atoms can be excluded. The positions of all hydrogen atoms obtained by the least-squares refinement, are well-defined. Moreover, all C–H distances (Table 9) are found in the range between 0·91 and 1·00 Å. Hence we may conclude that the presumed effect, if present, is very small and that more accurate diffraction data at different temperatures are needed to clarify this problem.

The thermal parameters of the 9-atom rigid body are listed in Table 7. The translational part of the motion is nearly isotropic, the principal axes T_1, T_2 and T_3 being nearly equal. The direction of the largest prin-

Table 5. *Observed and calculated* U_{ij} *values of non-hydrogen atoms in CBC at* 20°C

The units are 10^{-4} Å^2 and the crystallographic axes are the reference system. The temperature factor is defined as $\exp(-2\pi^2\sum_{ij} h_i h_j a_i^* a_j^* U_{ij})$.

	U_{11}		U_{22}		U_{33}		$2U_{12}$		$2U_{23}$		$2U_{13}$	
	obs	calc	obs	calc	obs	calc	obs	calc	obs	calc	obs	calc
Cl	1181	1206 (5)	516	541 (6)	1236	1237 (5)	182	136 (5)	481	500 (6)	602	608 (7)
O	1018	1013 (8)	873	882 (9)	603	611 (8)	−991	−989 (15)	190	266 (13)	74	73 (13)
C(1)	694	697 (12)	717	659 (11)	466	497 (10)	−445	−443 (17)	276	152 (16)	150	148 (16)
C(2)	716	689 (12)	483	517 (9)	575	543 (11)	−88	−143 (15)	198	212 (15)	133	62 (17)
C(3)	485	515 (10)	552	521 (9)	614	620 (10)	−92	−29 (15)	213	193 (15)	114	167 (15)
C(4)	526	497 (9)	511	495 (9)	416	450 (9)	−147	−131 (14)	92	119 (14)	6	−14 (14)
C(5)	513	472 (10)	588	635 (10)	738	750 (12)	−16	−23 (15)	294	378 (17)	153	85 (17)
C(6)	551	569 (11)	789	771 (12)	926	833 (14)	−181	−142 (18)	390	344 (21)	330	434 (19)
C(7)	582	613 (10)	573	580 (10)	574	557 (10)	−328	−342 (15)	249	218 (16)	−51	−53 (16)
C(8)	958	1066 (15)	578	531 (12)	1066	737 (17)	−381	−372 (21)	326	207 (21)	28	98 (24)
C(9)	830	962 (14)	936	732 (14)	648	548 (12)	−511	−693 (21)	544	254 (20)	88	124 (19)
C(10)	665	632 (13)	888	964 (14)	983	1075 (15)	−660	−683 (21)	495	675 (23)	−133	−239 (21)

cipal libration axis L_1 approximately coincides with the largest dimension of the molecule (Fig. 1) and has an orientation of 62, 28 and 95° with **a**, **b** and **c**, respectively (Fig. 4).

Table 7. *Rigid-body thermal parameters of CBC*

Origin defined at (fractional coordinates) $x=0{\cdot}1774$, $y=0{\cdot}5803$, $z=0{\cdot}2640$.
Axes of reference are the directions **a**, **c** × **a** and **c***. Estimated standard deviations are given in parenthesis. Consult formula (20) of Schomaker & Trueblood (1968) for reduced $\bar{\mathbf{T}}$.

Principal axes of reduced $\bar{\mathbf{T}}$

	r.m.s. amplitude (Å)	direction		cosines
T_1	0·23	−0·519	−0·824	0·228
T_2	0·21	0·826	−0·552	−0·116
T_3	0·20	0·222	0·728	0·967

Principal axes of $\bar{\mathbf{L}}$

	r.m.s. amplitude (°)	direction		cosines
L_1	7·8	0·471	0·836	−0·282
L_2	4·1	0·150	0·239	0·959
L_3	2·2	0·869	−0·494	−0·012

Tensor components of **T** (10^3 Å^2)

59·8 (1·3)	−0·2 (1·1)	−2·7 (1·3)
	51·3 (1·1)	0·6 (1·4)
		44·0 (2·1)

Tensor components of **L** (sg degree)

17·5 (1·6)	22·4 (1·3)	−10·4 (1·6)
	44·6 (4·4)	−5·7 (1·2)
		20·4 (1·1)

Displacements of libration axes from intersecting (Å)

Parallel to L_1	0·21	−0·32
Parallel to L_2	1·51	−0·25
Parallel to L_3	0·71	−0·43

Effective screw translation (Å)

