863. *The Structure of Isoclovene: X-Ray Analysis of the Hydrochloride and Hydrobromide.*

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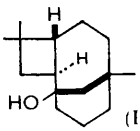
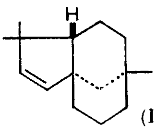
The structure and stereochemistry of the tricyclic sesquiterpene isoclovene, $C_{15}H_{24}$, have been completely determined (IV) by *X*-ray analysis of the hydrochloride. The crystals are monoclinic, space group $P2_1$, with two molecules per unit cell. The ambiguity of a false symmetry centre between the phase determining atoms was gradually overcome by the use of three-dimensional methods. In the final results the discrepancy *R* over the 973 observed structure factors is 12·5%. The bond lengths and angles (Figs. 3 and 4) have average standard deviations of about 0·04 Å and 2°.

ISOCLOVENE, a tricyclic sesquiterpene of hitherto virtually unknown constitution, was first isolated by Henderson, McCrone, and Robertson [1] from the reaction mixture produced by treating β-caryophyllene alcohol (caryolan-1-ol) with phosphorus pentoxide. The other main product was believed at that time to be clovene. Subsequent chemical work,[2] when coupled with the results of Robertson and Todd's *X*-ray crystallographic analysis of the halides of β-caryophyllene alcohol,[3] has enabled structures (I) and (II) to be ascribed to β-caryophyllene alcohol and clovene, respectively.


(I)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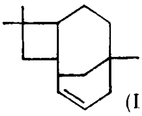
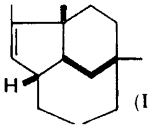

(III)


(IV)

Lutz and Reid [4] reinvestigated the dehydration of β-caryophyllene alcohol and showed that the mixture of oils obtained consisted of isoclovene and, not clovene, but a new isomer which they called pseudoclovene and for which they proposed structure (III). Although the latter workers do not appear to have studied isoclovene in any great detail, further investigations have been carried out by Money [5] who, on the basis of the infrared spectrum of isoclovene (peaks at 795s and 840m cm.$^{-1}$), inferred that the known ethylenic linkage was secondary-tertiary. In addition, Kuhn–Roth oxidation showed that the molecule contained three methyl groups.

The constitution of isoclovene, however, and its relation to clovene and pseudoclovene have remained obscure and further chemical work is rendered difficult by the ease with which unexpected molecular transformations occur in this series. On the other hand, isoclovene is characterised by a beautifully crystalline hydrochloride and hydrobromide

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[1] Henderson, McCrone, and Robertson, *J.*, 1929, 1368.

[2] Barton, Bruun, and Lindsey, *J.*, 1952, 2210; Aebi, Barton, and Lindsey, *J.*, 1953, 3124.

[3] Robertson and Todd, *Chem. and Ind.*, 1953, 437; *J.*, 1955, 1254.

[4] Lutz and Reid, *J.*, 1954, 2265.

[5] Money, B.Sc. Thesis, Glasgow, 1957.

from which the pure hydrocarbon can be regenerated.[1] An *X*-ray investigation of these crystals was therefore undertaken, and from the results of this study, which are described below, the structure and stereochemistry of isoclovene can now be given unambiguously as (IV). The mechanism of the conversion of the alcohol (I) into isoclovene (IV) represents an unusual and difficult problem which cannot be completely solved on the basis of the known chemical facts. Some possible routes have already been discussed by Professor Barton in terms of carbonium-ion mechanism.[6]

Our *X*-ray work commenced with a study of the isomorphous isoclovene hydrochloride and hydrobromide, but owing to lack of resolution in the two-dimensional projections and to the complications of the isomorphous substitution method [7] in a non-centric space group ($P2_1$) this work was later abandoned in favour of a three-dimensional study of the hydrochloride by the heavy-atom method.[8]

The fundamental difficulty of a false symmetry centre between the two phase-determining atoms was, of course, again encountered. However, a selection of possible atomic sites was obtained by direct triple Fourier synthesis based solely on the chlorine phases and also by the application of superposition methods [9] to sharpened Patterson syntheses. At this stage a consideration of cell dimensions and van der Waals distances enabled the regions probably occupied by the two molecules to be delineated. A judicious selection of the more prominent peaks from what appeared to be one molecular site, and the gradual incorporation of these atoms into the phasing calculations, enabled the troublesome pseudo-symmetry to be overcome in the later electron-density distributions.

