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Crystal and Molecular Structure of a Chlorosulfonate of a Novel Cage Chlorocarbon, $C_{10}Cl_{11}SO_3Cl$, Determined by the Symbolic Addition Method*

BY Y. OKAYA

IBM Watson Research Center, Yorktown Heights, New York, 10598, *U.S.A.*

AND A. BEDNOWITZ*

**Chemistry Department, Brookhaven National Laboratory, Upton, New York* 11973, *U.S.A.*

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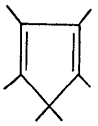
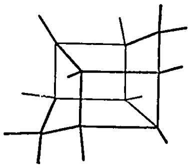
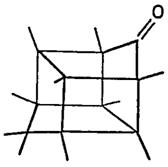
Hexachlorocyclopentadiene C_5Cl_6 dimerizes by catalytic action of aluminum chloride into an unusually stable chlorocarbon, $C_{10}Cl_{12}$. The configuration of the molecule has been studied by various physicochemical methods such as thermal and dielectric studies on crystals, nuclear magnetic resonance, infrared spectroscopic and dipole moment measurements of solutions. The results indicate that the molecule possesses a cage structure which consists of two cyclopentane rings connected by four single bonds. Since $C_{10}Cl_{12}$ exhibits a disordered structure in the room temperature phase, a chlorosulfonate group was introduced onto one of the apex carbon atoms. The material crystallizes in the monoclinic system with $a=16{\cdot}75_8\pm0{\cdot}003$, $b=8{\cdot}75_3\pm0{\cdot}002$, $c=14{\cdot}45_9\pm0{\cdot}003$, $\beta=112{\cdot}0°\pm0{\cdot}1$; the space group is $P2_1/a$. The crystal structure was determined by the symbolic addition method and refined by the least-squares method. The basic cage structure is *trans* consisting of four cyclopentane and two cyclobutane rings, all of which are puckered. The chlorocarbon chlorosulfonate may then be called undecachloropentacyclo[$5.3.0.0^{2,6}.0^{3,9}.0^{4,8}$]decan-5-chlorosulfonate. The bond distances and angles in this fused ring system are discussed in detail.

Introduction

Hexachlorocyclopentadiene (I) C_5Cl_6 dimerizes by catalytic action of aluminum chloride to an unusually stable chlorocarbon, $C_{10}Cl_{12}$, of m.p. 485° (Prins, 1946; Newcomer & McBee, 1949*b*). It was also reported that hexachlorocyclopentadiene gives a related ketone on reaction with liquid sulfur trioxide (Gilbert & Giolito, 1958). From an infrared study of the chlorocarbon and the ketone, McBee, Roberts, Idol & Earle (1956) concluded that these compounds have cage structures and proposed the structures (II) and (III) for the chlorocarbon and the ketone respectively. However, from the infrared spectroscopic evidence, it was impossible to decide the relative orientation of the top and the bottom five-membered rings in (II) and (III); there exist two possible configurations for the cage structure; one is *trans* as shown in (II) and the other gauche (IV) obtained by rotating one of the rings by 90°. The third possibility of *cis* configuration can be ruled out because of the relatively bulky chlorine groups on the two apex carbon atoms. Various physico-chemical results on the chlorocarbon favor the highly symmetrical *trans* (II) structure. A crystallographic study of $C_{10}Cl_{12}$ at room temperature reveals a cubic disordered structure which, below a transition point of 2·5 °C, transforms into a probably ordered orthorhombic structure. By raising the temperature, the cubic disordered structure changes into a cubic face-centered close-packed structure (at 122°) indicating rotation or completely statistical arrangement of molecules in the structure (see Fig. 1). Although these transitions are accompanied by specific heat anomalies, there is no dielectric anomaly associated with them (Okaya, Pepinsky & Gilbert, 1960); the molecule was also found to possess no dipole moment (Zijp & Gerding, 1958). A recent nuclear magnetic resonance study on $C_{10}H_{12}$ obtained from $C_{10}Cl_{12}$ by the action of $LiAlH_4$ also favors the *trans* configuration (McBee, Dilling & Braendlin, 1962).


(I)


(II)



(III)

In view of these various data on the basic configuration of the cage structure, it seemed worthwhile to carry out crystal structure analyses of some cage chlorocarbon derivatives. The structure analysis will also reveal the shape of the cyclopentane as well as cyclobutane rings in such a condensed system. Since the basic chlorocarbon $C_{10}Cl_{12}$ shows disorder at room temperature, an attempt was made to introduce asymmetry into the structure. The symmetric as well as asymmetric cage molecules, $C_{10}Cl_{10}Br_2$, obtained by bromination on the apex carbon atoms (Gilbert & Lombardo, 1962) also exhibit similar disorder (Okaya, Pepinsky & Gilbert, 1960). Some of the results of

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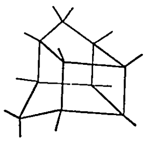
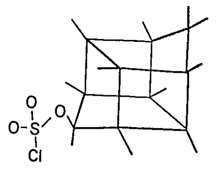
thermal studies are shown in Table 1. An effort was then made to incorporate a larger group on one of the apex carbons; this was achieved by causing hexachlorocyclopentadiene to react with HSO_3Cl thereby making a chlorosulfonate (V), $C_{10}Cl_{11}OSO_2Cl$ (Newcomer & McBee, 1949*a*). Since the chlorosulfonate thus obtained can easily be transformed into the cage ketone by mild reaction with sodium hydroxide, it can safely be assumed that there is no change involved in the basic cage configuration. It should also be mentioned here that action of phosphorus pentachloride on the ketone produces the chlorocarbon $C_{10}Cl_{12}$ (II) (Gilbert, 1957). The present paper deals with the crystal and molecular structures of the chlorosulfonate directly determined by the symbolic addition method.



(IV)



(V)

Experimental

Crystals of the chlorosulfonate used in data collection were obtained from toluene solution; they were ground into approximately spherical shape and Weissenberg photographs were taken around the *c* and *b* axes with filtered Cu *Kα* radiation. The crystal belongs to the monoclinic system with $a = 16{\cdot}75_8 \pm 0{\cdot}003$, $b = 8{\cdot}75_3 \pm 0{\cdot}002$, $c = 14{\cdot}45_9 \pm 0{\cdot}003$, $\beta = 112{\cdot}0° \pm 0{\cdot}1°$; the space group is $P2_1/a$. There are four molecular units of $C_{10}Cl_{11}OSO_2Cl$ in the unit cell. Three-dimensional intensity data were obtained by visual comparison of multiple-film exposure photographs with a calibrated scale; the intensity data thus obtained were corrected for the Lorentz–polarization as well as absorption effects on an IBM 704 computer. Out of about 3500 independent reflections accessible in the Cu *Kα* limiting sphere, about 3000 reflections were strong enough to be observed. The data collection and the preliminary data handling were carried out in 1959 at the then Crystallographic Laboratory of the Pennsylvania State University. Since it was obvious that the crystal structure was not amenable to be studied by the conventional Patterson method, the structure determination

Table 1. *Phase transitions of the crystals of* $C_{10}Cl_{12}$ *and brominated compounds* $C_{10}Cl_{10}Br_2$*

	Lower transition			Upper transition		
	Temperature (°C)	Q (Kcal.mol^{-1})	S (cal.mol^{-1}. deg^{-1})	Temperature (°C)	Q (Kcal.mol^{-1})	S (cal.mol^{-1}. deg^{-1})
$C_{10}Cl_{12}$	2·5	$1{\cdot}0_0$	3·6	122	$1{\cdot}2_0$	3·0
Asymmetric $C_{10}Cl_{10}Br_2$	77	$1{\cdot}8_0$	5·1	107	$1{\cdot}3_0$	3·0
Symmetric $C_{10}Cl_{10}Br_2$	−24	0·64	2·6	135	1·81	4·5

* Taken from data of Okaya, Pepinsky & Gilbert (1960).