Parallel to L_1	0·008
Parallel to L_2	0·021
Parallel to L	−0·048

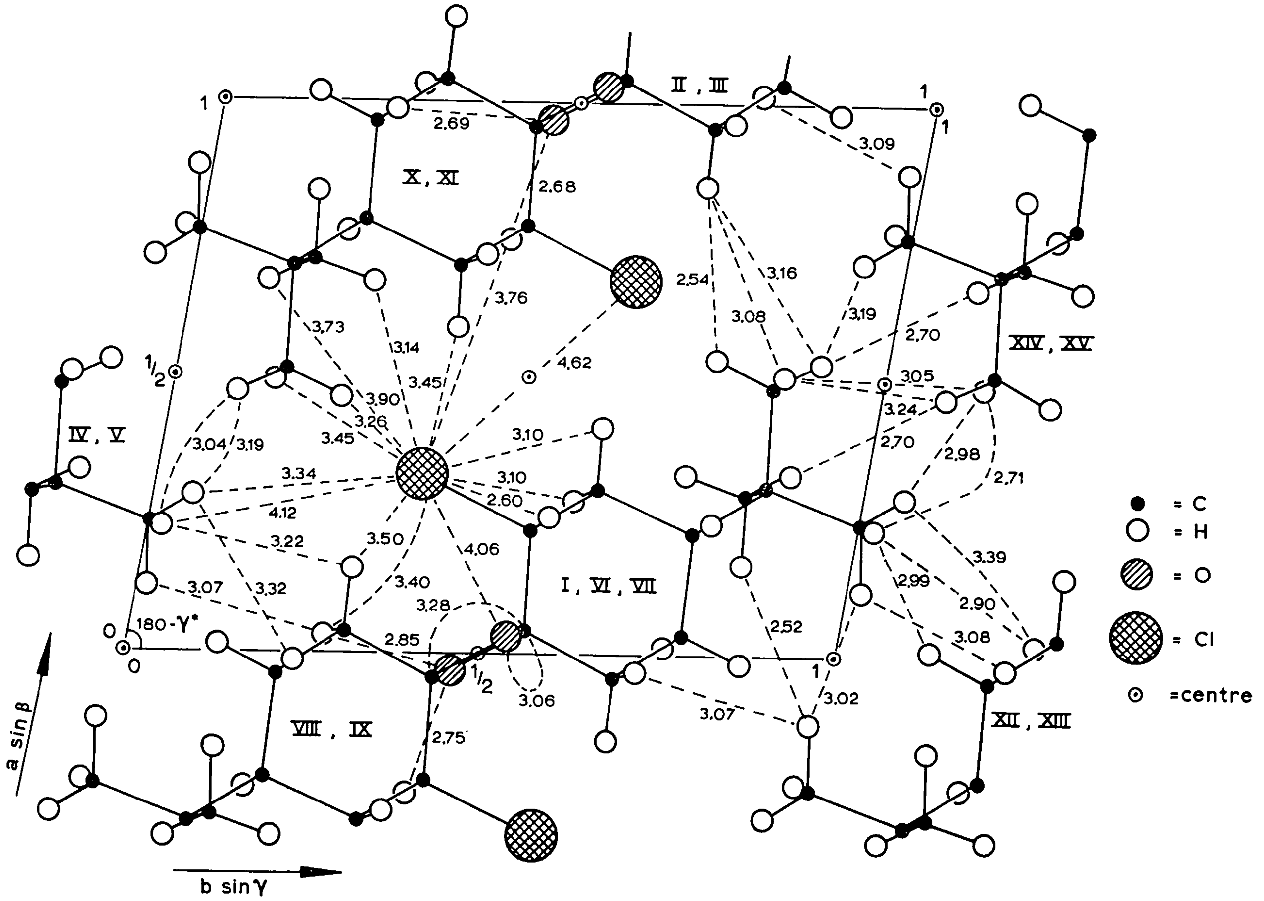


Fig. 4. Projection of the crystal structure along [001]. Dashed lines indicate intermolecular contacts. Roman numerals are explained in the next.

