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The Structure of $C_9Cl_7(COOH).(O_2C_2H_4)$, a Condensed Cage Pentacyclononane Compound, Determined by the Symbolic Addition Method*

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The crystal structure of $C_9Cl_7(COOH).(O_2C_2H_4)$, a pentacyclononane derivative, was studied to obtain information on the configuration of the basic carbon cage structure which can be derived from a cube by adding one apex carbon atom. The crystal is monoclinic and the cell constants are: $a=17\cdot496\pm0\cdot004$, $b=8\cdot005\pm0\cdot003$, $c=24\cdot193\pm0\cdot004$ Å, $\beta=95\cdot6\pm0\cdot1°$; the space group is $I2/a$. Three-dimensional intensity data were collected on the CCXD, a computer-controlled diffractometer, which is operated by an IBM 1620 under a stored data-collection program. The crystal structure was determined by systematic application of the symbolic addition method and refined by a full-matrix least-squares program on an IBM 7094. The molecule can be called 6,6-ethylenedioxyheptachloropentacyclo-$[5.2.0.0^{2,5}.0^{3,9}.0^{4,8}]$nonane-3-carboxylic acid. The configurations of the cyclobutane and cyclopentane rings in the condensed cage molecule are discussed in detail. The carboxyl group is involved in the formation of centrosymmetric dimers which are stacked in the structure with the usual van der Waals contacts.

Introduction

Scherer, Lunt & Ungefug (1965) reported ring contraction through Favorskii rearrangement of chlorinated pentacyclodecanones by the action of alkali on I and III. In each case a pentacyclononane carboxylic acid has been formed (see Fig. 1). The position of the carboxyl group in IV could be uniquely assigned since the starting decanone possesses a symmetric cage structure; whereas for II the choice between the two non-equivalent sites for the group was decided by measuring the *pKa* value of the acid V obtained by a hydrolysis of II which effected a removal of the ethylene glycol residue.

Crystals of II and IV were studied as a part of our continuing effort to determine the shape and size of condensed poly-ring carbon cage structures. Although IV is a simpler molecule than II, the crystallographic data revealed that there are two non-equivalent molecules per asymmetric unit. This led to determination of the crystal structure of II, which possesses an extra ethylenedioxy group on the apex carbon.

Experimental

Crystals of II, $C_9Cl_7(COOH)(O_2C_2H_4)$, were obtained from CH_2Cl_2 solution of the sample supplied by Professor K. Scherer. One such crystal was ground into an approximately spherical shape and mounted on a G.E. Goniostat on CCXD, a computer-controlled diffractometer system, operated by an IBM 1620 in a closed-loop manner (Cole, Okaya & Chambers, 1963). The crystallographic constants were measured on the diffractometer and used as input parameters to the subsequent data-collection program; the values were $a=17\cdot496\pm0\cdot004$, $b=8\cdot005\pm0\cdot003$, $c=24\cdot193\pm0\cdot004$ Å,

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* The experimental part of this work was performed at IBM Research Center, Yorktown Heights, New York.

$\beta = 95{\cdot}6 \pm 0{\cdot}1°$. The apparent space group for this choice of axes was *I*2/*a* or *Ia*. This unconventional choice of axes (resulting in a body-centered monoclinic cell) was employed to make the β angle close to 90°. A base-centered lattice may be obtained by a simple linear transformation of axes; *e.g.* $a = 17{\cdot}496 \pm 0{\cdot}004$, $b = 8{\cdot}005 \pm 0{\cdot}003$, $c = 28{\cdot}441 \pm 0{\cdot}004$ Å, $\beta = 122{\cdot}2 \pm 0{\cdot}1_0$, with space group *A*2/*a* or *Aa*. There are eight formula units with observed density of 1·809 g.cm^{-3}, as compared to the calculated 1·815 g.cm^{-3}.

Three-dimensional intensity data were taken with Mo $K\alpha$. For each reflection the alignment of the specimen and the general accuracy of the system were tested first by optimizing the ω setting. The integrated intensity data were then recorded by making (θ–2θ) step scans; the number of steps was twenty-four. The intervals of the 2θ step scan varied according to the 2θ values; they are 0·09° for 2θ less than 40° and 0·11° thereafter. These intervals had been determined in such a way that the first three and the last three of the 24 steps represent the background at the 2θ value. In the course of optimizing the ω setting, the maximum and minimum counts were recorded; when the difference was smaller than the statistical fluctuation of the system, such a reflection was treated as unobserved. The data-collection program written in the 1620 SPS (Symbolic Programming System) language is exexplained elsewhere (Okaya, 1964).

Structure determination

The structure was solved by the symbolic addition method (Karle & Karle, 1963). An absolute scale factor and overall temperature factor were determined by a Wilson statistical analysis of the three-dimensional data and the normalized structure-factor magnitudes were then obtained. These were used in the phase determination by the direct method.

The ambiguity in the space group assignment was first resolved by a statistical analysis of the magnitudes of the *E* factors (Table 1), which shows beyond reasonable doubt the presence of a center of symmetry. The space group is thus determined as *I*2/*a* (or *A*2/*a* for the base centered transformed cell described above). The following set of equivalent positions for *I*2/*a* was used in the analysis; (8); (0,0,0) $(\frac{1}{2},\frac{1}{2},\frac{1}{2}) \pm (x,y,z;\ \frac{1}{2}+x,\frac{1}{2}-y,z)$.

Table 1. *Space group assignment by E factor statistics*

	Observed	Calculated for:* Centro	Non-centro
$\langle E^2\rangle$	1·0	1·0	1·0
$\langle \lvert E\rvert\rangle$	0·769	0·798	0·886
$\langle E^2-1\rangle$	0·974	0·968	0·736
$\lvert E\rvert > 3{\cdot}0$	0·3%	0·3%	0·01%
$\lvert E\rvert > 2{\cdot}0$	4·8%	5·0%	1·8%

* Karle, Dragonette & Brenner (1965).

The phases of the larger *E* factors were systematically studied by the use of a program written in FORTRAN IV, *SORTE* (Bednowitz & Post, 1966), on an IBM 7094. After a number of iterative cycles, with application of the symbolic addition method, about 150 *E* factors were given with reasonably certain signs. These phases were then used in evaluating a three-dimensional *E* factor map, which was then scanned by a peak-searching program to obtain the positions of seven large peaks. Starting from these seven peaks the positions of other atoms were obtained by iterative structure-factor and electron density evaluations. After the sixteen remaining light atoms had been found in the electron density function, a view of the structure was drawn on an IBM 1627 XY-plotter based on a structure drawing program for an IBM 7094. The overall molecular shape of the compound is shown in Fig. 2.