The refinement of the structure then proceeded by standard methods. Seven successive three-dimensional Fourier syntheses were carried out, during which the discrepancy *R* dropped from 47·4% to 20·4%. This was followed by twelve cycles of least-squares refinement, during which *R* decreased further to a final value of 12·5% over the 973 observed structure factors (Table 10). The course of the analysis is summarised in Table 2.

The final electron density distribution over one molecule is shown in Fig. 1. In this view along the *b* axis the ring systems are not clearly visible, but a roughly perspective drawing indicating the stereochemistry, and also the numbering system used in the later Tables, is given in Fig. 2. The molecular dimensions and estimated standard deviations (which average about 0·04 Å and 2°) are given in detail in Tables 6 and 7 but are summarised in Figs. 3 and 4.

The molecule is a very compact structure, consisting of a five-membered, a six-membered, and a seven-membered ring fused together, each ring retaining its preferred conformation. The five-membered ring is hinge-shaped, the six-membered ring has the chair conformation; the seven-membered ring is also in chair form but obviously distorted because all the angles are consistently greater than tetrahedral, the mean value being 116·5° (Fig. 4). This ring may be described as somewhat flattened, a situation probably due to steric repulsion as well as to ring strain. The increase in bond angles may be compared with those observed in cyclononylamine hydrobromide [10] and in 1,6-*trans*-diaminocyclodecane dihydrochloride [11] where similar large values for the ring angles have been found. In isoclovene, however, the fusion with neighbouring rings will cause additional strain. For example, in an ideal structure the non-bonded distance C(6) $\cdots$ C(10) would be only about 2·6 Å, whereas the distance found is 3·38 Å (Table 8).

The five-membered and six-membered rings are *cis*-fused, as are the five-membered and seven-membered rings. The six-membered and seven-membered rings are linked by a methylene bridge *cis* relative to C(14) and chlorine. The average carbon–carbon

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[6] Clunie and Robertson, *Proc. Chem. Soc.*, 1960, 82.

[7] Robertson, *J.*, 1936, 1195; Bokhoven, Schoone, and Bijvoet, *Acta Cryst.*, 1951, **4**, 275.

[8] Robertson and Woodward, *J.*, 1937, 219; 1940, 36.

[9] Buerger, *Acta Cryst.*, 1951, **4**, 531.

[10] Bryan and Dunitz, *Helv. Chim. Acta*, 1960, **43**, 3.

[11] Huber-Buser and Dunitz, *Helv. Chim. Acta*, 1960, **43**, 760.

bond distance is 1·55 Å, with a maximum spread of 0·05 Å (average standard deviation, 0·04 Å). The quasi-axial carbon–chlorine bond of 1·86 Å can perhaps be considered as significantly longer than the expected value of 1·78 Å obtained from a consideration of atomic radii. Values as high as 1·85 Å and 1·84 Å for equatorial carbon–chlorine bonds in pentachlorocyclohexene have been reported by Pasternak.[12] For an axial carbon–chlorine bond in 1,2,3,4-tetrachloro-1,2,3,4-tetrahydronaphthalene a value of 1·81 Å has been cited.[13] The bond lengthening found in the present case may be simply a steric effect due to the proximity of two methyl and one methylene groups.

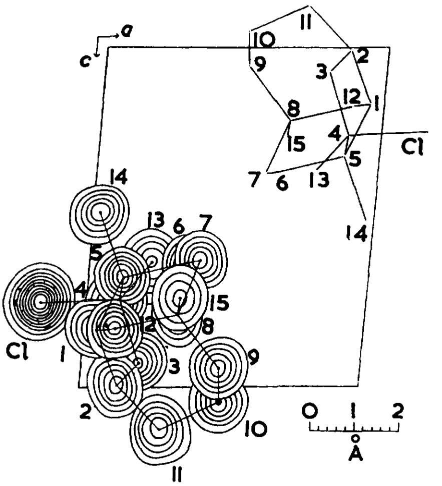

FIG. 1. Final electron-density distribution as superimposed contour sections drawn parallel to (010). Scale, carbon 1 eÅ^{-3}, chlorine 2 eÅ^{-3} per line. Atoms are drawn on section levels instead of accurately through atomic centres.

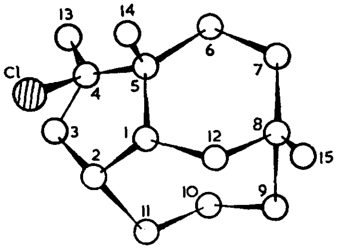

FIG. 2. Stereochemistry of isoclovene hydrochloride.