Molecular geometry

Taking into account the errors in cell edges as well as a 30% increase of positional standard errors the estimated standard deviations of bond lengths are 0·002 Å for C(2)–Cl, 0·004 Å for distances involving the methyl carbon atoms, 0·003 Å for all other heavy-atom distances and 0·03 Å for the C–H distances. The average standard deviations of valence and torsional angles not involving hydrogen atoms are 0·2 and 0·3°. Finally, the standard errors of valency angles involving hydrogen atoms are 2°. The bond distance, valency and endocyclic torsional angles are corrected for thermal motion according to a procedure proposed by Cruickshank (1956). We used a peak-breadth parameter of 0·10 Å for these corrections. It should be stated that the e.s.d.'s of the distances corrected for thermal motion are higher than those of the uncorrected distances. The extra uncertainty might be in the range of 0·001 to 0·003 Å, resulting in e.s.d.'s of 0·004 to 0·006 Å in the C–C bonds. Uncorrected and corrected bond lengths are listed in Table 8, the corrected distances, valency and torsional angles are also depicted in Fig. 1. Finally, bond lengths and angles involving hydrogen atoms are tabulated in Table 9.

Table 8. *Bond lengths* (Å) *in CBC*

u and *c* refer to values uncorrected and corrected for thermal motion

	l_u	l_c		l_u	l_c
C(1)–C(2)	1·505	1·520	C(2)–Cl	1·781	1·792
C(1)–C(6)	1·493	1·501	C(1)–O	1·207	1·213
C(2)–C(3)	1·523	1·529	C(4)–C(7)	1·551	1·554
C(5)–C(6)	1·525	1·530	C(7)–C(8)	1·527	1·536
C(3)–C(4)	1·530	1·542	C(7)–C(9)	1·528	1·541
C(4)–C(5)	1·521	1·534	C(7)–C(10)	1·521	1·533

Table 9. *Bond lengths and bond angles involving hydrogen atoms*

C–H		C–C–H		C–C–H	
2–21	0·97 Å	1–2–21	106·6°	7– 9– 92	110·2°
3–31	1·00	2–3–31	109·3	7– 9– 93	113·1
3–32	0·93	2–3–32	109·3	7–10–101	112·0
4–41	0·97	3–4–41	105·4	7–10–102	109·8
5–51	0·99	4–5–51	108·1	7–10–103	111·0
5–52	0·94	4–5–52	110·1	⟨C–C–H⟩	110
6–61	0·94	5–6–61	111·2	σ(C–C–H)	2
6–62	0·97	5–6–62	111·9	H–C–H	
8–81	0·95	3–2–21	111·9	31–3–32	106·8
8–82	0·94	4–3–31	110·0	51–5–52	105·9
8–83	0·98	4–3–32	110·2	61–6–62	105·7
9–91	0·97	5–4–41	106·9	81–8–82	111·2
9–92	0·97	7–4–41	105·5	81–8–83	103·5
9–93	0·94	6–5–51	110·9	82–8–83	107·9
10–101	0·99	6–5–52	109·3	91–9–92	108·0
10–102	0·92	1–6–61	109·3	91–9–93	109·2
10–103	0·95	1–6–62	106·2	92–9–93	106·3
⟨C–H⟩	0·96	7–8–81	112·1	101–10–102	104·9
σ(C–H)	0·02	7–8–82	109·4	101–10–103	109·4
		7–8–83	112·5	102–10–103	109·5
		7–9–91	111·7	⟨H–C–H⟩	107
				σ(H–C–H)	2

Accepting equivalence of all C–H distances their mean value (0·96 Å) agrees quite well with the values usually determined for this distance in X-ray investigations. The standard error (0·02 Å) is in accord with the least-squares prediction. The mean values of the C–C–H and H–C–H angles (110 and 107°) agree quite well with observations made by Portheine *et al.* (1972) (110 and 106°) and by Braun, Hornstra & Leenhouts (1969) (109 and 108°).

Inspection of the endocyclic torsional angles (Fig. 1) indicates the chair conformation of the ring. The mean absolute value (56·5°) shows that CBC is slightly more puckered than cyclohexane for which Geise, Buys & Mijlhoff (1972) report a value of 55·9°. The distribution of individual endocyclic valency and torsional angles suggests C_s symmetry of the ring skeleton. There is, however, a small difference in length between the bonds C(1)–C(6) and C(1)–C(2): $\Delta = 0{\cdot}019$ Å with e.s.d. $\sigma(\Delta) < 0{\cdot}008$ Å. This probably significant difference might be due to the different exocyclic bonds of C(2) and C(6). The lack in C_s symmetry of the ring is also reflected in the conformation of the $C(CH_3)_3$ group. In agreement with Altona & Sundaralingam's (1970) prediction the t-Bu group is indeed 'off-staggered' (see Fig. 2). The mean deviation from the ideally staggered position (−1·5°), although significant, is, however, rather small and in agreement with the small or absent extra libration effect (see previous section). Faber & Altona (1971) report values of 11·2 and 6·1° for the 'off-staggering' of t-Bu groups in the more asymmetrically substituted compound *cis-trans*-2,5-di-t-butylcyclohexanol tosylate. A Newman projection along the bond C(2)–C(1) is drawn in Fig. 3. It can be seen than the oxygen atom is nearly eclipsed with respect to the chlorine atom.