The atomic coordinates were then refined by least-squares with anisotropic thermal parameters to represent the thermal vibration. After several cycles with a full matrix program, the error index, $R = \Sigma\,\lvert\lvert F_o\rvert - \lvert F_c\rvert\rvert / \Sigma\lvert F_o\rvert$ was reduced to 0·056 (or 0·068 if all acciden-

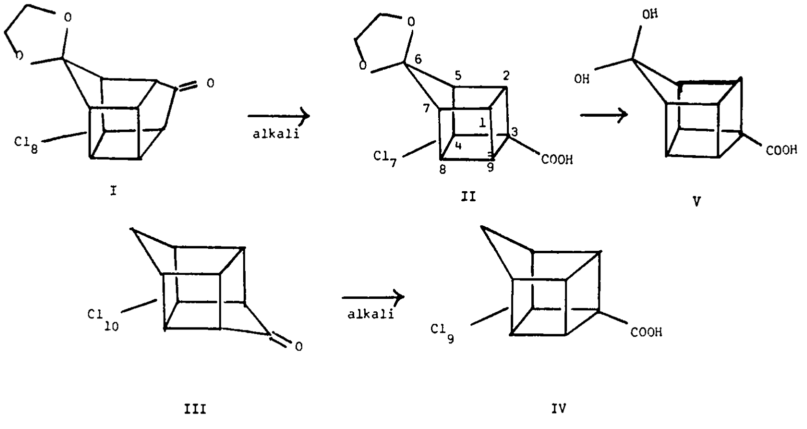


Fig. 1. Syntheses of the pentacyclononane acids studied in the present work. The numbering of atoms in the cage structure is given for molecule II.

tally absent reflections were included as $F_{\text{obs}} \equiv 0$) for about 1800 reflections. The atomic coordinates, their standard deviations and anisotropic thermal parameters are shown in Table 2. The atomic scattering factors were those listed in *International Tables for X-ray Crystallography* (1962). The weighting scheme used in the least-squares refinement was: $\omega = 1{\cdot}0$ for $|F_{\text{obs}}| \leq 50{\cdot}0$ and $50{\cdot}0/|F_{\text{obs}}|$ for larger F values; the unobserved reflections were given weights of zero. Shifts of atomic coordinates at the last cycle of the refinement were negligible compared to their standard deviations. The positions of hydrogen atoms were not studied. Comparison between the observed and calculated structure factors is given in Table 3.

Discussion

The direct determination of the structure of the acid, II, led to the unambiguous solution of the configuration of the molecule. The molecule can be called 6,6-ethylenedioxyheptachloropentacyclo[5.2.0.0^{2,5}.0^{3,9}.0^{4,8}]-nonane-3-carboxylic acid. The observed position of the carboxyl group substantiates the earlier chemical evidence based on the acidity measurement. In Fig. 1, the numbering of atoms based on the pentacyclo system is shown. The basic cage structure has an approximate mirror plane going through carbon atoms 6,4,3, 9,7, a diagonal of this homocubane molecule, and this accounts for the inability to resolve the acid into optical isomers.

As shown in Fig. 3, this molecule belongs to a series of pentacyclo condensed cage compounds obtained by adding apex points to a cube. The configuration of the starting cube, cubane, C_8H_8 [Fig. 3(*a*)] has been studied

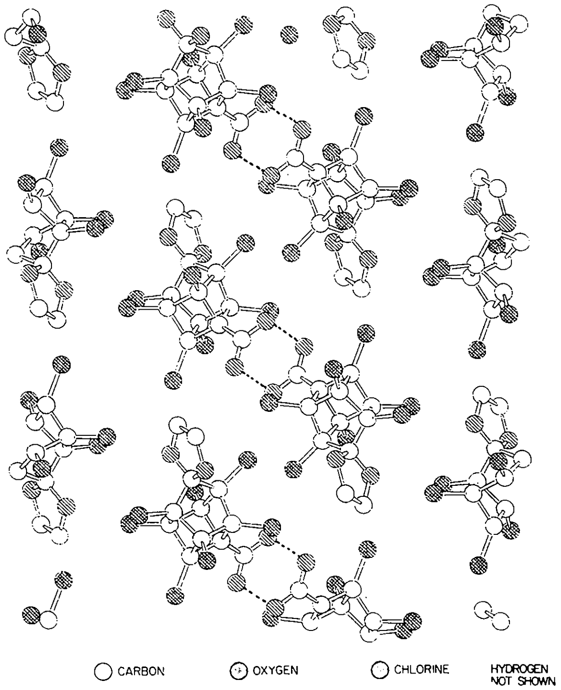
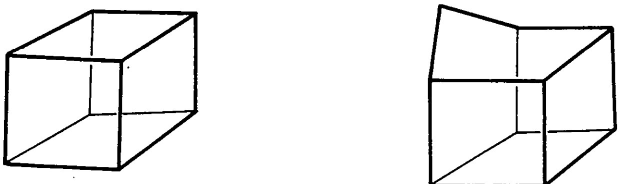


Fig. 2. Projection of the structure along the *b* axis. The O–H---O hydrogen bonds involved in the usual carboxyl dimer formation are shown by broken lines. The Figure was drawn on an IBM 1627 XY plotter based on a structure drawing program for the IBM 7094 (Okaya, 1968). The atomic coordinates and the peak heights, which were used in deciding proper shades for the atoms, are those obtained from electron density maps before the refinement stage.



(a) C_8H_8

Pentacyclo-[4. 2. 0. 0^{2,5}. 0^{3,8}. 0^{4,7}]-octane

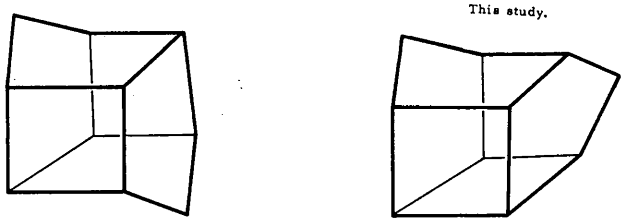
C_8H_8, cubane; Fleischer (1964).

(b) C_9H_{10}

Pentacyclo-[5. 2. 0. 0^{2,5}. 0^{3,9}. 0^{4,8}]-nonane

$C_9Cl_7(O_2C_2H_4)(COOH)$;

This study.



(c) sym-$C_{10}H_{12}$

Pentacyclo-[5. 3. 0. 0^{2,6}. 0^{3,9}. 0^{4,8}]-decane

$C_{10}Cl_{11}OSO_2Cl$; Okaya and Bednowitz (1967).

(d) gauche-$C_{10}H_{12}$

Pentacyclo-[5. 3. 0. 0^{2,6}. 0^{3,10}. 0^{4,8}]-decane.

Fig. 3. The pentacyclo-cage systems discussed in the text.

by Fleischer (1964). The various crystallographic studies were made on symmetric decane derivatives, [Fig. 3(*c*)]; *e.g.* the configuration of the cage was determined by a crystal structure analysis of its undecachlorochlorosulfonate derivative, $C_{10}Cl_{11}OSO_2Cl$ (Okaya & Bednowitz, 1967).

The bond distances are shown in Fig. 4. It is obvious from the data that the formation of a condensed system has little effect on the bond distances; all the C–C and C–Cl bonds exhibit normal single-bond separations. By contrast, the bond angles and the configurations of the rings show more interesting features. Fig. 6 lists the bond angles in the ring system and Fig. 7 shows data on planarity of various groups in the cage. The two cyclobutane rings, 1–2–3–9, and 8–4–3–9, which have the apex carbon 6, only as a next nearest neighbor,

Table 2(*a*). *Atomic coordinates* (*in fractions of cell edges*) *and their standard deviations* (*in* 10^{-4} Å)

		x	y	z
Chlorine atoms	Cl(1)	0·14580 (24)	0·23967 (32)	0·23219 (24)
	Cl(2)	0·20534 (23)	0·43094 (24)	0·12481 (29)
	Cl(4)	0·10473 (22)	−0·04770 (25)	−0·00156 (23)
	Cl(5)	0·26656 (20)	0·15070 (26)	0·03736 (24)
	Cl(7)	0·16370 (24)	−0·17277 (31)	0·21669 (25)
	Cl(8)	0·04265 (21)	−0·23852 (24)	0·10524 (28)
	Cl(9)	−0·03155 (21)	0·12702 (27)	0·15871 (26)
Oxygen atoms				
Carboxyl	O(1)	0·08332 (52)	0·37560 (62)	0·00538 (60)
	O(2)	−0·01518 (46)	0·39473 (58)	0·05737 (53)
Ethylenedioxy	O(3)	0·25272 (48)	−0·18603 (56)	0·10761 (55)
	O(4)	0·29916 (49)	0·02115 (60)	0·16384 (56)
Carbon atoms				
	C(1)	0·13645 (76)	0·14458 (79)	0·16792 (78)
	C(2)	0·16586 (68)	0·23558 (83)	0·11676 (85)
	C(3)	0·08539 (67)	0·20320 (82)	0·08331 (77)
	C(4)	0·12228 (70)	0·03731 (79)	0·06418 (76)
	C(5)	0·20521 (70)	0·09472 (81)	0·08584 (76)
	C(6)	0·23479 (70)	−0·03389 (82)	0·12992 (79)
	C(7)	0·16233 (78)	−0·03740 (90)	0·16007 (81)
	C(8)	0·09283 (69)	−0·05300 (81)	0·11514 (83)
	C(9)	0·05655 (72)	0·11093 (78)	0·13372 (76)
	C carb	0·04622 (63)	0·33378 (79)	0·04571 (77)
	C ethy(1)	0·33294 (76)	−0·22833 (82)	0·12270 (109)
	C ethy(2)	0·36530 (74)	−0·07662 (86)	0·15315 (101)