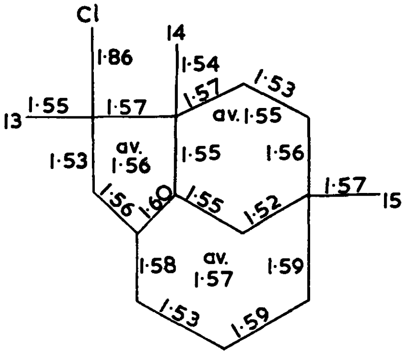

FIG. 3. Bond lengths (Å).

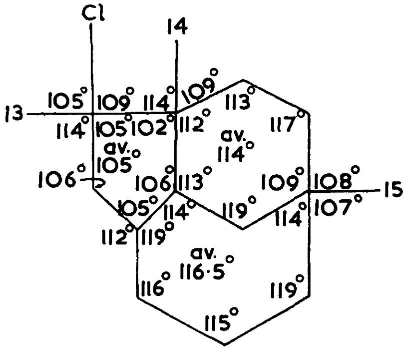

FIG. 4. Bond angles.

EXPERIMENTAL

In an attempt to employ the isomorphous substitution method fairly extensive measurements were made on isoclovene hydrobromide, including structure factor determinations for the three axial zones. As this work was later abandoned in favour of a three-dimensional

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[12] Pasternak, *Acta Cryst.*, 1951, **4**, 316.
[13] Lasheen, *Acta Cryst.*, 1952, **5**, 593.

study of isoclovene hydrochloride, only elementary crystal data for the hydrobromide are given here.

Crystal Data.—Isoclovene hydrobromide, $C_{15}H_{25}Br$; M, 285·3; m. p. 74°; d, calc. 1·330, found 1·323 (flotation, zinc chloride solution). Monoclinic, $a = 6{\cdot}48 \pm 0{\cdot}01$, $b = 13{\cdot}81 \pm 0{\cdot}04$, $c = 7{\cdot}96 \pm 0{\cdot}01$ Å, $\beta = 91{\cdot}9°$. Absent spectra, (0k0) when k is odd. Space group, $P2_1$ ($C_2{}^2$) or $P2_1/m$ ($C_{2h}{}^2$). $P2_1/m$ must be rejected because the compound is optically active. Two molecules per unit cell. Volume of the unit cell = 712 Å³. Absorption coefficient for X-rays, ($\lambda = 1{\cdot}542$ Å) $\mu = 38{\cdot}6$ cm.$^{-1}$. Total number of electrons per unit cell = $F(000) = 300$.

Small but well-formed prismatic needles elongated along a were obtained by slow crystallisation from acetone solutions. From two-dimensional Patterson and Fourier syntheses of the structure factors obtained from the axial zones, the following co-ordinates were obtained for bromine:

$$x/a = 0{\cdot}177,\ y/b = 0{\cdot}250,\ z/c = 0{\cdot}242.$$

Isoclovene hydrochloride, $C_{15}H_{25}Cl$; M, 240·8; m. p. 87°; d, calc. 1·152, found 1·151 (flotation, zinc chloride solution). Monoclinic, $a = 6{\cdot}35 \pm 0{\cdot}01$, $b = 13{\cdot}91 \pm 0{\cdot}04$, $c = 7{\cdot}89 \pm 0{\cdot}02$ Å, $\beta = 95{\cdot}3°$. Absent spectra, (0k0) when k is odd. Space group, $P2_1$ ($C_2{}^2$) or $P2_1/m$ ($C_{2h}{}^2$). $P2_1/m$ must be rejected because the compound is optically active. Two molecules per unit cell. Volume of the unit cell = 694 Å³. Absorption coefficient for X-rays ($\lambda = 1{\cdot}542$ Å) $\mu = 22{\cdot}2$ cm.$^{-1}$. Total number of electrons per unit cell = $F(000) = 264$. Crystal habit is similar to that of the hydrobromide.

Experimental Measurements.—Intensities were estimated visually from equi-inclination Weissenberg photographs taken with Cu-$K\alpha$ radiation. The layer lines surveyed and the dimensions of the crystal specimens are given in Table 1. As the cross-sections were small and nearly square no attempt was made to apply systematic absorption corrections.

TABLE 1.

Reflections surveyed.