The valency angles hardly need comment. The mean value of endocyclic valency angles (111·3°) barely deviates from the values reported by Geise *et al.* (1972) for cyclohexane (111·1°) and methylcyclohexane (111·3°). Owing to the carbonyl group the valency angle C(6)–C(1)–C(2) (112·8°) is slightly larger than the average, while the opposing angle C(3)–C(4)–C(5) (108·7°), adjacent to the $C(CH_3)_3$ group is slightly smaller. In the tosylate compound the endocyclic valency angles adjacent to the $C(CH_3)_3$ groups are 108·4 and 109·1°.

The mean value of the bond lengths C(1)–C(6) and C(1)–C(2) is 1·510 Å and agrees with the accepted value (1·505 Å) for sp^3–sp^2 hybridization (Lide, 1962). The average of the four sp^3–sp^3 ring-bond distances (1·534 Å) can be compared with the recently determined value (1·528 Å) by Geise *et al.* for cyclohexane and methylcyclohexane. The mean distance of C(7) to the methyl carbon atoms (1·537 Å) does not differ significantly from the average sp^3–sp^3 ring distance. Its length may also be compared with the single bond C–C distance in open chains (1·533 Å) observed by Bartell & Kohl (1963).

The bond length of C(4)–C(7) (1·554 Å) differs significantly from the accepted open chain value. Its lengthening is due to overcrowding by the methyl groups. Dallinga & Toneman (1969) found values of

1·574 and 1·547 Å for this bond distance in 1*a*- and 1*e*-chloro-4-t-butylcyclohexane, while Faber & Altona (1971) report values of 1·543 and 1·549 Å. Bartell (1966) calculated a value of 1·573 Å for the central bond distance in hexamethylethane.

The packing

A projection of the structure along [001] is shown in Fig. 4. The reference molecule I (x,y,z) is surrounded at a distance less than 4·20 Å by fourteen molecules (II–XV) having the following coordinates.

$\pm1+x$	y	z	(II, III)
x	$\pm1+y$	z	(IV, V)
x	y	$\pm1+z$	(VI, VII)
$-x$	$1-y$	$-z$	(VIII)
$-x$	$1-y$	$1-z$	(IX)
$1-x$	$1-y$	$-z$	(X)
$1-x$	$1-y$	$1-z$	(XI)
$-x$	$2-y$	$-z$	(XII)
$-x$	$2-y$	$1-z$	(XIII)
$1-x$	$2-y$	$-z$	(XIV)
$1-x$	$2-y$	$1-z$	(XV)

The packing is governed by three types of interaction:

1. Dipole–dipole interactions between antiparallel carbonyl groups opposite to centres of symmetry at $0,\frac{1}{2},\frac{1}{2}$. We notice short distances for C(1)···O′ C(1)···C′(1) and O···O′ (3·06, 3·28 and 3·29 Å, respectively). The same phenomenon is observed in the packing of cyclohexane-1,4-dione (Mossel & Romers, 1964) where a C–O vector perpendicular to a screw axis interferes with its symmetry-related neighbour at corresponding distances for C–O′, O···O′ and C···C′ (3·12, 3·30 and 3·20 Å, respectively). Very short intermolecular distances in the range between 2·96 and 3·17 Å between carbon and carbonyl oxygen are also observed in the crystal structure of β-chloroglutaric anhydride (Koer *et al.* 1972).

2. Contacts between chlorine and hydrogen atoms. The chlorine atom is surrounded by 14 hydrogen atoms (three of which are intramolecular contacts) at a mean distance of 3·42 Å. The shortest intermolecular Cl···H contact being 3·14 Å, the largest 4·12 Å.

3. H···H interactions which are mainly formed in a range between 2·70 and 3·20 Å. The shortest H···H contacts are H(103)···H(61) (2·54 Å) and H(93)···H(81) (2·52 Å).

All calculations were performed on the IBM 360/50 computer of the Central Computing Laboratory of the University of Leiden. We acknowledge the assistance of Dr G. J. Verschoor during the measurements.

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