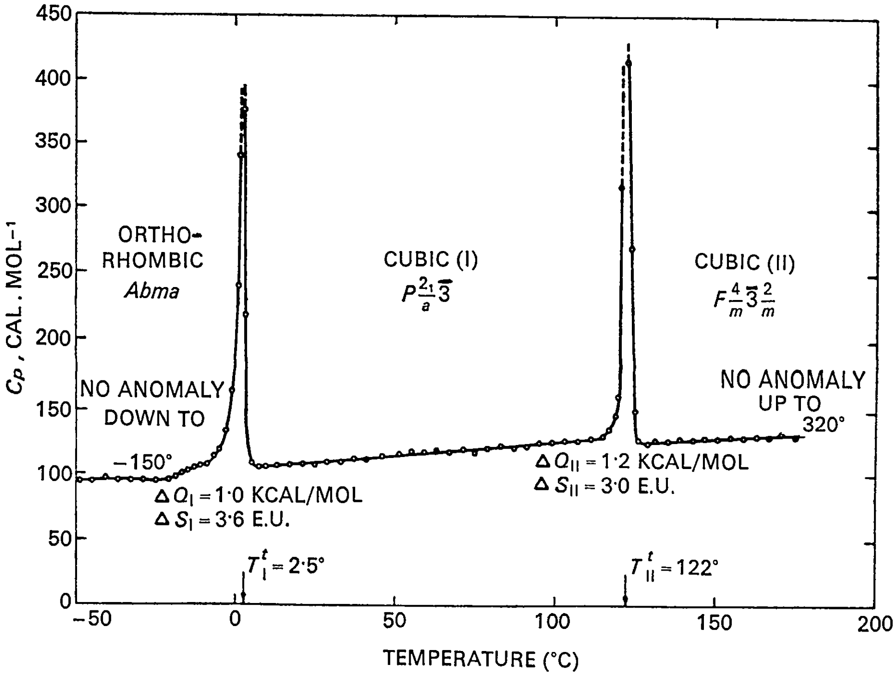


Fig. 1. Specific heat curve of $C_{10}Cl_{12}$. The three phases are identified with their space group.

was set aside until it became possible to utilize the symbolic addition direct method for determining centrosymmetric structures.

An absolute scale factor and mean isotropic temperature factor were obtained by a Wilson statistical analysis. The normalized structure factor magnitudes, $E_{\mathbf{H}}$, were computed using the relation

$$E^2_{\mathbf{H}} = F^2_{\mathbf{H}} / \sum_{j=1}^{N} \varepsilon f^2_{j\mathbf{H}} \tag{1}$$

where ε is unity for all reflections other than $h0l$ and $0k0$, for which $\varepsilon = 2{\cdot}0$. N is the number of atoms in the unit cell and the F's are on an absolute scale and corrected for thermal motion. Only those normalized structure factors greater than 1·4 (13% of the total number observed) were used in the phase determination.

Phase determination

The symbolic addition procedure (Karle & Karle, 1963) was used to determine the phases directly from the normalized structure factor magnitudes. A computer program, SORTE, written in FORTRAN IV, was used to aid the implementation of the symbolic addition procedure (Bednowitz & Post, 1965). The phase relation used by this procedure is the Σ_2 formula (Karle & Hauptman, 1953)

$$sE_{\mathbf{H}} \approx s \sum_{\mathbf{K}} E_{\mathbf{K}} E_{\mathbf{H}-\mathbf{K}} \tag{2}$$

where s means 'sign of'. This formula describes the phase interaction between the reflection **H** and all other pairs **K**, **H** − **K**. The symbolic addition procedure requires only a few initially known phases in order to determine enough additional signs by formula (2) so that the main features of the structure can be obtained by Fourier analysis.

Among the starting set of 'known' phases are three reflections chosen with arbitrary phase (+ or −) thereby fixing the origin for this space group. In addition, several other reflections were chosen and assigned symbolic phases. The starting set used in the present study is listed in Table 2. Each reflection chosen for the starting set has a relatively large $|E|$ and enters into relatively many interactions as found in a listing of the first pass of the SORTE program.

The first symbols assigned after the origin fixing (+) signs were a and b. The symbolic addition process did not progress very far before it was evident that additional symbols would be necessary. After assigning c, d and e, the process ran smoothly. As there were many early indications that $a \equiv be$, the symbol a was replaced by be, thereby reducing the total of unknown symbols to four.

Table 2. *Starting set of assigned phases and symbols for the application of* Σ_2

Sign	hkl	E
+	$3,2,\bar{4}$	3·08
+	$6,4,3$	2·81
+	$4,1,\overline{10}$	2·43
a	$11,6,\bar{7}$	3·22
b	$4,2,\bar{1}$	2·73
c	$12,7,\bar{3}$	3·00
d	$11,2,\bar{1}$	2·65
e	$12,2,\bar{4}$	3·22

The probability formula associated with the Σ_2 relation is

$$P_+(E_{\mathbf{H}}) = \tfrac{1}{2} + \tfrac{1}{2} \tanh \frac{\sigma_3 |E_{\mathbf{H}}| \, \Sigma_{\mathbf{K}} \, E_{\mathbf{K}} \, . \, E_{\mathbf{H}-\mathbf{K}}}{\sigma_2^{3/2}}\,, \tag{3}$$

where $\sigma_n = \sum_{j=1}^{N} Z_j^n$. Z_j is the atomic number of the jth atom, and $P_+(E_{\mathbf{H}})$ is the probability of the sign of $E_{\mathbf{H}}$ being positive. In order to use this relation to make a programmed choice of the symbolic sign, (3) was modified to

$$P_s(E_{\mathbf{H}}) \geq \tfrac{1}{2} + \tfrac{1}{2} \tanh \frac{\sigma_3 |E_{\mathbf{H}}| \, . \, R_s}{\sigma_2^{3/2}}\,, \tag{4}$$

where P_s is the probability of s being a correct symbol. R_s is the difference between the sum of the double products associated with the dominant symbol (s) and the sum of the double products of all other symbols. Usually for large E there will be a dominant symbol whose associated double product sum is greater than the total sum associated with all other symbols. Occasionally a primary reflection (**H**) does not have a dominant symbol. In that case the phase is assumed to be temporarily indeterminate. Often this temporary indeterminancy can be removed by a suitable choice of sign values or relations among the symbolic phases. In fact many of the temporarily indeterminate reflections are extremely useful in obtaining information on relations between symbols, *e.g.* the relation $a \equiv be$ was obtained in just this fashion. After 254 reflections had been determined symbolically, several relations were evident among the symbols, suggesting the following sign values: $b = -$; $c = +$; $d = +$; $e = -$.

Inserting these values enabled the phase determining process to develop a total of 383 signs (plus five truly indeterminate phases) with $|E|$ greater than 1·4. Using these phases an E map was computed and an automatic peak search listed the maxima of the map in decreasing magnitude. The thirteen largest peaks were assumed to be the chlorine and sulfur positions. The lowest of these was about twice the magnitude of the peaks in the rapidly varying background. A check of the interpeak distances for twelve out of the thirteen largest peaks indicated that the distances were all greater than 2·8 Å; these results were in agreement with the expected distribution of chlorine to chlorine distances in the cage structures. Fig. 2 shows the final coordinates superimposed on a composite drawing of the three-dimensional E map.