Layer lines	Crystal cross section × length along rotation axis
(0kl) — (5kl)	0·2 × 0·2 × 0·4 mm.³
(h0l)	0·4 × 0·3 × 0·15 mm.³
(hk0) — (hk4)	0·25 × 0·3 × 0·2 mm.³

Each reflection was estimated at least twice by using a different step wedge before the averaging process was performed. In all 1485 reflections were measured out of a possible 2725 (54·5%) and this yielded a total of 973 independent structure factors (Table 10), after the usual Lorentz, polarisation, and rotation correction factors had been applied. The absolute

TABLE 2.

Course of analysis.

Operation	Data used			R (%)	$\Sigma w\Delta^2$
3D Patterson function	973 F_o			—	—
3D minimum function	973 F_o			—	—
1st 3D F_o synthesis	Cl	885 F_o		47·4	—
2nd ,, ,,	Cl + 7C	614 F_o		40·2	—
3rd ,, ,,	Cl + 11C	776 F_o		38·7	—
4th ,, ,,	Cl + 13C	788 F_o		33·8	—
5th ,, ,,	Cl + 15C	882 F_o		27·9	—
6th ,, ,,	Cl + 15C	973 F_o		23·4	—
7th ,, ,,	Cl + 15C	973 F_o		20·4	—
1st least-squares cycle	Isotropic temp. factors	600 F_o		18·9	—
2nd ,, ,,	,, ,, ,,	600 F_o		16·4	—
3rd ,, ,,	,, ,, ,,	600 F_o		15·3	—
4th ,, ,,	,, ,, ,,	800 F_o		16·7	—
5th ,, ,,	Anisotropic temp. factors	973 F_o		15·6	259
6th ,, ,,	,, ,, ,,	,,		14·6	221
7th ,, ,,	,, ,, ,,	,,		13·7	209
8th ,, ,,	,, ,, ,,	,,		13·3	194
9th ,, ,,	,, ,, ,,	,,	H atoms	13·0	191
10th ,, ,,	,, ,, ,,	,,	,,	12·9	185
11th ,, ,,	,, ,, ,,	,,	,,	12·6	179
12th ,, ,,	,, ,, ,,	,,	,,	12·5	179

TABLE 3.

Atomic co-ordinates and anisotropic temperature factors.

Atom	x/a	y/b	z/c	β_{11}	β_{22}	β_{33}	β_{23}	β_{31}	β_{12}
C(1)	−0·0440	−0·0470	0·1723	0·0326	0·0069	0·0222	−0·0043	0·0174	−0·0053
C(2)	−0·1275	−0·1055	0·0074	0·0424	0·0097	0·0261	0·0002	0·0306	0·0063
C(3)	−0·2027	−0·2045	0·0762	0·0394	0·0064	0·0279	−0·0010	−0·0011	0·0009
C(4)	−0·1137	−0·2110	0·2596	0·0406	0·0062	0·0255	−0·0009	−0·0007	0·0002
C(5)	−0·1179	−0·1048	0·3267	0·0304	0·0075	0·0221	0·0003	0·0094	0·0103
C(6)	−0·3516	−0·0806	0·3671	0·0200	0·0107	0·0254	0·0082	0·0031	−0·0036
C(7)	−0·3972	0·0274	0·3705	0·0407	0·0088	0·0247	0·0007	0·0093	0·0071
C(8)	−0·3316	0·0869	0·2131	0·0416	0·0069	0·0289	−0·0007	0·0198	−0·0021
C(9)	−0·4947	0·0734	0·0553	0·0333	0·0092	0·0309	0·0047	0·0018	−0·0073
C(10) ...	−0·5039	−0·0274	−0·0402	0·0387	0·0085	0·0225	−0·0048	−0·0050	0·0002
C(11) ...	−0·2980	−0·0578	−0·1193	0·0438	0·0099	0·0219	0·0040	−0·0054	−0·0031
C(12) ...	−0·1069	0·0607	0·1744	0·0331	0·0067	0·0255	−0·0011	0·0099	−0·0025
C(13) ...	−0·2208	−0·2893	0·3659	0·0514	0·0086	0·0329	0·0095	−0·0101	−0·0036
C(14) ...	0·0228	−0·0878	0·4887	0·0342	0·0102	0·0267	0·0006	−0·0119	−0·0017
C(15) ...	−0·3345	0·1960	0·2633	0·0615	0·0084	0·0384	−0·0027	0·0060	−0·0019
Cl	0·1650	−0·2512	0·2499	0·0341	0·0087	0·0347	−0·0043	0·0049	0·0076

TABLE 4.

Approximate hydrogen atom co-ordinates.