Table 2(*b*). *Anisotropic temperature factors used in the expression*

$$\exp\left[-\{\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl\}\right].$$

		β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Chlorine atoms							
	Cl(1)	0·00541	0·03744	0·00180	0·00307	−0·00013	−0·00587
	Cl(2)	0·00470	0·01449	0·00417	−0·00390	−0·00039	−0·00371
	Cl(4)	0·00419	0·02269	0·00179	0·00329	−0·00066	−0·00404
	Cl(5)	0·00345	0·02416	0·00249	0·00048	0·00183	0·00351
	Cl(7)	0·00511	0·03483	0·00234	0·00589	0·00066	0·00979
	Cl(8)	0·00353	0·01450	0·00394	−0·00259	0·00006	0·00209
	Cl(9)	0·00313	0·02730	0·00291	0·00366	0·00225	0·00175
Oxygen atoms							
Carboxyl	O(1)	0·00434	0·02334	0·00275	0·00623	0·00292	0·00520
	O(2)	0·00307	0·02049	0·00206	0·00567	0·00059	0·00064
Ethylenedioxy	O(3)	0·00367	0·01528	0·00236	0·00310	−0·00061	−0·00118
	O(4)	0·00337	0·02161	0·00237	0·00080	−0·00171	−0·00321
Carbon atoms							
	C(1)	0·00332	0·02114	0·00144	0·00186	0·00059	−0·00246
	C(2)	0·00221	0·01414	0·00214	−0·00182	0·00001	−0·00156
	C(3)	0·00213	0·01241	0·00168	0·00160	0·00063	−0·00022
	C(4)	0·00254	0·01480	0·00117	0·00137	0·00047	0·00074
	C(5)	0·00256	0·01512	0·00139	0·00084	0·00106	0·00099
	C(6)	0·00234	0·01284	0·00197	0·00009	0·00034	0·00036
	C(7)	0·00321	0·01583	0·00170	0·00120	0·00029	0·00338
	C(8)	0·00213	0·01421	0·00206	−0·00084	−0·00084	0·00150
	C(9)	0·00302	0·01724	0·00138	0·00149	0·00096	0·00013
	C carb	0·00165	0·01380	0·00190	0·00093	−0·00029	−0·00278
	C ethy(1)	0·00237	0·02452	0·00402	0·00518	−0·00037	−0·00200
	C ethy(2)	0·00223	0·02295	0·00338	0·00518	−0·00097	−0·00167