Starting from the coordinates of those relatively heavy peaks found in the E map, the positions of the carbon atoms in the cage skeleton and the oxygen atoms in the chlorosulfonate group were obtained by iterative structure-factor calculations and difference electron-density syntheses. After the thirteen light atoms had been found in the difference syntheses, various views of the structure were drawn on an IBM 1627 X–Y plotter based on calculation done on an IBM 7094 computer (Okaya, 1966). The overall molecular shape is quite obvious in the drawings thus obtained (Fig. 3) and revealed the *trans* structure of the cage and the presence of a chlorosulfonate group on one of the apex carbon atoms.

The atomic coordinates of all the atoms were then subjected to the least-squares treatment with anisotropic temperature factors to account for their thermal vibrations. After several cycles of this treatment with a full matrix refinement program, the error index $\Sigma||F_o|-|F_c||/\Sigma|F_o|$ was reduced to 12·9%. Since the intensity data were recorded photographically and the accuracy is not too high, it was decided to terminate the refinement at this stage; the atomic coordinates, their standard deviations and thermal parameters thus obtained are shown in Table 3. Following are some of the details of the computation procedures; all computations at the later stages were done on IBM 7094 computers at the IBM Research Center and the Brookhaven National Laboratory. The atomic scattering factors used in the structure-factor calculation were those listed in *International Tables for X-ray Crystallography* (1962). The weighting scheme used in the least-squares refinement was:

$$w=1{\cdot}0 \text{ for } |F_{\text{obs}}| \leq 50{\cdot}0$$
$$w=50{\cdot}0/|F_{\text{obs}}| \text{ for } |F_{\text{obs}}| > 50{\cdot}0;$$

unobserved reflections were given zero weights. The shifts of parameters at the last stage were negligible compared with their standard deviations. None of the 383 reflections whose signs were directly determined had been assigned a phase different from that found by the structure factor calculation. Comparison between the observed and calculated structure factors is given in Table 4.

Discussion

The direct determination of the structure of the chlorosulfonate led to the unambiguous solution of the cage structure; it is composed of two five-membered saturated carbon rings connected by four single C–C bonds. The two apex atoms are in *trans* relationship with each other. The result is in agreement with indirect deductions based on various physico-chemical methods. The nomenclature of these compounds has been giving difficulty, although not ambiguity; the basic chlorocarbon $C_{10}Cl_{12}$ (II) can be expressed by any of the following pentacyclodecane schemes; (*A*) Dodecachloropentacyclo$[5.3.0.0^{2,6}.0^{4,10}.0^{5,9}]$decane with carbons 3 and 8 as the apices; (*B*) $[5.3.0.0^{2,6}.0^{3,9}.0^{4,8}]$ with 5 and 10; (*C*) $[5.2.1.0^{2,6}.0^{3,9}.0^{5,8}]$ with 4 and 10; and (*D*) $[3.3.2.0^{2,6}.0^{3,9}.0^{7,10}]$ with 4 and 8 as the apex carbon atoms. Accordingly the ketone, $C_{10}Cl_{10}O$ (III) can be decachloropentacyclo[ ]-decan-

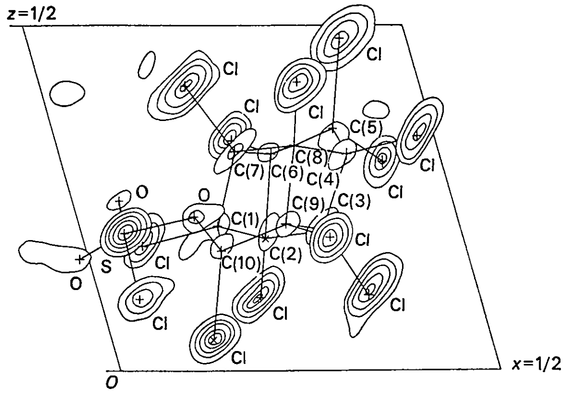


Fig. 2. Composite drawing of the three-dimensional *E*-factor map, computed with 383 directly determined phases. The final atomic coordinates are shown by crosses.

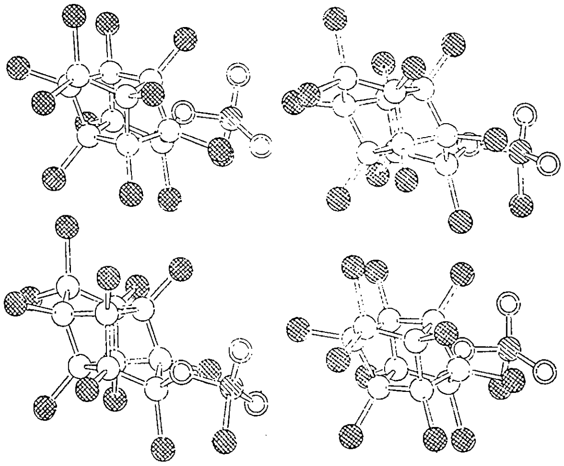


Fig. 3. Several views of the molecule illustrating the cage structure; the views are drawn on an IBM $X-Y$ plotter by rotating the structure around various axes. The atomic coordinates and the peak heights, which were used in deciding proper shades for the atoms, are those obtained from electron-density maps before the refinement stage. The drawings are direct output from the plotter and no retouching was done on them.

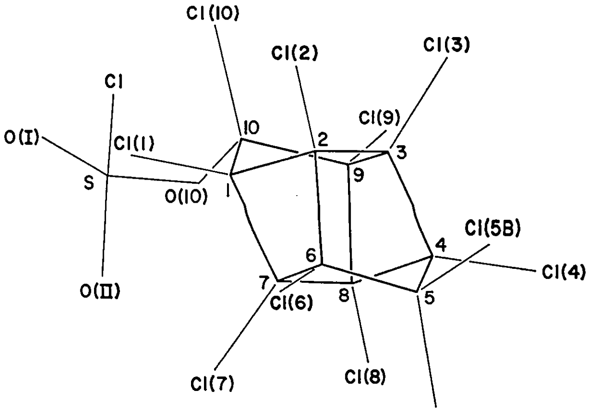


Fig. 4. Drawing of the molecule showing the skeleton structure and the numbering sequence based on one of the various representations of the pentacyclo system as discussed in the text. Note the chlorosulfonate group has been placed on carbon atom 10, which is equivalent to carbon atom 5.

Table 3. *Atomic coordinates and anisotropic temperature factors*

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

Atom	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
Skeleton carbon atoms						
C(1)	0·17669	6	0·15054	11	0·21470	9
C(2)	0·24756	7	0·25032	11	0·19611	10
C(3)	0·31814	7	0·12748	12	0·19823	9
C(4)	0·37968	7	0·13283	12	0·31149	10
C(5)	0·37531	8	0·28919	13	0·35116	12
C(6)	0·27708	8	0·30187	12	0·30828	10
C(7)	0·23592	7	0·15048	11	0·32543	9
C(8)	0·31022	7	0·02801	12	0·32821	11
C(9)	0·27795	7	−0·02235	12	0·21424	11
C(10)	0·17974	6	−0·00922	12	0·17219	11
Chlorine atoms						
Cl(1)	0·07571	1	0·23363	3	0·18403	3
Cl(2)	0·21729	2	0·38267	3	0·10062	3
Cl(3)	0·35857	2	0·14324	3	0·10481	3
Cl(4)	0·48057	1	0·05362	3	0·33980	3
Cl(5A)	0·41427	3	0·28875	4	0·48455	3
Cl(5B)	0·42680	2	0·42877	3	0·30693	3
Cl(6)	0·23463	2	0·47250	3	0·33333	3
Cl(7)	0·19441	2	0·13160	4	0·41576	3
Cl(8)	0·33843	2	−0·10662	3	0·42052	3
Cl(9)	0·31935	2	−0·19250	3	0·18868	3
Cl(10)	0·13366	2	−0·01851	3	0·04263	2
Chlorosulfonate						
S	0·05940	1	−0·19396	3	0·20159	3
Cl(S)	0·05175	2	−0·35647	4	0·10313	4
O(10)	0·15271	5	−0·12591	8	0·22311	7
O(I)	−0·00554	5	−0·08551	11	0·15388	10
O(II)	0·06752	7	−0·26620	12	0·29141	10