Atom no.	x/a	y/b	z/c	Atom no.	x/a	y/b	z/c
H(1)	0·116	−0·013	0·164	H(11′)	−0·252	−0·130	−0·154
H(2)	−0·030	−0·058	−0·069	H(12)	−0·153	0·109	0·049
H(3)	−0·140	−0·269	−0·001	H(12′)	0·022	0·105	0·226
H(3′)	−0·352	−0·244	0·067	H(13)	−0·364	−0·248	0·347
H(6)	−0·448	−0·133	0·295	H(13′)	−0·159	−0·361	0·350
H(6′)	−0·345	−0·113	0·492	H(13″)	−0·162	−0·276	0·497
H(7)	−0·530	0·058	0·441	H(14)	−0·119	−0·074	0·560
H(7′)	−0·288	0·067	0·449	H(14′)	−0·030	−0·138	0·586
H(9)	−0·438	0·001	−0·049	H(14″)	0·132	−0·055	0·492
H(9′)	0·391	0·025	0·100	H(15)	−0·335	0·241	0·150
H(10)	0·362	−0·073	−0·077	H(15′)	−0·474	0·218	0·333
H(10′)	−0·464	−0·099	0·087	H(15″)	−0·200	0·208	0·345
H(11)	−0·348	−0·018	−0·228				

TABLE 5.

Standard deviations in the positional co-ordinates.

Atom	$\sigma(x)$ (Å)	$\sigma(y)$ (Å)	$\sigma(z)$ (Å)	$\sigma(r)$ (Å) *	Atom	$\sigma(x)$ (Å)	$\sigma(y)$ (Å)	$\sigma(z)$ (Å)	$\sigma(r)$ (Å) *
C(1)	0·012	0·012	0·012	0·023	C(9)	0·013	0·014	0·015	0·027
C(2)	0·012	0·013	0·015	0·027	C(10) ...	0·013	0·012	0·014	0·026
C(3)	0·013	0·014	0·013	0·027	C(11) ...	0·014	0·013	0·015	0·028
C(4)	0·013	0·013	0·013	0·026	C(12) ...	0·012	0·013	0·013	0·025
C(5)	0·011	0·012	0·012	0·023	C(13) ...	0·015	0·016	0·015	0·030
C(6)	0·010	0·013	0·015	0·026	C(14) ...	0·013	0·015	0·015	0·029
C(7)	0·013	0·013	0·014	0·027	C(15) ...	0·016	0·017	0·016	0·033
C(8)	0·012	0·013	0·013	0·025	Cl	0·003	0·004	0·005	0·008

* Factor of 2 included to allow for non-centrosymmetry.

TABLE 6.

Bond lengths (Å) with standard deviations.

C(1)–C(2)	1·600 ± 0·036	C(4)–C(5)	1·570 ± 0·035	C(8)–C(9)	1·590 ± 0·037
C(1)–C(5)	1·547 ± 0·033	C(4)–C(13) ...	1·550 ± 0·040	C(8)–C(12) ...	1·518 ± 0·036
C(1)–C(12) ...	1·550 ± 0·034	C(5)–C(6)	1·568 ± 0·035	C(8)–C(15) ...	1·568 ± 0·041
C(2)–C(3)	1·563 ± 0·038	C(5)–C(14) ...	1·540 ± 0·038	C(9)–C(10) ...	1·590 ± 0·038
C(2)–C(11) ...	1·583 ± 0·039	C(6)–C(7)	1·531 ± 0·038	C(10)–C(11) ...	1·533 ± 0·039
C(3)–C(4)	1·527 ± 0·037	C(7)–C(8)	1·561 ± 0·037	C(4)–Cl	1·860 ± 0·027

scale was obtained at first approximately and later more accurately by correlation with the calculated values. From a study of about 400 reflections observed more than once the mean standard deviation of the structure amplitudes was found to be about $\sigma|F_i| = 0{\cdot}09|F_i|$.

Analysis of the Structure.—Approximate halogen positions had already been obtained from

TABLE 7.

Bond angles with standard deviations.