Table 3. *Comparison of observed and calculated structure factors* (×10)

```
      FO  FC
H,K=  0,  0
L=2 697 658
  412981217
  6 358 326
  814351381
 10  53  68
 12   0  14
 14 184 170
 16 127 143
 18 746 749
 20   0  12
 22 145 134

H,K=  0,  1
L=1 530 592
  3 246 276
  5 737 690
  7 957 974
  9 819 811
 11 879 832
 13   0  61
 15  72  60
 17  47  56
 19   0  53
 21 258 246

H,K=  0,  2
L=0 374 386
  2 665 683
  4 179 228
  614041388
  8  35  39
 10 301 272
 12 475 446
 14 448 432
 16 464 438
 18   0   1
 20   0  83

H,K=  0,  3
L=1 526 543
  312471262
  5 393 402
  7  97 110
  9   0  12
 11 244 251
 13  46  35
 15   0  63
 17   0  22
 19   0  58

H,K=  0,  4
L=0  44  47
  2 278 280
  4 273 280
  6 502 495
  8 376 352
 10 493 477
 12   0  55
 14  49  90
 16 179 189
 18 143 120

H,K=  0,  5
L=1  51  44
  3 448 464
  5 200 198
  7 374 374
  9 122 127
 11   0  11
 13 188 194
 15  95 109

H,K=  0,  6
L=0 255 262
  2   0   9
  4 392 386
  6 180 173
  8 379 380
 10 161 168
 12   0  28

H,K=  0,  7
L=1 145 145
  3 104 129
  5 106 113
  7   0  33

H,K=  1,  1
L=0 938 974
  212611201
  4  83 104
  6   0  14
  8 177 183
 10 384 370
 12 576 558
 14   0  11
 16 149 158
 18 333 308
 20   0  31
 22 131 131

H,K=  1,  2
L=1   0  34
  3 643 641
  5 973 924
  7 372 377
  9 858 815
 11 455 457
 13   0  76
 15 124 101
 17 175 155
 19 326 314
 21  31  54

H,K=  1,  3
L=0 175 167
  212091209
  4 869 855
  6 457 436
  8 287 277
 10 668 650
 12 120 114
 14  72  60

      FO  FC
H,K=  1,  3
 16 225 195
 18  69  29
 20  55  80

H,K=  1,  4
L=1 447 457
  3 172 184
  5 173 177
  7 475 478
  9  90  75
 11 232 191
 13  99 102
 15   0   2
 17 280 242

H,K=  1,  5
L=0 230 221
  2 322 283
  4 221 225
  6  60  39
  8  26  80
 10   0  15
 12   0   0
 14 133 131
 16 138 138

H,K=  1,  6
L=1 491 487
  3  56  18
  5 416 400
  7 154 157
  9 274 249
 11   0  37
 13 179 161

H,K=  1,  7
L=0  88  95
  2 203 224
  4  83  62
  6   0  67

H,K=  2,  0
L=0 668 661
  2 9611009
  4  24  45
  6  39  41
  8 986 948
 10 690 674
 12 952 893
 14 104 124
 16 425 408
 18  70  89
 20 328 283
 22   0  54

H,K=  2,  1
L=1 553 471
  3 516 499
  5 682 665
  7 395 396
  9 133 102
 11 596 580
 13  74  39
 15  46  79
 17 241 244
 19 253 260
 21 223 231

H,K=  2,  2
L=0 358 376
  2 266 244
  4 466 502
  6 434 423
  8 578 558
 10 271 247
 12 273 262
 14 533 508
 16  97 103
 18   0  31
 20   0   5

H,K=  2,  3
L=1 138 170
  3 182 163
  5 431 429
  7   0  28
  9 250 230
 11 143 138
 13 328 333
 15 184 152
 17 423 413
 19  70  52

H,K=  2,  4
L=0 125 128
  2  56  38
  4   0  33
  6 404 399
  8 298 279
 10 313 308
 12  62  75
 14 232 200
 16  81  54
 18   0  71

H,K=  2,  5
L=1 603 615
  3 493 469
  5 159 150
  7   0   5
  9   0  47
 11 528 519
 13  53  53
 15  78  97

H,K=  2,  6
L=0 198 194
  2 108  81
  4   0  73
  6 131 110
  8   0   9
 10   0  75
 12 145 143

      FO  FC
H,K=  2,  7
L=1   0   9
  3  95 110
  5 214 200
  7 317 318

H,K=  3,  1
L=0 113  96
  2 482 428
  4 929 896
  6 966 987
  8 734 738
 10 266 263
 12   0  50
 14   0  73
 16  92 118
 18 308 311
 20   0  78

H,K=  3,  2
L=1 149 139
  311931163
  514791393
  7 319 314
  9 447 420
 11   0  49
 13 308 308
 15 411 422
 17 118  94
 19 129 145
 21 172 169

H,K=  3,  3
L=0 152 133
  2 698 677
  4 172 144
  6 494 528
  8 294 289
 10 127 117
 12 290 268
 14 147 163
 16 143 116
 18 273 248
 20  74  74

H,K=  3,  4
L=1  90  83
  3 191 187
  5 583 577
  7 436 404
  9  63  55
 11 459 472
 13 175 201
 15 223 236
 17   0  18

H,K=  3,  5
L=0 225 203
  2 344 333
  4   0  39
  6 319 307
  8   0  22
 10 216 221
 12  90  80
 14 301 286
 16   0  25

H,K=  3,  6
L=1 104  66
  3   0  44
  5 136 157
  7   0  22
  9 186 183
 11   0  38

H,K=  3,  7
L=0  72  66
  2 267 266
  4 207 204
  6   0  11

H,K=  4,  0
L=0 163 100
  2 846 861
  423202337
  6   0  11
  8 773 745
 10 273 261
 1211691156
 14 496 487
 16 191 153
 18 141 171
 20 186 159

H,K=  4,  1
L=1 471 482
  31057 994
  5 835 846
  7 567 581
  9  97  82
 11 188 194
 13 313 312
 15 299 282
 17 340 349
 19 136 143
 21  72  31

H,K=  4,  2
L=0 117 113
  2 402 442
  4 730 720
  6 972 928
  8   0  32
 10 282 272
 12  24  48
 14  76  80
 16  86  82
 18 186 176
 20 329 323

H,K=  4,  3
L=1 787 747
  3 315 334
  5  67  81

      FO  FC
H,K=  4,  3
  7 406 389
  9 471 475
 11 164 169
 13   0  30
 15 296 291
 17 141 119
 19  72  27

H,K=  4,  4
L=0 596 592
  2 588 573
  4 163 156
  6 269 257
  8 180 185
 10 493 482
 12   0  56
 14 164 165
 16  62  84
 18  46  76

H,K=  4,  5
L=1 745 738
  3 115 123
  5 203 208
  7  39  68
  9 743 727
 11   0  88
 13 193 194
 15   0  40

H,K=  4,  6
L=0   0  79
  2   0   0
  4  53  59
  6   0  60
  8 117 114
 10 101  68
 12   0  74

H,K=  4,  7
L=1 202 190
  3 285 278
  5   0  46

H,K=  5,  1
L=010091040
  2 454 436
  4 639 612
  6 104  91
  8 138 160
 10 368 341
 12 237 234
 14 235 242
 16 425 434
 18 308 296
 20  79 104

H,K=  5,  2
L=1 450 408
  3  81  78
  5   0  40
  7 510 474
  9  83 109
 11 374 388
 13 255 248
 15 170 170
 17 177 162
 19 115 100

H,K=  5,  3
L=0 455 487
  2   0  41
  4 562 567
  6 447 422
  8 196 206
 10 333 323
 12   0  21
 14 305 311
 16 150 112
 18 273 284

H,K=  5,  4
L=1   0  76
  3 227 217
  5 164 185
  7 278 267
  9 280 255
 11 255 242
 13 154 168
 15   0  29
 17   0  47

H,K=  5,  5
L=0  94  46
  2 202 192
  4 101 104
  6 228 213
  8  65  33
 10  85  31
 12 335 363
 14 122  90

H,K=  5,  6
L=1 257 263
  3 223 206
  5 349 343
  7 129 104
  9 127 124
 11 177 188

H,K=  5,  7
L=0 170 177
  2   0  50

H,K=  6,  0
L=0 108 113
  219151882
  4 734 722
  6 762 748
  8 161 137
 10 934 905
 12 523 539
 14 562 552

      FO  FC
H,K=  6,  0
 16 569 549
 18 209 164
 20   0  78

H,K=  6,  1
L=1 372 374
  3 450 440
  5 274 267
  7 275 288
  9 161 147
 11  79 121
 13 101  68
 15 216 210
 17   0  74
 19  44  89

H,K=  6,  2
L=0 487 483
  2 405 407
  4  53  79
  6  88 105
  8 487 490
 10 636 624
 12 360 348
 14 223 254
 16 234 242
 18 326 306

H,K=  6,  3
L=1 333 331
  3 234 231
  5 558 551
  7 131 131
  9 619 624
 11   0  47
 13 200 215
 15   0  14
 17 248 254

H,K=  6,  4
L=0 196 195
  2 179 160
  4   0  19
  6 170 161
  8   0  42
 10   0  35
 12 349 351
 14 177 174
 16  46  49

H,K=  6,  5
L=1 335 301
  3 117  81
  5 439 451
  7 205 176
  9 129 113
 11 227 251
 13 113 116

H,K=  6,  6
L=0 109 125
  2 244 246
  4 106  99
  6 170 149
  8   0  33
 10 143 118

H,K=  7,  1
L=0 551 521
  2 349 332
  4 253 247
  6  23  62
  8 257 257
 10  51  35
 12  26   7
 14 253 254
 16 133 159
 18  85  87

H,K=  7,  2
L=1 106 104
  3 615 625
  5 454 459
  7 406 395
  9 805 781
 11 434 432
 13 134 143
 15 134 124
 17 159 185

H,K=  7,  3
L=0 290 296
  2 161 158
  4 434 437
  6 191 189
  8 587 575
 10 218 224
 12   0  21
 14 187 169
 16 175 181

H,K=  7,  4
L=1 615 602
  3 267 267
  5 108  76
  7 168 172
  9 159 144
 11 225 201