(*b*) Anisotropic temperature factors

The β's are used in the expression $\exp\{-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl)\}$

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Skeleton carbon atom						
C(1)	0·00278	0·01377	0·00348	0·00120	0·00319	0·00120
C(2)	0·00325	0·01062	0·00441	0·00005	0·00353	0·00178
C(3)	0·00361	0·01542	0·00356	0·00147	0·00482	0·00273
C(4)	0·00300	0·01518	0·00434	0·00253	0·00230	0·00174
C(5)	0·00439	0·01645	0·00519	0·00077	0·00398	−0·00091
C(6)	0·00440	0·01273	0·00457	0·00006	0·00515	0·00041
C(7)	0·00403	0·01172	0·00317	0·00142	0·00440	0·00136
C(8)	0·00342	0·01263	0·00512	0·00079	0·00390	0·00075
C(9)	0·00355	0·01440	0·00482	0·00064	0·00524	−0·00195
C(10)	0·00288	0·01293	0·00547	−0·00008	0·00382	0·00177
Chlorine atoms						
Cl(1)	0·00331	0·01595	0·00843	0·00417	0·00627	0·00250
Cl(2)	0·00531	0·01538	0·00565	0·00366	0·00600	0·00694
Cl(3)	0·00464	0·01891	0·00596	−0·00056	0·00808	−0·00072
Cl(4)	0·00289	0·01837	0·00865	0·00263	0·00347	0·00161
Cl(5A)	0·00681	0·02289	0·00422	−0·00213	0·00144	−0·00311
Cl(5B)	0·00476	0·01489	0·00916	−0·00422	0·00640	−0·00010
Cl(6)	0·00658	0·01340	0·00709	0·00206	0·00739	−0·00354
Cl(7)	0·00659	0·02255	0·00553	0·00082	0·00954	0·00212
Cl(8)	0·00537	0·01678	0·00590	0·00127	0·00302	0·00793
Cl(9)	0·00458	0·01244	0·00895	0·00263	0·00673	−0·00208
Cl(10)	0·00491	0·01892	0·00369	−0·00086	0·00342	−0·00132
Chlorosulfonate						
S	0·00337	0·01547	0·00652	−0·00233	0·00484	−0·00059
Cl(S)	0·00622	0·01785	0·01135	−0·00338	0·00709	−0·00756
O(10)	0·00341	0·01480	0·00503	−0·00244	0·00359	0·00206
O(I)	0·00326	0·02204	0·00917	0·00138	0·00448	−0·00086
O(II)	0·00695	0·02787	0·00696	−0·00887	0·00610	0·00543

n-one; where *n* stands for one of the two apex carbons of each scheme (customarily use the smaller number). For the chlorosulfonate, $C_{10}Cl_{11}OSO_2Cl$ (V), the structure of which has been studied here, one can assign undecachloropentacyclo[ ]decan-*n*-chlorosulfonate.

One of the combinations, (*B*) undecachloropentacyclo-$[5.3.0.0^{2,6}.0^{3,9}.0^{4,8}]$decan-5-chlorosulfonate, has been given in the abstract. The atomic numbering based on scheme (*B*) will be used in the following discussions. Since the basic cage structure is centrosymmetric and carbon atoms 5 and 10 in this scheme are equivalent, the chlorosulfonate has been placed on carbon atom 10 rather than on 5 for the number sequence shown in Fig. 4.

The connection of the two five-membered rings described above produces two four-membered cyclobutane rings and eventually makes additional two five-membered cyclopentane rings. Fig. 5 is a drawing of the molecule in a stereoscopic pair to demonstrate the ring system, molecular configuration and the orientation and relative magnitude of the thermal ellipsoids. It is of interest to study in detail the shape and size of these ring systems. The problem in building this skeleton is that of stretching two opposite edges of a cube*

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* Cubane, C_8H_8, pentacyclo$[4.2.0.0^{2,5}.0^{3,8}.0^{4,7}]$octane, may be considered as the starting cube. The structure of this cube molecule was studied by Fleischer (1964); C–C, $1{\cdot}55_3$ and $1{\cdot}54_9$ Å; C–C–C $89{\cdot}3$, $89{\cdot}6$ and $90{\cdot}5°$.

Table 4. *Comparison of observed and calculated structure factors* (×10)

H	K	L	FOBS	FCAL
0	0	3	478	675
	0	4	826	1144
	0	5	233	243
	0	6	718	859
	0	7	738	813
	0	8	1273	1341
	0	9	766	746
	0	10	995	1027
	0	11	343	361
	0	12	197	175
	0	13	927	734
	0	14	308	230
	0	15	141	135
	0	16	131	147
2	0	0	673	970
	0	1	123	172
	0	2	1125	1502
	0	4	573	656
	0	5	990	1119
	0	6	427	376
	0	7	421	442
	0	10	195	227
	0	11	187	182
	0	12	276	261
	0	13	333	316
	0	14	89	64
	0	15	167	119
4	0	0	140	240
	0	1	993	1056
	0	2	1804	2383
	0	3	840	918
	0	4	255	254
	0	5	85	104
	0	6	37	21
	0	7	68	108
	0	8	127	83
	0	9	910	875
	0	11	40	71
	0	12	174	137
	0	13	341	267
	0	14	33	45
	0	15	299	273
6	0	0	122	119
	0	1	392	452
	0	2	77	106
	0	3	858	896
	0	4	269	247
	0	5	1055	1010
	0	7	864	829
	0	8	318	302
	0	9	559	467
	0	10	292	259
	0	11	224	169
	0	12	144	137
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	0	9	163	171
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	1	14	161	129
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	1	16	123	85
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	1	2	446	545
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	1	12	44	74
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	1	1	112	148
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	1	4	672	814
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	1	7	177	183
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	1	9	45	70
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	1	1	661	733
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	1	9	299	259
	1	10	220	189
	1	11	165	166
	1	14	113	87
	1	15	27	33
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	1	1	1227	1297
	1	2	430	509
	1	3	136	168
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	1	7	397	420
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	1	11	92	104
	1	13	307	205
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	1	1	530	529
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	2	16	94	112
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	2	13	90	95
	2	15	160	171
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	2	8	229	249
	2	9	401	405
	2	11	547	556
	2	12	92	97
	2	15	237	245
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	2	8	149	136
	2	9	137	150
	2	10	112	113
	2	13	193	157
	2	14	73	72
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	3	13	162	122
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	3	15	72	109
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	3	11	152	134
	3	13	116	83
	3	14	63	46
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	3	7	81	59
	3	8	88	102
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	3	4	216	169
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	3	6	176	165
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	3	1	69	79
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	3	3	39	28
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	3	2	111	149
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	4	5	363	318
	4	6	1546	1384
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	4	8	291	305
	4	9	537	553
	4	10	470	497
	4	12	189	189
	4	14	30	38
	4	15	97	102
	4	16	73	88
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	4	2	158	195
	4	3	714	791
	4	4	174	186
	4	5	521	514
	4	6	207	213
	4	7	401	431
	4	8	119	152
	4	9	544	561
	4	10	237	268
	4	11	223	248
	4	13	74	81
	4	14	95	131
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	4	3	367	396
	4	4	114	107
	4	5	98	124
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	4	10	186	209
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	4	14	56	61
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	4	10	86	90
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	4	13	132	138
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	4	6	185	195
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	4	12	37	60
	4	13	97	96
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	4	11	160	149
	4	13	155	170
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	4	7	249	268
	4	8	193	203
	4	9	50	80
	4	12	17	10
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	4	3	213	229
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	4	6	53	64
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	4	9	95	97
	4	10	78	83
	4	11	110	121
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	4	4	219	228
	4	5	53	72
	4	6	90	116
	4	10	131	142
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	4	4	236	268
	4	5	177	217
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	4	1	331	332
	4	2	293	301
	4	3	348	406
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	4	5	86	123
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	4	3	102	117
	4	5	112	142
	4	6	88	111
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	4	8	59	61
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	4	6	83	102
	4	7	46	57
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	4	3	130	131
	4	4	236	262
	4	5	159	182
	4	6	45	56
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Table 4 (*cont.*)