C(2)–C(1)–C(5)	105·7° ± 1·9°	C(5)–C(4)–C(13)	117·7° ± 2·2°	C(7)–C(8)–C(9)	111·6° ± 2·1°
C(2)–C(1)–C(12)	113·9 ± 1·9	C(13)–C(4)–Cl	104·7 ± 1·8	C(7)–C(8)–C(12)	108·6 ± 2·1
C(5)–C(1)–C(12)	115·1 ± 1·9	C(1)–C(5)–C(4)	102·4 ± 1·9	C(7)–C(8)–C(15)	107·9 ± 2·2
C(1)–C(2)–C(3)	105·2 ± 2·0	C(1)–C(5)–C(6)	111·8 ± 1·9	C(9)–C(8)–C(12)	113·5 ± 2·1
C(1)–C(2)–C(11)	119·2 ± 2·1	C(1)–C(5)–C(14)	112·6 ± 2·0	C(9)–C(8)–C(15)	107·2 ± 2·1
C(3)–C(2)–C(11)	112·1 ± 2·1	C(4)–C(5)–C(14)	113·8 ± 2·0	C(15)–C(8)–C(12)	107·8 ± 2·2
C(2)–C(3)–C(4)	105·9 ± 2·1	C(6)–C(5)–C(4)	107·5 ± 1·9	C(8)–C(9)–C(10)	118·8 ± 2·1
C(3)–C(4)–C(5)	104·5 ± 2·0	C(6)–C(5)–C(14)	108·5 ± 2·0	C(9)–C(10)–C(11)	114·9 ± 2·2
C(3)–C(4)–C(13)	113·5 ± 2·2	C(5)–C(6)–C(7)	113·3 ± 2·1	C(2)–C(11)–C(10)	115·6 ± 2·2
C(3)–C(4)–Cl	106·9 ± 1·8	C(6)–C(7)–C(8)	116·7 ± 2·2	C(1)–C(12)–C(8)	118·7 ± 2·1
C(5)–C(4)–Cl	109·2 ± 1·6				

TABLE 8.

Some intramolecular non-bonded distances (Å).

C(1)...Cl	= 3·18	C(3)...C(10)	= 3·23	C(7)...C(14)	= 3·21
C(1)...C(13)	= 3·89	C(4)...C(14)	= 2·61	C(7)...C(10)	= 3·36
C(1)...C(7)	= 2·98	C(5)...Cl	= 2·80	C(8)...C(11)	= 3·31
C(1)...C(10)	= 3·31	C(5)...C(13)	= 2·67	C(9)...C(15)	= 2·54
C(2)...Cl	= 3·30	C(5)...C(11)	= 3·70	C(10)...C(12)	= 3·21
C(2)...C(13)	= 3·87	C(5)...C(8)	= 3·10	C(11)...C(12)	= 3·04
C(2)...C(14)	= 3·87	C(6)...C(12)	= 2·97	C(12)...C(15)	= 2·49
C(2)...C(9)	= 3·44	C(6)...C(10)	= 3·38	C(12)...C(14)	= 3·30
C(3)...Cl	= 2·73	C(6)...C(14)	= 2·52	C(13)...C(14)	= 3·32
C(3)...C(13)	= 2·57	C(6)...C(13)	= 3·02	C(13)...Cl	= 2·71
C(3)...C(14)	= 3·84	C(7)...C(15)	= 2·53	C(14)...Cl	= 3·11

TABLE 9.

Some intermolecular distances (Å).

C(1)...C(9_{I})	3·856 ± 0·036	C(14)...C(11_{II})	3·880 ± 0·041
C(1)...C(10_{I})	3·495 ± 0·035	C(15)...$\mathrm{Cl}_{\mathrm{III}}$	3·960 ± 0·034
Cl...C(6_{I})	3·922 ± 0·027	C(9)...C(3_{IV})	3·734 ± 0·038
Cl...C(13_{I})	3·959 ± 0·031		

(The subscripts refer to the following positions: I $1 + x, y, z$. II $x, y, 1 + z$. III $1 - x, \frac{1}{2} + y, \bar{z}$. IV $\bar{x}, \frac{1}{2} + y, \bar{z}$.)

our preliminary two-dimensional work on isoclovene hydrobromide. For the hydrochloride a three-dimensional sharpened Patterson function incorporating all the observed structure factors was prepared in sections parallel to the *b* axis. A completely resolved chlorine–chlorine vector peak was not at first obtained, but by the application of superposition methods[9] unambiguous co-ordinates were finally derived, and refined by later Fourier syntheses.

The fundamental difficulty of the analysis, in space group $P2_1$, is, of course, due to the fact that the two phase-determining chlorine atoms are related by a two-fold screw axis and false symmetry is thereby introduced to the electron-density distribution derived from these phases. This difficulty was further aggravated in the present case by the discovery that the z-co-ordinate of the chlorine atom was extremely close to the special value of 0·250, with the result that the chlorine atoms made almost zero contribution to certain sets of reflections, with the consequent introduction of additional spurious symmetry in certain projections.