 13   0  40
 15 156 153

H,K=  7,  5
L=0 136 136
  2 157 154
  4 360 347
  6   0  19
  8 207 234
 10 108 127
 12   0   5

H,K=  7,  6
L=1 188 174
  3 129 139

      FO  FC
H,K=  7,  6
  5  90 132
  7   0  39

H,K=  8,  0
L=0  62  64
  2 454 429
  4 768 745
  6 720 704
  8 147 172
 10 565 586
 12  90 107
 14 223 219
 16 349 394
 18 172 201

H,K=  8,  1
L=1   0   0
  3 212 218
  5 596 614
  7 230 231
  9  44  18
 11 313 299
 13   0  69
 15  85  76
 17  70  86

H,K=  8,  2
L=0  99 111
  2 892 900
  4 131 140
  6   0  57
  8 427 431
 10 713 712
 12   0   8
 14   0  50
 16   0  58
 18   0  56

H,K=  8,  3
L=1 319 306
  3 228 218
  5 337 337
  7 337 323
  9   0  14
 11 250 268
 13 118 128
 15  31  61

H,K=  8,  4
L=0  90  90
  2 509 474
  4 379 388
  6 124 150
  8 180 162
 10  51  79
 12   0  30
 14   0  51

H,K=  8,  5
L=1   0  69
  3   0  17
  5 441 410
  7   0  18
  9   0  75
 11   0   6

H,K=  8,  6
L=0 182 175
  2   0  25
  4 250 239
  6 163 155

H,K=  9,  1
L=0 266 237
  2 241 217
  4   0  32
  6   0  36
  8 127 141
 10  60  47
 12 227 259
 14 344 340
 16 234 239

H,K=  9,  2
L=1 133 118
  3 133 145
  5 338 329
  7   0  51
  9 147 142
 11   0 142
 13 141 132
 15 290 315

H,K=  9,  3
L=0 494 484
  2 170 156
  4 294 296
  6  72  90
  8 124 118
 10 203 215
 12   0  31
 14 173 157

H,K=  9,  4
L=1  62  72
  3  39  26
  5 145 160
  7 125 138
  9 276 299
 11   0 111
 13   0  28

H,K=  9,  5
L=0 124 101
  2   0  57
  4   0  38
  6  99 111
  8 166 205

H,K= 10,  0
L=0 484 483
  2 858 859
  4   0   9

      FO  FC
H,K= 10,  0
  6 718 704
  8 322 344
 10 280 280
 12 221 242
 14 407 423
 16 315 367

H,K= 10,  1
L=1 358 379
  3 298 282
  5   0  15
  7 560 565
  9 207 204
 11 147 171
 13 125 137
 15 115  89

H,K= 10,  2
L=010691077
  2   0  59
  4 113 126
  6 280 279
  8 431 447
 10 255 271
 12   0   5
 14   0  55

H,K= 10,  3
L=1 122 142
  3 337 351
  5 260 255
  7   0  23
  9 278 289
 11 124 140
 13 189 181

H,K= 10,  4
L=0   0  69
  2 219 198
  4   0  27
  6 234 215
  8 200 206
 10 260 263

H,K= 10,  5
L=1 289 271
  3 399 401
  5  31  68
  7 141 117

H,K= 11,  1
L=0   0  67
  2 473 468
  4   0   7
  6 214 219
  8 159 144
 10 413 452
 12   0  43
 14   0  45

H,K= 11,  2
L=1 345 355
  3 200 224
  5 282 296
  7 363 360
  9   0  53
 11 179 174
 13   0  18

H,K= 11,  3
L=0  69  96
  2 404 399
  4  90  66
  6   0  60
  8 179 172
 10   0  87
 12 285 312

H,K= 11,  4
L=1 246 220
  3 234 250
  5 180 143
  7 203 198
  9 404 407

H,K= 11,  5
L=0   0  51
  2   0  66

H,K= 12,  0
L=0 177 173
  2 195 213
  4   0  71
  6  95 130
  8   0  35
 10 173 190
 12 392 421

H,K= 12,  1
L=1 267 301
  3 354 373
  5   0  29
  7 172 187
  9 113 140
 11  31  60
 13   0  79

H,K= 12,  2
L=0 246 241
  2   0  27
  4 173 146
  6  72  67
  8 173 185
 10  99  92
 12  86 126

H,K= 12,  3
L=1 539 541
  3 340 344
  5 173 159
  7  76  82
  9   0  19

      FO  FC
H,K= 12,  4
L=0   0   6
  2 120 139
  4 161 155
  6 218 221

H,K= 13,  1
L=0 161 169
  2 349 356
  4 360 359
  6   0  54
  8 189 175
 10 223 270

H,K= 13,  2
L=1 294 261
  3 115 109
  5 198 194
  7 290 271
  9 218 237

H,K= 13,  3
L=0 198 210
  2   0  53
  4  83  96
  6 292 309

H,K= 14,  0
L=0 282 270
  2   0  53
  4 459 470
  6  53  95
  8   0  48

H,K= 14,  1
L=1  74  61
  3   0  37
  5 124 150
  7 292 281

H,K= 14,  2
L=0  95  68
  2   0  98
  4 376 353

H,K= 16,  0
L=0 393 376

H,K= -1,  1
L=2 879 856
  4 209 210
  6 769 844
  8 541 553
 10   0  45
 12 202 217
 14 486 488
 16 180 168
 18   0   7
 20   0  29
 22 122 114

H,K= -1,  2
L=1 517 512
  3 800 789
  5 821 855
  7 532 552
  9 910 938
 11 505 524
 13 448 460
 15 484 475
 17 258 258
 19 172 162
 21   0  60

H,K= -1,  3
L=2 500 503
  4 241 220
  6 333 317
  8 390 399
 10 374 398
 12 216 230
 14   0  44
 16 427 414
 18 143 161
 20 195 185

H,K= -1,  4
L=1 368 377
  3   0  25
  5 140 123
  7 704 704
  9  76  45
 11   0  15
 13 195 190
 15 115 134
 17  62  77

H,K= -1,  5
L=2 539 533
  4   0   5
  6 368 360
  8  39  54
 10 298 293
 12   0  58
 14  88  79
 16  46   7

H,K= -1,  6
L=1  86 101
  3  76  97
  5 313 313
  7 203 199
  9 143 151
 11   0  40
 13 159 175

H,K= -1,  7
L=2  62  75
  4   0  84
  6  90 119

H,K= -2,  0
L=218512076
  4 184 144

      FO  FC
H,K= -2,  0
  6 519 503
  815141645
 10 759 794
 12 399 416
 14 381 404
 16   0  23
 18 390 406
 20 280 316
 22 125 127

H,K= -2,  1
L=1 152 125
  3  97 182
  5  49  68
  7 237 246
  9 214 192
 11 218 202
 13 445 455
 15 301 310
 17 228 260
 19 478 476
 21   0  32

H,K= -2,  2
L=2 209 220
  412381278
  6 526 515
  8 423 440
 10 264 272
 12  83  47
 14 299 318
 16 152 165
 18   0  67
 20   0  57

H,K= -2,  3
L=1 305 322
  3 159 156
  5 379 417
  7  30  66
  9 290 291
 11 342 324
 13 381 384
 15 143 162
 17 129 130
 19   0  70

H,K= -2,  4
L=2 510 538
  4 211 211
  6 459 461
  8 154 162
 10 356 366
 12 230 252
 14 195 191
 16 306 314
 18 189 168

H,K= -2,  5
L=1 255 266
  3   0  68
  5 381 390
  7 773 784
  9 124 103
 11 404 420
 13   0  32
 15 141 131

H,K= -2,  6
L=2 289 279
  4 168 148
  6 235 217
  8 140 137
 10 214 203
 12  31  90

H,K= -2,  7
L=1 138 170
  3 118  79
  5 246 277
  7  72  77

H,K= -3,  1
L=2 230 256
  4 972 990
  6   0  13
  8 120 137
 10 218 249
 12 273 293
 14 519 543
 16 120 114
 18   0  26
 20  53  20

H,K= -3,  2
L=113211327
  3 425 467
  5 404 382
  7 400 398
  9 223 218
 11 353 363
 13   0   0
 15 115 113
 17 266 266
 19 289 287
 21   0  35

H,K= -3,  3
L=2 189 179
  4 686 697
  6   0  62
  8  90 115
 10 255 251
 12  78  76
 14   0  60
 16 246 274
 18 109 144
 20   0 102

H,K= -3,  4
L=1 212 200
  3 351 379
  5 289 296

      FO  FC
H,K= -3,  4
  7 157 147
  9 594 581
 11 523 525
 13 494 511
 15 243 236
 17 157 141

H,K= -3,  5
L=2 108  83
  4   0  41
  6  53  37
  8 237 252
 10  40  78
 12  79  77
 14  53  71
 16   0  77

H,K= -3,  6
L=1  81 118
  3   0  21
  5  58  74
  7 164 156
  9   0   7
 11 127 129

H,K= -3,  7
L=2 250 244
  4   0  47
  6   0  79

H,K= -4,  0
L=2 635 616
  4 260 281
  6 392 381
  8 205 223
 10 743 737
 12   0  50
 14 894 916
 16 289 291
 18 409 381
 20   0  76

H,K= -4,  1
L=1 386 333
  3 537 563
  5 894 901
  714981449
  9 565 589
 11  94 119
 13 337 363
 15  95 128
 17 129 161
 19 161 166
 21   0  20

H,K= -4,  2
L=215681566
  4 642 660
  6 138 171
  8   0  22
 10 218 242
 12 464 484
 14 196 210
 16 138 130
 18   0  87
 20 195 203

H,K= -4,  3
L=1 842 843
  3 418 410
  5   0  50
  7 234 214
  9  35   5
 11   0  76
 13 186 205
 15 246 248
 17 283 305
 19  95 103

H,K= -4,  4
L=2 232 264
  4 260 292
  6 228 243
  8  74  68
 10   0  44
 12   0  40
 14  97 122
 16   0  21
 18 136 122

H,K= -4,  5
L=1 315 335
  3   0  19
  5 530 537
  7 115  95
  9 434 475
 11 168 149
 13 131 108
 15   0  16

H,K= -4,  6
L=2  83 107
  4 200 202
  6  95 100
  8 168 175
 10  94  89
 12 170 117

H,K= -4,  7
L=1 106 148
  3   0  72
  5 150 140
```