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	5	8	249	235
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	5	13	84	74
	5	14	134	123
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	5	1	49	144
	5	2	761	674
	5	3	137	167
	5	4	75	74
	5	5	241	242
	5	6	186	210
	5	7	321	345
	5	10	212	220
	5	11	172	174
	5	12	203	211
	5	13	176	159
	5	14	58	71
	5	15	77	115
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	5	1	724	436
	5	3	169	162
	5	4	87	73
	5	6	129	138
	5	7	146	171
	5	8	95	97
	5	10	110	132
	5	11	230	269
	5	13	70	82
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	5	1	355	394
	5	2	363	368
	5	4	65	86
	5	5	402	377
	5	7	549	541
	5	8	194	194
	5	9	207	225
	5	10	79	72
	5	11	119	117
	5	13	151	152
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	5	1	508	555
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	5	3	139	187
	5	4	127	117
	5	5	193	197
	5	6	253	230
	5	7	110	126
	5	8	264	251
	5	10	193	186
	5	12	175	152
	5	13	61	56
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	5	2	87	89
	5	3	195	217
	5	4	192	202
	5	5	203	218
	5	6	207	216
	5	7	411	433
	5	8	127	137
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	5	12	90	83
	5	13	65	79
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	5	4	216	274
	5	5	131	164
	5	6	36	61
	5	7	156	169
	5	11	101	133
	5	12	159	147
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	5	6	77	89
	5	11	191	175
8	5	2	99	159
	5	4	131	141
	5	5	233	276
	5	6	90	113
	5	9	54	73
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	1	-9	186	181
	1	-10	102	128
	1	-13	103	155
20	1	-1	113	161
	1	-2	143	185
	1	-5	268	252
	1	-6	108	96
	1	-7	176	214
	1	-8	92	111

and making two sets of two five-membered rings with angles as close as possible to the normal carbon single-bond angle. This construction leads to a distortion of the four-membered rings from the ideal square configuration. Fig. 6(*a*) illustrates the bond distances in all those ring systems. In spite of the presence of OSO_2Cl on one of the apex carbon atoms, the basic cage is approximately centrosymmetric. The configurations of the cyclobutane rings in the cage molecule can be compared with several other compounds with cyclobutane rings. For example, the centrosymmetric isomer of 1,2,3,4-tetraphenylcyclobutane, $C_4H_4(C_6H_5)_4$ (Dunitz, 1949; refined later by Margulis, 1965), has a planar square ring with the average C–C of 1·57 Å. Also with a square planar ring is 1,2,3,4-*cis-trans-cis*-tetracyanocyclobutane, $C_4H_4(CN)_4$, with an average C–C of 1·55 Å (Greenberg & Post, 1966). For the case of octachlorocyclobutane, C_4Cl_8 (Owen & Hoard, 1951; Margulis, 1965), the ring is non-planar with angles around 88° and the average C–C of 1·57 Å. An electron diffraction study of cyclobutane, C_4H_8, gives $1·548 \pm 0·003$ Å for the C–C distances (Almenningen, Bastiansen & Skancke, 1961) with a puckered configuration. For the present molecule, the average C–C is 1·57 Å with the angles around 87° thus forming puckered rings. The carbon atoms in the cage skeleton can be classified in three categories; (*a*) C(5) and C(10), the apex carbon atoms; (*b*) C(2), C(3), C(7) and C(8) which are furthest from the apices; and (*c*) C(1), C(9), C(4) and C(6) which have apex carbon atoms as neighbors. As shown in Fig. 7, the angles in the four puckered cyclopentane rings exhibit interesting features. The two angles around the carbons in group (*a*) are about 96°; those around group (*b*) and (*c*) are 101° and 108°, respectively. Similar narrow angles around the apex carbons can be found in C_8F_{12}, a saturated dimer of hexafluorobutadiene; the average apex angle is 97° (Karle, Karle, Owen & Hoard, 1965). Because of the strain, the cyclopentane rings take configurations different from the free cyclopentane molecule. The bond angles formed outside of the skeleton have interesting features worth mentioning. Fig. 8 lists those angles involving chlorine atoms and the bridge oxygen atom of the chlorosulfonate, O(10).

Table 4 (*cont.*)