No very direct method is known for overcoming these difficulties and recourse must be had to a knowledge of what is chemically reasonable in attempting to interpret the results. By using almost the full three-dimensional data, a minimum function was prepared and also a Fourier synthesis based solely on the chlorine phases. From a careful comparison of these two functions the peaks that were most likely to represent atomic sites were selected. At this stage each peak was duplicated by the false symmetry, but the selection of true sites was helped by the apparently compact nature of the molecules and the comparatively large gaps between them (compare Figs. 5 and 6). At first only seven atoms were included in the phasing calculations in addition to chlorine, but a notable decrease in the discrepancy factor R indicated that a substantially correct choice had been made. In later stages of the work many false peaks still remained, but these were generally of smaller height and more irregular in outline than the peaks representing genuine atoms. The five-membered and six-membered rings first clearly emerged as definite molecular features. It at first appeared possible that the remaining ring might also be six-membered, but the discovery of atom C(10) in a mirror image position to C(9) across the pseudo-symmetry centre at ($\frac{1}{4}$00) established the true nature of this ring and effectively completed the structure determination.

The course of the analysis is summarised in Table 2. For the 6th and 7th F_0 synthesis the "*n* shift" rule of Shoemaker and others[14] with $n = 1{\cdot}6$ was applied. After the 7th F_0 synthesis, refinement was continued by the method of least squares, only the diagonal elements of the matrix of normal equations being used. The isotropic temperature factors used in the initial cycles were $B = 3{\cdot}5$ Å^2 for carbon and $B = 4{\cdot}4$ Å^2 for chlorine, with the scattering factors of Berghuis and others[15] for carbon, and of James and Brindley[16] for chlorine. In the last four cycles hydrogen contributions were included in the structure factors calculated by the least-squares programme, but the hydrogen co-ordinates were not refined. They were assumed to lie in the geometrically required positions, with a carbon–hydrogen bond length of 1·05 Å. The methyl-hydrogen atoms were assumed to lie in positions staggered with respect to the substituents on the adjacent carbon atoms.

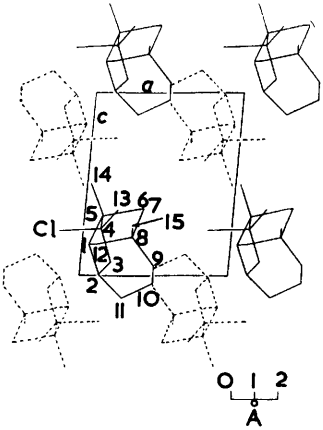

FIG. 5. The structure projected on (010).

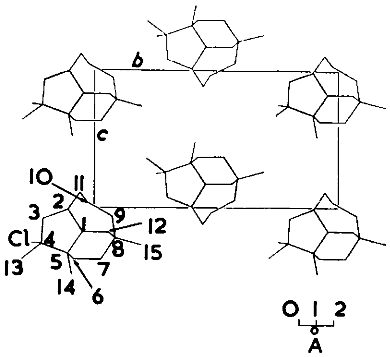

FIG. 6. The structure projected on (100).

The weighting system used for the least-squares analysis was as follows:

$$\sqrt{w} = 1 \text{ if } |F_0| \leqslant 8|F_{\text{min.}}|$$

$$\sqrt{w} = \frac{8|F_{\text{min.}}|}{|F_0|} \text{ if } |F_0| > 8|F_{\text{min.}}|$$

The value of $\sum w\Delta^2$, the quantity minimised in the least-squares process, decreased steadily to 185 in the 10th cycle and then remained almost constant. Together with the almost constant value of *R* during the last few cycles, this indicated that refinement within the scope of the present data and the methods used was complete.

The final co-ordinates and anisotropic temperature factors are given in Tables 3 and 4. The thermal parameters listed are derived from the 12th least squares cycle, by using the programme of Rollett[17] and the form

$$T = 2^{-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{23}kl + \beta_{31}lh + \beta_{12}hk)}$$

where $\beta_{11} = 1{\cdot}4427B_{11}$, etc. Equivalent isotropic temperature factors deduced by the method of Rossmann and others[18] are $B = 4{\cdot}4$ Å^2 for the average carbon atom and $B = 4{\cdot}45$ Å for the chlorine atom.

[14] Shoemaker, Donohue, Schomaker, and Corey, *J. Amer. Chem. Soc.*, 1950, **72**, 2328.

[15] Berghuis, Haanappel, Potters, Loopstra, MacGillavry, and Veenendaal, *Acta Cryst.*, 1955, **8**, 478.