Table 3 (*cont.*)

FO FC	FO FC	FO FC	FO FC	FO FC	FO FC	FO FC	FO FC	FO FC	FO FC
H,K= -5, 1	H,K= -5, 5	H,K= -6, 2	H,K= -7, 1	H,K= -7, 5	H,K= -8, 3	H,K= -9, 2	H,K=-10, 2	H,K=-11, 2	H,K=-12, 3
L=2 934 942	L=2 152 120	6 72 85	L=2 559 530	10 0 47	7 308 314	15 0 18	L=2 109 100	13 0 62	L=1 161 178
4 707 731	4 57 81	8 542 518	4 0 56	12 83 90	9 315 322		4 81 89		3 379 366
6 243 250	6 294 285	10 127 140	6 575 564		11 427 432	H,K= -9, 3	6 0 25	H,K=-11, 3	5 296 273
8 425 453	8 159 148	12 94 69	8 227 240	H,K= -7, 6	13 202 205	L=2 117 107	8 393 361	L=2 62 88	7 457 440
10 271 264	10 65 75	14 177 171	10 159 170	L=1 0 2	15 149 166	4 177 156	10 299 275	4 218 229	9 0 72
12 423 430	12 179 182	16 129 94	12 0 20	3 0 72		6 147 151	12 133 114	6 289 293	
14 333 331	14 54 77	18 0 9	14 0 29	5 244 252	H,K= -8, 4	8 166 154	14 233 198	8 221 223	H,K=-12, 4
16 310 328			16 0 30	7 420 434	L=2 335 323	10 85 84		10 134 147	L=2 102 101
18 131 141	H,K= -5, 6	H,K= -6, 3	18 246 223		4 0 55	12 344 352	H,K=-10, 3	12 202 210	4 269 266
20 250 277	L=1 79 77	L=1 454 459		H,K= -8, 0	6 221 199	14 118 145	L=1 60 45		6 0 14
	3 0 29	3 267 285	H,K= -7, 2	L=2 131 132	8 168 125		3 0 36	H,K=-11, 4	
H,K= -5, 2	5 122 112	5 317 301	L=1 338 359	4 535 549	10 88 69	H,K= -9, 4	5 461 477	L=1 0 7	H,K=-13, 1
L=1 386 375	7 0 52	7 24 54	3 188 207	6 895 903	12 0 2	L=1 457 479	7 69 73	3 298 326	L=2 248 237
3 608 621	9 157 159	9 1172 1131	5 0 23	8 619 616	14 494 511	3 0 52	9 0 14	5 0 1	4 0 72
5 517 541	11 0 89	11 136 155	7 40 50	10 225 222		5 303 314	11 218 228	7 0 38	6 152 149
7 354 374		13 0 66	9 70 25	12 163 139	H,K= -8, 5	7 156 145	13 0 6	9 344 328	8 133 118
9 315 325	H,K= -5, 7	15 58 80	11 358 348	14 45 42	L=1 0 79	9 266 280			10 253 230
11 321 322	L=2 0 21	17 122 122	13 127 129	16 305 302	3 78 70	11 209 222	H,K=-10, 4	H,K=-11, 5	
13 466 463			15 587 607	18 0 17	5 179 180	13 255 244	L=2 329 339	L=2 0 70	H,K=-13, 2
15 102 94	H,K= -6, 0	H,K= -6, 4	17 251 282		7 267 265		4 129 139		L=1 0 63
17 262 252	L=2 1465 1505	L=2 347 354		H,K= -8, 1	9 310 349	H,K= -9, 5	6 452 452	H,K=-12, 0	3 202 191
19 108 95	4 388 395	4 225 250	H,K= -7, 3	L=1 351 381	11 0 55	L=2 179 163	8 0 5	L=2 227 212	5 118 94
	6 1396 1435	6 0 54	L=2 104 134	3 661 662		4 125 110	10 0 89	4 904 869	7 180 175
H,K= -5, 3	8 1138 1093	8 358 364	4 294 304	5 165 168	H,K= -8, 6	6 0 21		6 54 66	9 0 8
L=2 53 57	10 0 80	10 0 13	6 60 27	7 109 81	L=2 198 203	8 0 13	H,K=-10, 5	8 118 99	
4 688 676	12 452 445	12 108 107	8 464 464	9 95 103	4 203 207		L=1 0 53	10 328 334	H,K=-13, 3
6 294 296	14 283 279	14 53 70	10 155 158	11 344 351	6 0 67	H,K=-10, 0	3 299 287	12 0 107	L=2 0 25
8 347 365	16 0 6	16 129 96	12 239 267	13 239 241		L=2 212 217	5 243 234		4 360 369
10 241 249	18 289 379		14 345 334	15 81 75	H,K= -9, 1	4 267 261	7 69 32	H,K=-12, 1	6 0 9
12 494 494	20 312 316	H,K= -6, 5	16 0 43	17 390 385	L=2 512 507	6 104 94		L=1 140 166	
14 85 93		L=1 67 82			4 558 546	8 407 420	H,K=-11, 1	3 26 69	H,K=-14, 0
16 0 61	H,K= -6, 1	3 0 58	H,K= -7, 4	H,K= -8, 2	6 90 56	10 273 271	L=2 101 107	5 241 238	L=2 439 423
18 221 207	L=1 525 538	5 0 27	L=1 195 201	L=2 158 185	8 124 140	12 478 487	4 478 464	7 0 23	4 276 292
	3 484 454	7 411 412	3 129 127	4 92 107	10 714 687	14 92 99	6 200 192	9 0 58	6 255 255
H,K= -5, 4	5 292 307	9 241 246	5 539 550	6 0 10	12 305 312	16 457 439	8 0 80	11 78 121	8 365 358
L=1 184 194	7 565 557	11 203 174	7 253 248	8 462 471	14 111 96		10 200 191	13 530 492	
3 0 61	9 159 135	13 143 129	9 219 205	10 324 331	16 273 260	H,K=-10, 1	12 0 120		H,K=-14, 1
5 498 519	11 326 357		11 173 177	12 0 21		L=1 575 535	14 156 141	H,K=-12, 2	L=1 353 350
7 235 238	13 344 342	H,K= -6, 6	13 227 222	14 285 284	H,K= -9, 2	3 528 521		L=2 101 89	3 117 100
9 159 158	15 778 763	L=2 471 498	15 0 39	16 392 380	L=1 172 179	5 429 396	H,K=-11, 2	4 79 66	5 353 354
11 92 112	17 0 65	4 0 13		18 0 33	3 977 965	7 102 81	L=1 117 122	6 40 10	7 423 402
13 83 69	19 230 237	6 338 357	H,K= -7, 5		5 122 92	9 541 520	3 81 50	8 0 212	
15 239 231		8 0 22	L=2 0 12	H,K= -8, 3	7 133 125	11 292 289	5 354 365	10 0 84	H,K=-14, 2
17 228 222	H,K= -6, 2	10 92 81	4 141 165	L=1 353 345	9 592 571	13 134 106	7 528 520	12 62 41	L=2 133 145
	L=2 191 204		6 113 115	3 409 397	11 54 36	15 0 1	9 102 75		4 384 404
	4 585 614		8 72 45	5 338 332	13 262 239		11 207 216		

are completely coplanar, whereas the remaining two cyclobutanes, 7–8–9–1, and 5–4–3–2, have a puckered configuration. This result added another datum to the controversial question of configurations of relatively unstrained cyclobutane rings. A partial list of observed cyclobutane configurations in various types of compounds is given in the earlier paper (Okaya & Bed-