H	K	L	FOBS	FCAL
1	2	-1	62	56
	2	-2	822	724
	2	-3	242	231
	2	-5	475	434
	2	-6	272	281
	2	-7	310	310
	2	-8	245	217
	2	-9	253	230
	2	-10	51	68
	2	-11	44	74
	2	-12	123	128
	2	-13	106	76
	2	-16	165	137
	2	-17	93	121
2	2	-1	839	524
	2	-2	262	271
	2	-3	1537	1195
	2	-4	181	119
	2	-5	343	349
	2	-6	143	113
	2	-7	447	426
	2	-8	73	64
	2	-9	703	589
	2	-10	205	178
	2	-11	90	75
	2	-12	127	132
	2	-13	240	246
	2	-14	108	126
	2	-15	70	62
	2	-16	152	154
	2	-17	24	56
3	2	-1	1510	1517
	2	-2	1619	1429
	2	-3	177	161
	2	-4	2609	2514
	2	-5	1074	721
	2	-6	284	173
	2	-7	507	423
	2	-8	1426	1127
	2	-9	126	108
	2	-10	289	252
	2	-13	104	87
	2	-14	473	477
	2	-15	95	75
	2	-17	160	156
4	2	-1	2528	2510
	2	-2	1316	1267
	2	-3	574	550
	2	-4	564	521
	2	-5	337	277
	2	-6	333	320
	2	-7	1265	1153
	2	-8	194	148
	2	-9	1127	1035
	2	-10	227	176
	2	-14	78	94
	2	-15	396	367
	2	-16	75	57
	2	-17	24	67
5	2	-1	33	29
	2	-2	624	643
	2	-3	134	126
	2	-4	856	802
	2	-5	122	137
	2	-6	626	546
	2	-7	466	348
	2	-8	805	617
	2	-9	257	215
	2	-10	936	829
	2	-11	35	51
	2	-12	314	260
	2	-14	244	256
	2	-16	43	34
	2	-18	31	22
6	2	-1	755	749
	2	-3	714	877
	2	-4	115	106
	2	-5	63	3
	2	-7	464	457
	2	-8	82	65
	2	-9	459	423
	2	-10	68	82
	2	-11	35	70
	2	-14	111	132
	2	-15	30	53
	2	-16	125	124
	2	-17	76	114
7	2	-1	922	745
	2	-2	308	287
	2	-3	265	251
	2	-4	134	120
	2	-5	516	427
	2	-6	680	577
	2	-8	293	315
	2	-9	107	139
	2	-10	146	157
	2	-11	35	41
	2	-12	136	119
	2	-14	78	48
	2	-16	314	358
8	2	-1	466	387
	2	-2	486	452
	2	-3	608	631
	2	-4	142	134
	2	-5	686	723
	2	-6	710	722
	2	-7	438	426
	2	-9	388	436
	2	-10	140	122
	2	-11	174	146
	2	-13	430	476
	2	-15	60	71
	2	-16	64	87
	2	-17	73	70
9	2	-1	880	803
	2	-3	707	711
	2	-5	1279	1232
	2	-7	404	420
	2	-9	292	205
	2	-10	357	357
	2	-11	234	310
	2	-12	87	16
	2	-13	349	366
	2	-14	244	290
	2	-15	145	141
	2	-16	59	85
	2	-17	145	171
	2	-18	60	114
10	2	-1	625	568
	2	-2	407	391
	2	-3	170	185
	2	-4	266	257
	2	-5	113	113
	2	-7	298	257
	2	-8	206	183
	2	-9	99	100
	2	-10	447	472
	2	-11	242	282
	2	-15	150	122
	2	-17	99	105
11	2	-1	1095	854
	2	-2	426	405
	2	-4	297	302
	2	-5	229	215
	2	-6	68	62
	2	-7	418	391
	2	-8	258	269
	2	-9	357	387
	2	-10	113	115
	2	-11	723	777
	2	-13	276	256
	2	-14	356	381
	2	-16	245	274
	2	-17	162	224
	2	-19	50	51
12	2	-2	423	353
	2	-4	1173	1086
	2	-5	236	217
	2	-6	85	67
	2	-7	374	375
	2	-8	261	257
	2	-9	143	174
	2	-10	448	494
	2	-11	263	312
	2	-12	251	274
	2	-14	134	159
	2	-17	140	188
13	2	-2	71	75
	2	-3	567	457
	2	-6	377	365
	2	-7	682	732
	2	-8	315	340
	2	-9	100	97
	2	-11	154	137
	2	-12	94	89
	2	-13	226	253
	2	-15	277	309
	2	-16	159	207
	2	-17	212	315
14	2	-2	71	89
	2	-6	339	329
	2	-7	87	93
	2	-8	158	153
	2	-9	70	94
	2	-11	94	110
	2	-12	216	266
	2	-13	133	108
	2	-15	58	54
	2	-16	79	81
15	2	-1	96	107
	2	-2	139	97
	2	-4	149	149
	2	-5	234	198
	2	-6	333	350
	2	-7	137	143
	2	-8	138	159
	2	-9	117	108
	2	-10	149	174
	2	-11	363	409
	2	-13	136	125
	2	-15	52	59
	2	-16	73	108
16	2	-1	282	291
	2	-2	376	345
	2	-3	353	323
	2	-5	106	88
	2	-7	147	151
	2	-8	161	175
	2	-9	64	70
	2	-10	139	144
	2	-14	78	78
	2	-16	47	74
17	2	-1	141	132
	2	-2	94	107
	2	-3	122	148
	2	-4	296	324
	2	-5	77	101
	2	-6	89	75
	2	-7	152	184
	2	-8	162	205
	2	-9	222	212
	2	-10	121	109
	2	-11	132	126
	2	-12	61	57
	2	-13	336	353
	2	-14	127	144
18	2	-1	78	83
	2	-2	64	60
	2	-3	188	159
	2	-5	169	152
	2	-6	117	111
	2	-7	65	44
	2	-10	88	73
	2	-12	92	91
19	2	-3	79	85
	2	-6	225	251
	2	-7	203	226
	2	-10	283	321
	2	-12	98	121
	2	-13	95	150
20	2	-1	60	100
	2	-3	135	140
	2	-5	96	118
	2	-6	63	70
	2	-7	97	94
	2	-9	74	92
	2	-11	60	75
1	3	-1	91	121
	3	-2	831	981
	3	-3	441	447
	3	-4	376	338
	3	-5	207	199
	3	-6	864	751
	3	-7	381	363
	3	-8	326	129
	3	-9	521	477
	3	-10	256	278
	3	-11	140	136
	3	-12	174	165
	3	-13	129	120
	3	-15	273	210
	3	-16	94	101
2	3	-1	1172	980
	3	-2	2009	1896
	3	-3	695	651
	3	-4	412	353
	3	-5	157	130
	3	-6	739	592
	3	-7	73	78
	3	-8	177	155
	3	-10	92	93
	3	-11	37	89
	3	-12	402	347
	3	-13	174	166
	3	-14	251	171
	3	-16	151	127
	3	-17	40	31
3	3	-1	659	886
	3	-2	606	774
	3	-3	993	1033
	3	-4	673	703
	3	-5	112	124
	3	-6	304	293
	3	-7	499	485
	3	-8	310	317
	3	-9	716	642
	3	-11	568	511
	3	-13	386	411
	3	-15	199	154
	3	-16	35	71
4	3	-1	111	123
	3	-2	573	653
	3	-3	977	1045
	3	-4	677	704
	3	-5	86	82
	3	-6	31	54
	3	-7	634	663
	3	-8	668	620
	3	-9	482	424
	3	-10	322	282
	3	-11	362	343
	3	-12	343	375
	3	-14	297	267
	3	-16	157	143
5	3	-1	633	642
	3	-2	600	677
	3	-3	657	716
	3	-4	98	87
	3	-5	455	434
	3	-6	423	434
	3	-7	1311	1327
	3	-8	179	211
	3	-9	662	655
	3	-10	177	167
	3	-11	943	870
	3	-12	110	105
	3	-13	188	158
	3	-14	160	151
	3	-15	176	163
	3	-16	53	65
6	3	-1	338	314
	3	-2	323	295
	3	-3	45	24
	3	-4	535	553
	3	-6	1047	1005
	3	-7	111	121
	3	-8	427	399
	3	-9	30	74
	3	-10	651	624
	3	-11	338	327
	3	-12	218	229
	3	-13	213	220
	3	-14	163	147
	3	-15	88	75
	3	-16	169	140
	3	-17	36	63
7	3	-1	202	217
	3	-2	204	181
	3	-3	31	57
	3	-4	1320	1270
	3	-5	359	332
	3	-6	480	471
	3	-8	741	686
	3	-9	235	264
	3	-10	212	239
	3	-11	707	711
	3	-12	56	85
	3	-13	124	136
	3	-14	58	37
	3	-15	165	138
	3	-16	100	114
	3	-17	111	92
8	3	-1	726	256
	3	-2	108	128
	3	-3	428	469
	3	-5	829	830
	3	-6	195	161
	3	-7	802	730
	3	-9	784	737
	3	-11	235	215
	3	-12	193	167
	3	-13	139	110
	3	-14	116	75
	3	-15	192	123
	3	-16	225	175
	3	-17	85	66
9	3	-1	308	343
	3	-3	194	201
	3	-5	400	446
	3	-6	409	432
	3	-7	220	226
	3	-8	469	602
	3	-9	264	238
	3	-10	65	65
	3	-11	112	78
	3	-12	231	217
	3	-13	238	187
	3	-14	157	117
	3	-16	93	92
10	3	-1	193	205
	3	-2	342	327
	3	-3	376	435
	3	-5	268	254
	3	-6	174	159
	3	-7	125	128
	3	-8	361	351
	3	-9	323	288
	3	-11	445	374
	3	-12	100	100
	3	-14	119	95
	3	-15	100	78
	3	-16	195	172
	3	-17	40	48
11	3	-1	261	288
	3	-2	597	638
	3	-3	140	149
	3	-4	242	243
	3	-5	332	324
	3	-6	938	951
	3	-7	169	165
	3	-8	958	906
	3	-9	243	209
	3	-10	112	81
	3	-11	218	158
	3	-12	139	134
	3	-13	244	213
	3	-14	133	107
	3	-15	313	257
	3	-16	420	307
	3	-17	215	211
12	3	-1	267	297
	3	-2	403	410
	3	-3	64	42
	3	-4	441	466
	3	-6	285	257
	3	-7	183	197
	3	-8	172	156
	3	-9	515	427
	3	-11	99	114
	3	-12	172	144
	3	-13	396	295
	3	-14	202	134
	3	-15	149	95
	3	-16	115	114
	3	-17	195	177
13	3	-1	112	114
	3	-2	145	134
	3	-3	79	41
	3	-4	325	327
	3	-5	243	248
	3	-6	618	559
	3	-7	243	232
	3	-8	102	80
	3	-12	190	141
	3	-13	67	46
	3	-14	85	77
	3	-15	91	87
	3	-16	160	148
	3	-17	163	185
14	3	-2	305	297
	3	-3	158	171
	3	-4	366	384
	3	-5	242	270
	3	-6	266	266
	3	-7	397	393
	3	-8	216	197
	3	-9	125	120
	3	-10	122	110
	3	-11	280	248
	3	-14	321	231
	3	-15	97	69
	3	-16	94	107
15	3	-1	231	263
	3	-3	125	114
	3	-4	178	201
	3	-5	389	414
	3	-6	259	285
	3	-7	211	168
	3	-10	159	123
	3	-12	180	126
	3	-14	460	375
	3	-16	31	44
16	3	-2	336	339
	3	-4	223	240
	3	-5	94	111
	3	-6	246	254
	3	-8	174	132
	3	-9	119	101
	3	-10	163	144
	3	-11	103	134
	3	-12	154	119
	3	-13	180	144
	3	-14	243	204
	3	-15	63	51
17	3	-1	135	128
	3	-2	110	105
	3	-3	106	75
	3	-4	188	141
	3	-5	134	114
	3	-6	178	171
	3	-7	295	251
	3	-8	130	89
	3	-10	78	77
	3	-11	137	131
	3	-12	67	69
	3	-13	80	88
18	3	-2	77	79
	3	-3	93	67
	3	-4	143	150
	3	-6	316	294
	3	-7	118	120
	3	-8	328	278
	3	-11	203	179
	3	-12	88	109
	3	-13	106	114
19	3	-1	123	132
	3	-3	222	244
	3	-5	171	178
	3	-6	143	145
	3	-7	181	180
	3	-9	161	152
	3	-11	80	72
	3	-12	225	290
20	3	-3	118	157
	3	-4	115	136
	3	-5	38	57
	3	-6	124	140
	3	-8	130	150
	3	-10	180	233
1	4	-1	224	212
	4	-2	78	96
	4	-3	87	133
	4	-4	437	366
	4	-5	33	27
	4	-6	458	405
	4	-8	100	88
	4	-9	77	91
	4	-10	77	68
	4	-13	237	236
	4	-14	76	91
	4	-15	106	134
2	4	-1	120	125
	4	-2	672	560
	4	-3	187	156
	4	-4	66	73
	4	-5	242	199
	4	-6	488	402
	4	-7	646	536
	4	-8	567	451
	4	-9	236	196
	4	-10	319	305
	4	-12	256	285
	4	-13	102	136
	4	-15	293	285
	4	-16	92	138
3	4	-1	654	684
	4	-3	183	161
	4	-4	330	291
	4	-5	251	201
	4	-6	116	123
	4	-7	978	912
	4	-8	181	194
	4	-9	360	393
	4	-11	221	252
	4	-12	32	118
	4	-13	113	138
	4	-14	197	204
	4	-15	277	262
	4	-16	31	75
4	4	-1	72	42
	4	-2	686	625
	4	-3	387	357
	4	-4	366	343
	4	-5	129	110
	4	-6	146	168
	4	-7	28	68
	4	-8	30	26
	4	-9	48	78
	4	-10	237	254
	4	-12	82	70
	4	-13	25	29
	4	-14	127	150
	4	-15	129	162
5	4	-1	865	821
	4	-2	275	253
	4	-3	465	468
	4	-4	499	441
	4	-5	422	338
	4	-6	324	283
	4	-7	250	211
	4	-8	30	58
	4	-9	366	320
	4	-10	213	243
	4	-11	142	158
	4	-14	68	97
	4	-15	126	150
	4	-16	25	59
6	4	-1	77	64
	4	-2	147	140
	4	-3	531	471
	4	-4	527	457
	4	-5	1068	880
	4	-6	304	247
	4	-7	663	562
	4	-8	383	373
	4	-9	173	179
	4	-10	766	720
	4	-11	41	80
	4	-12	317	311
	4	-14	109	97
	4	-16	216	254
	4	-17	48	86
7	4	-1	284	314
	4	-2	783	664
	4	-3	176	202
	4	-4	35	5
	4	-5	127	145
	4	-6	1046	917
	4	-8	210	234
	4	-9	114	136
	4	-10	33	71
	4	-12	120	149
	4	-13	324	404
	4	-14	97	130
	4	-15	138	170
	4	-16	93	126
8	4	-1	58	41
	4	-2	434	437
	4	-3	1232	1055
	4	-4	66	63
	4	-5	535	489
	4	-6	721	658
	4	-7	408	389
	4	-11	194	201
	4	-12	117	149
	4	-13	49	40
	4	-14	91	67
	4	-15	120	143
	4	-16	248	342
9	4	-1	150	147
	4	-2	720	692
	4	-3	484	455
	4	-4	168	155
	4	-5	203	217
	4	-6	51	26
	4	-7	418	465
	4	-8	191	214
	4	-10	219	237
	4	-13	84	121
10	4	-1	187	196
	4	-2	175	144
	4	-3	81	65
	4	-4	262	275
	4	-5	51	69
	4	-6	104	127
	4	-7	84	110
	4	-8	218	243
	4	-9	560	568
	4	-10	330	387
	4	-11	74	87
	4	-12	184	256
	4	-13	58	70
	4	-14	182	165
	4	-15	89	115
11	4	-2	150	124
	4	-3	273	259
	4	-4	945	867
	4	-5	391	379
	4	-6	393	462
	4	-7	194	215
	4	-8	275	270
	4	-10	290	311
	4	-11	127	172
	4	-12	254	318
	4	-14	126	142
12	4	-1	85	80
	4	-2	162	148
	4	-3	352	404
	4	-4	238	257
	4	-5	205	243
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Table 4 (*cont.*)