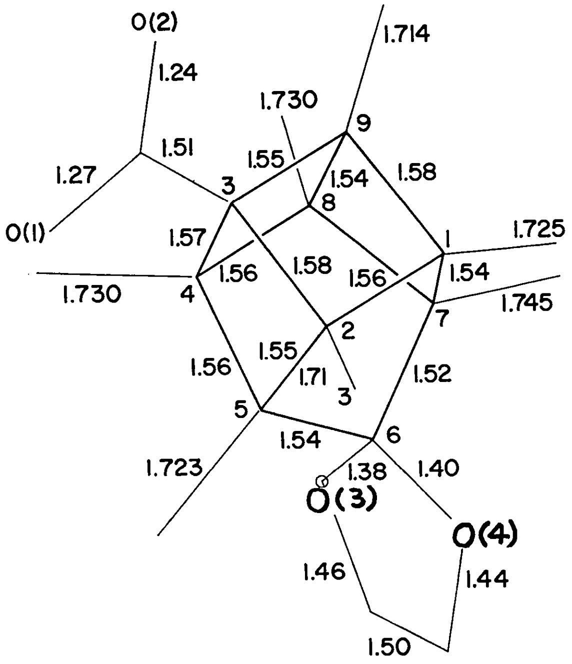


Fig.4. Bond distances in the molecule, in Å.

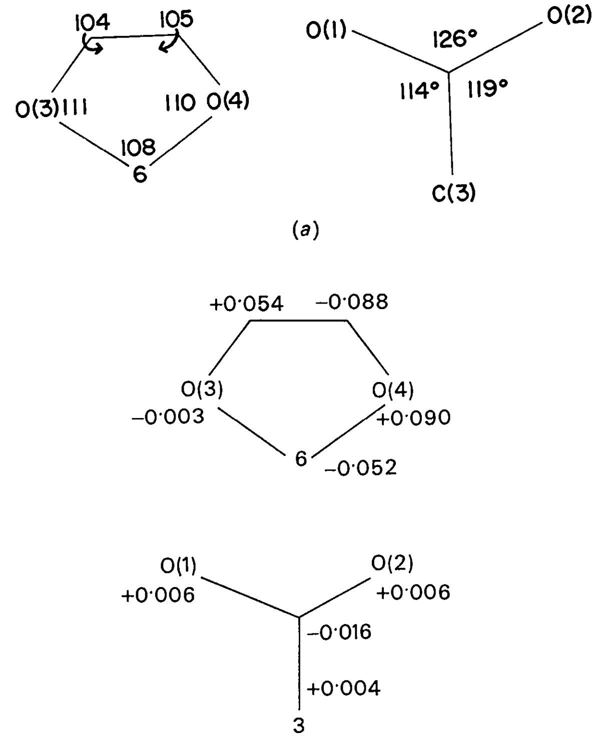


Fig.5. Configurations of the two derivative groups. (*a*) Bond angles. (*b*) Planarity of the groups. Deviations of atoms from the least-squares planes are shown. See Fig.4 for bond distances.

nowitz, 1967). The geometry of the two cyclopentane rings is shown in Figs. 6 and 7. For each group, the four atoms involved in other cyclobutane ring formations lie on a perfect plane out of which the apex, 6, projects by about 0·7 Å. As shown in Fig. 6(*b*) the bond angles show tendencies similar to those in the pentacyclodecane system [Fig. 3(*c*)] (Okaya & Bednowitz, 1967). The results well established that, for cyclopentane rings in such a condensed system, the least-constrained apex carbon atoms undergo the largest deviations from the normal valence angles. As discussed previously, one of the diagonals of the cage is an approximate mirror plane; the planarity of the diagonal is also studied and the results shown in Fig. 7(*b*).

The carbon atoms in the cage structure can be divided into four groups; (*a*) the apex, 6; (*b*) the nearest neighbors of the apex, 5 and 7; (*c*) four next nearest neighbors involved in cyclopentanes, 1,2,4 and 8; and (*d*) 3 and 9 of the base edge. Fig. 8(*a*)–(*d*) shows bond angles outside the ring system classified into these four groups. One notices obvious systematic variations in the bond angles. Similar variations have been observed in the decane compound (Okaya & Bednowitz, 1967).

The shape and size of the carboxyl and ethylenedioxy groups, can be seen in Figs. 4, and 5(*a*) and (*b*). The ethylenedioxy group (on 6) is a puckered ring with the C(6)–O distances shorter than CH_2–O by about 0·05 Å. The ring makes an angle of 88° with the diagonal mirror plane of the cage. The carboxyl group has the usual configuration found when there is a dimer formation.

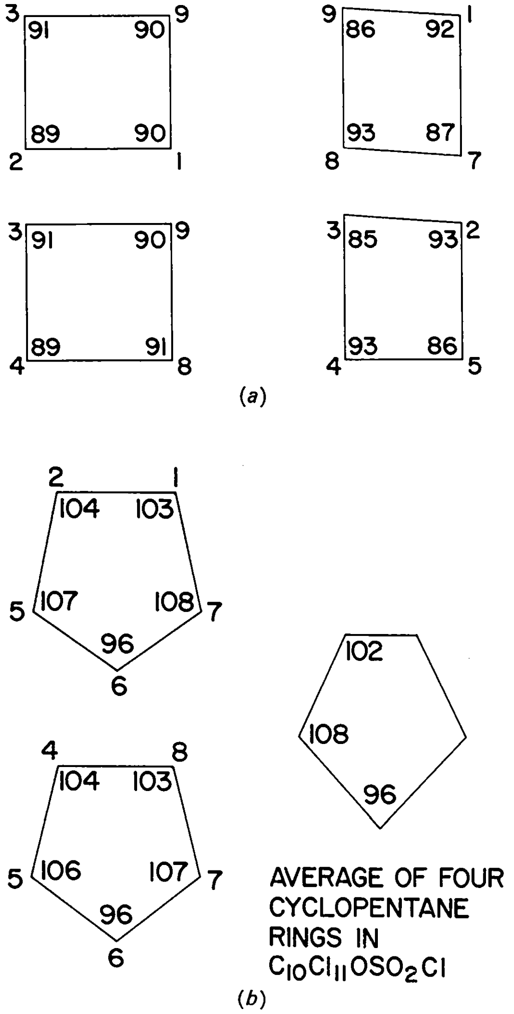

3 91 90 9
89 90
2 1
9 86 92 1
93 87
8 7
3 91 90 9
89 91
4 8
3 85 93 2
93 86
4 5
(a)
2 1
104 103
5 107 108 7
96
6
4 8
104 103
5 106 107 7
96
6
102
108
96
AVERAGE OF FOUR CYCLOPENTANE RINGS IN $C_{10}Cl_{11}OSO_2Cl$
(b)

Fig. 6. (*a*) Bond angles in the cyclobutane rings, in degrees. (*b*) Bond angles in the cyclopentane rings, in degrees. The average values of the four cyclopentanes in $C_{10}Cl_{11}.OSO_2Cl$, a pentacyclodecane compound, are shown for comparison. (Okaya & Bednowitz, 1967).