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	5	-12	108	142
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	5	-14	24	31
	5	-15	153	170
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	10	-7	119	154
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	10	-7	87	90
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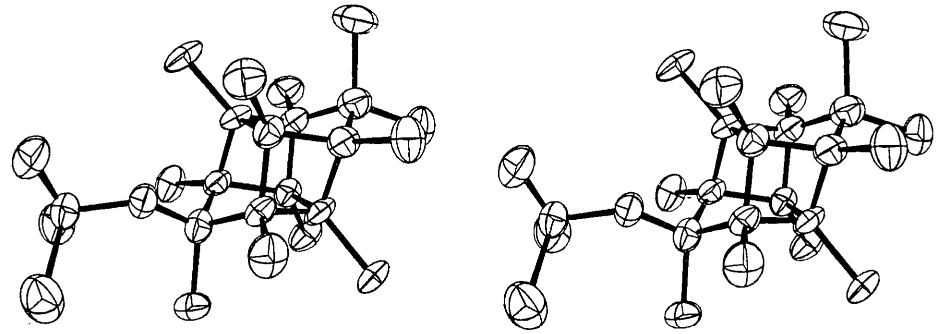

Fig. 5. Stereoscopic drawing of the structure depicting the relative magnitude of the thermal ellipsoids. Prepared with the computer program by Johnson (1965) and an incremental digital $X-Y$ plotter.