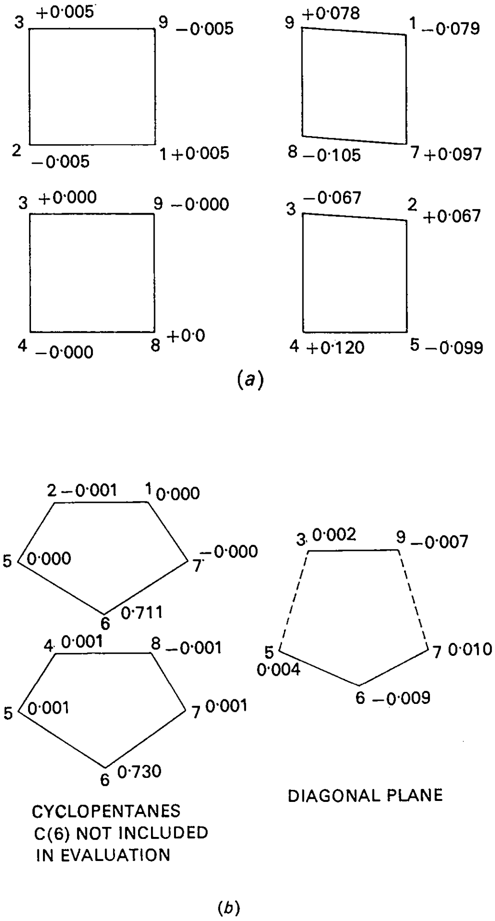

3 +0·005
9 −0·005
2 −0·005
1 +0·005
9 +0·078
1 −0·079
8 −0·105
7 +0·097
3 +0·000
9 −0·000
4 −0·000
8 +0·0
3 −0·067
2 +0·067
4 +0·120
5 −0·099
(a)
2 −0·001
1 0·000
5 0·000
7 −0·000
6 0·711
4 0·001
8 −0·001
5 0·001
7 0·001
6 0·730
CYCLOPENTANES C(6) NOT INCLUDED IN EVALUATION
3 0·002
9 −0·007
5 0·004
7 0·010
6 −0·009
DIAGONAL PLANE
(b)

Fig. 7. (*a*) Planarity of the cyclobutane rings. The deviations of atoms from the least-squares planes are shown. The cyclobutanes are divided into two groups, coplanar and puckered (see text). (*b*) The planarity of the cyclopentanes and the diagonal plane. In evaluating the least-squares planes for the cyclopentanes, the apex carbon atom was not included. From the plane of the diagonal (which included the apex, 6), other carbons in the cage are shifted by the following amounts (in Å): C(1), 1·07; C(8), −1·07; C(2), 1·07; C(4), −1·06.

The crystal structure is a usual carboxyl acid structure. The carboxyl groups are involved in forming dimers around centers of symmetry by O–H··O hydrogen bonds of 2·60 Å and the dimeric molecules thus formed stack in the structure by usual van der Waals contacts. All intermolecular approaches exhibit normal separations. The general feature of the structure can be obtained from Fig. 2.

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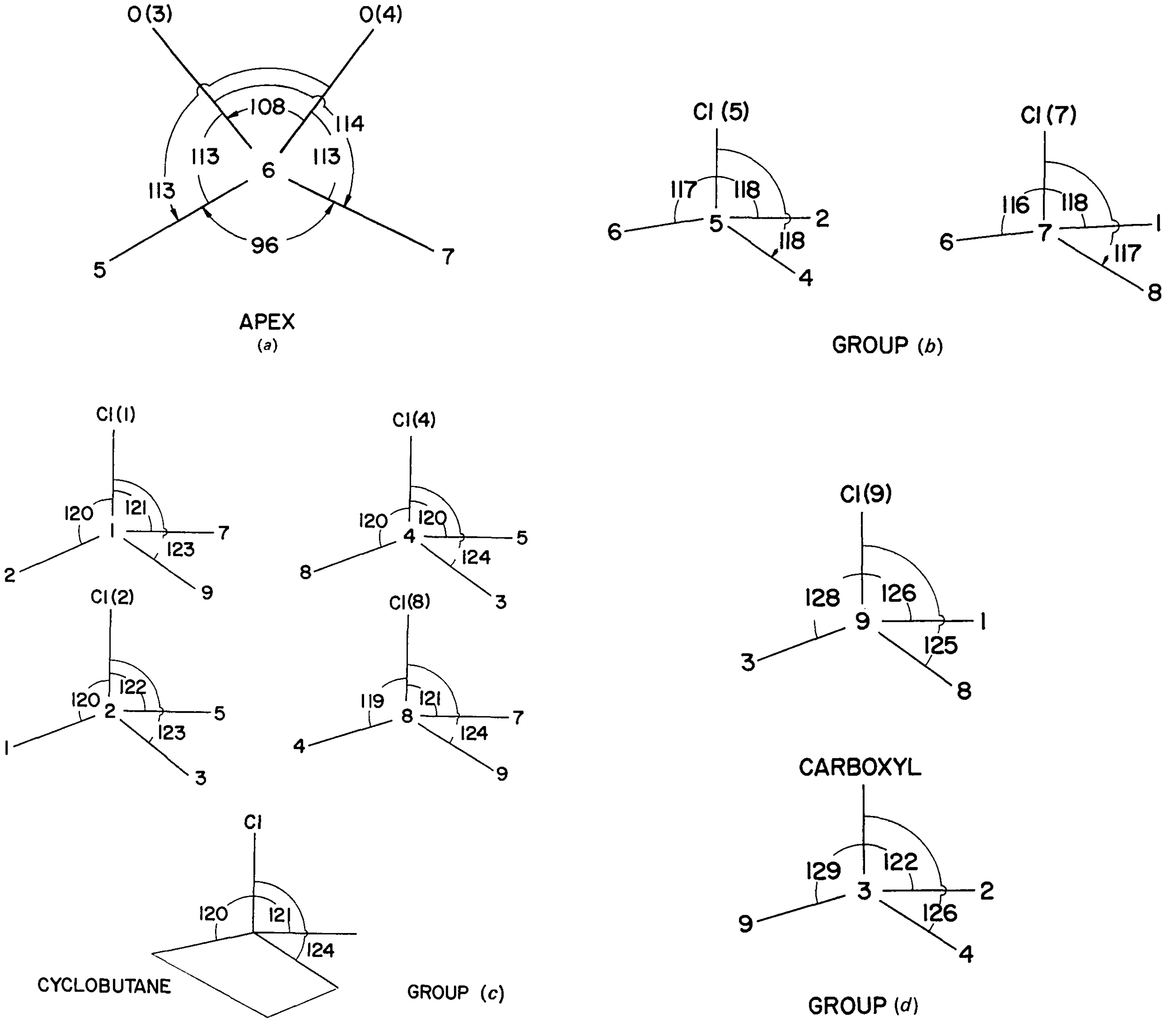
O(3)
O(4)
108
114
113
113
113
6
96
5
7
APEX
(a)
Cl (5)
117
118
5
2
118
6
4
Cl (7)
116
118
7
1
117
6
8
GROUP (b)
Cl (1)
120
121
1
7
123
2
9
Cl (4)
120
120
4
5
124
8
3
Cl (2)
120
122
2
5
123
1
3
Cl (8)
119
121
8
7
124
4
9
Cl
120
121
124
CYCLOBUTANE
GROUP (c)
Cl (9)
128
126
9
1
125
3
8
CARBOXYL
129
122
3
2
126
9
4
GROUP (d)

Fig. 8. Bond angles outside the cage skeleton, in degrees. (*a*) The apex carbon, 6. (*b*) Group *b*. (*c*) Group *c*. The average values for the four atoms are also shown. (*d*) Group *d*. For the four groups into which the skeleton carbon atoms are classified, see text.