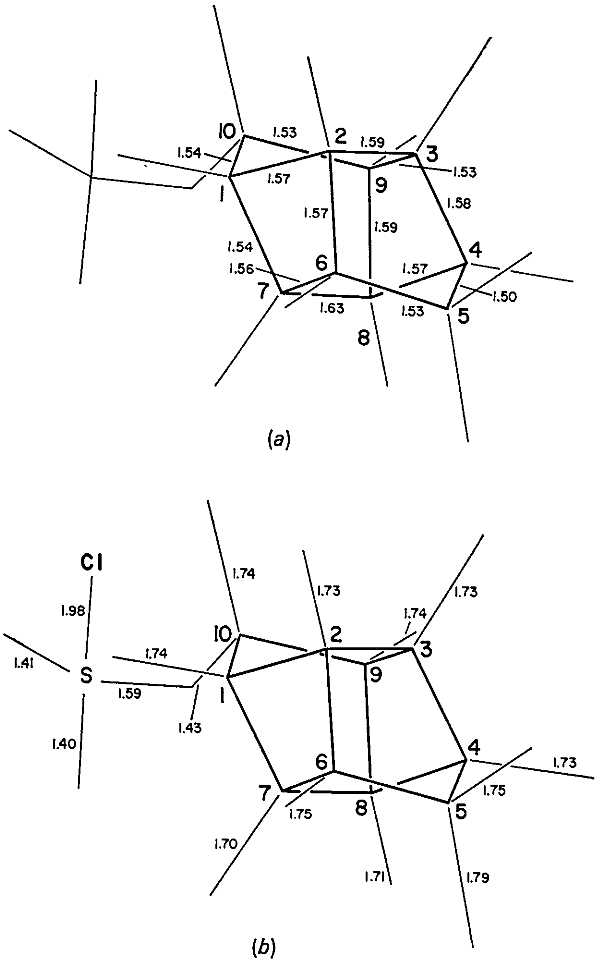

Fig. 6. Bond distances (*a*) in the skeleton. (*b*) from the skeleton.

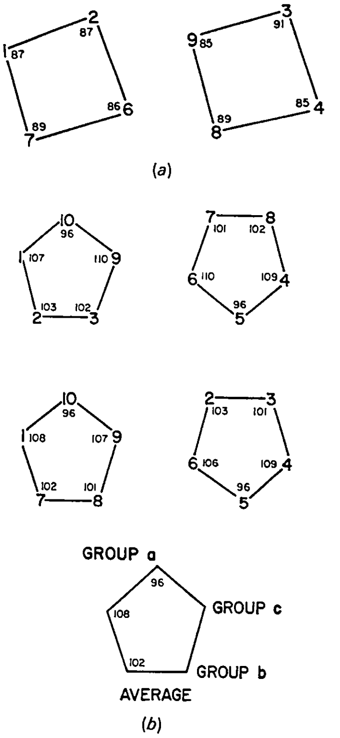

Fig. 7. Bond angles in the ring systems. (*a*) Cyclobutane rings. (*b*) Cyclopentane rings. For three groups into which the skeleton carbon atoms are classified, see text.

The carbon atoms in the skeleton can again be divided into the three groups mentioned previously, with respect to the angles in the rings. The three Cl–C–C angles around each carbon atom belonging to group (*b*) are about 121, 121 and 117°. The larger two angles are always formed by the chlorine and the carbon atoms in the cyclobutane rings to which the central carbon belongs; the third and smaller angle involves the next neighbor carbon in the cyclopentane ring. For the carbon atoms in group (*c*), the three Cl–C–C angles are similar with no systematic variation observable. The angles around the apex carbons, group (*a*), are also normal; the deviations from the usual tetrahedral angle can be easily explained by the narrow intra-ring angles. The C–Cl distances are also normal with the average value of 1·74 Å.

The shape and dimensions of the chlorosulfonate group are shown in Fig. 9. The large O(I)–S–O(II) angle of 123° can be attributed to the localized double bond character for these two oxygen atoms, because of the presence of a chlorine atom and the bridge formation. The large angle around the bridge oxygen O(10) is no doubt due to the steric hindrance caused by the two bulky groups attached to this atom.

The anisotropic temperature factors listed in Table 3 were decoded into their thermal vibrational ellipsoids. The cage skeleton carbon atoms exhibit relatively isotropic thermal motion with amplitudes smaller than those of the chlorine atoms which in addition exhibit more anisotropic motion. The greatest vibrational amplitudes and anisotropy of motion are experienced by the chlorosulfonate group which seems to be in torsional vibration about the O(10)–S bridging bond. These results are also evident in the stereoscopic drawing (Fig. 5).

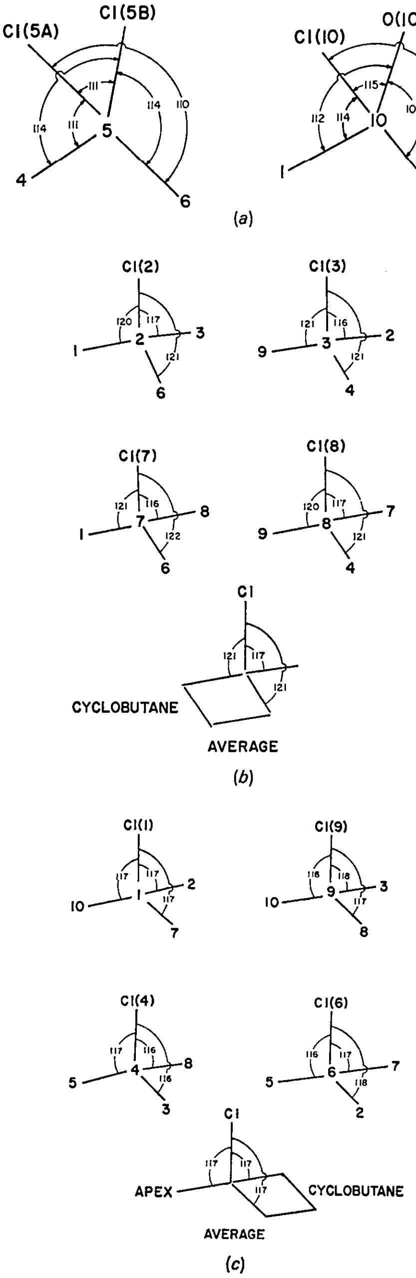

Fig. 8. Angles outside of the carbon skeleton. (*a*) Around apex carbons, group (*a*); (*b*), group (*b*); (*c*), group (*c*).

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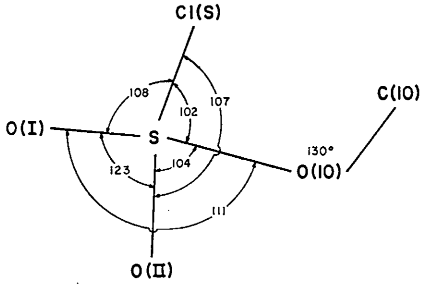

Fig. 9. Angles in the chlorosulfonate group.