[16] James and Brindley, *Phil. Mag.*, 1932, **12**, 81.

[17] Rollett, "Computing Methods and the Phase Problem in *X*-Ray Crystal Analysis," ed. Pepinsky, Robertson, and Speakman, Pergamon Press, Oxford, 1961, p. 87.

[18] Rossmann, Jacobson, Hirshfeld, and Lipscomb, *Acta Cryst.*, 1959, **12**, 530.

TABLE 10.

Observed and calculated structure factors.

h	k	l	$10\|F_o\|$	$10\|F_c\|$	α°
0	2	0	256	316	286
	4		289	351	322
	6		518	513	174
	8		67	64	161
	10		45	45	277
	12		31	40	288
	14		15	25	156
0	0	1	268	229	0
	1		683	633	53
	2		170	172	357
	3		435	445	189
	4		154	149	63
	5		130	131	338
	6		219	194	267
	7		259	280	219
	8		85	97	242
	9		91	101	8
	10		105	119	254
	11		36	38	122
	13		30	36	327
0	0	2	291	224	180
	1		291	199	208
	2		419	446	332
	3		291	279	206
	4		254	236	196
	5		117	113	142
	6		130	116	347
	7		119	129	320
	8		50	75	151
	9		76	83	358
	10		67	70	12
	11		49	55	330
	12		42	52	229
0	0	3	108	112	0
	1		121	120	199
	2		146	121	204
	3		86	81	36
	4		51	56	271
	5		236	216	196
	6		122	125	69
	7		57	68	24
	8		82	89	36
	9		160	155	182
	10		64	83	10
	11		60	73	10
	12		27	34	238
	13		43	42	162
0	0	4	143	123	0
	1		130	107	322
	2		241	198	179
	3		116	131	267
	4		43	66	31
	5		51	43	193
	6		93	105	185
	7		59	71	178
	8		87	94	0
	9		62	81	133
	10		53	57	264
	11		36	38	63
0	0	5	36	24	0
	1		80	89	325
	2		94	93	44
	3		241	204	217
	4		39	37	74
	5		63	72	26
	6		43	32	112
	7		71	90	229
	9		36	66	20
	10		60	28	195
0	0	6	22	14	180
	1		58	63	81
	2		135	156	340
	3		36	50	146
	4		102	129	211
	5		32	58	254
	6		56	54	82
	10		30	20	22
0	0	7	55	58	180
	1		54	62	140
	2		28	19	222
	5		59	80	229
0	0	8	35	53	0
0	0	9	32	20	180
0	3	7	58	75	6
1	0	0	168	199	0
	1		346	575	265
	2		311	417	226
	3		189	188	160
	4		79	82	249
	5		179	168	31
	6		168	174	191
	7		198	179	110
	8		118	116	62
	9		146	123	356
	10		23	27	148
	11		115	115	190
	12		31	37	261
	13		68	85	13
	14		29	22	180
	15		34	35	195
	17		38	39	11
1	0	1	178	176	0
	1		297	307	356
	2		104	126	87
	3		183	168	124
	4		170	188	141
	5		189	165	350
	6		155	156	52
	7		87	88	140
	8		189	182	154
	9		56	36	313
	10		130	137	2
	11		27	20	266
	12		105	105	170
	13		40	34	335
	14		44	42	12
	15		34	12	273
	16		29	31	163
1	0	-1	381	431	180
	1		374	408	205
	2		223	189	204
	3		94	90	26
	4		183	186	6
	5		236	208	190
	6		54	52	237
	7		113	106	102
	8		183	182	9
	9		56	48	175
	10		167	179	160
	11		68	66	132
	12		61	58	31
	13		42	42	125
	14		67	61	159
	16		48	47	349
1	0	2	452	374	180
	1		403	399	134
	2		63	57	157
	3		247	219	24
	4		215	236	153
1	5	2	135	159	131
	6		86	61	5
	7		139	152	326
	8		35	43	132
	9		63	65	218
	10		85	79	358
	11		45	48	311
	13		48	52	183
	14		23	15	3
1	0	-2	16	6	0
	1		293	342	199
	2		151	161	291
	3		178	172	304
	4		363	380	238
	5		216	219	200
	6		172	162	78
	7		103	109	331
	8		62	63	111
	9		59	52	122
	10		143	143	47
	11		98	96	32
	12		50	49	92
	13		48	46	145
	14		23	20	297
1	0	3	52	44	180
	1		264	244	194
	2		339	342	186
	3		96	95	12
	4		106	83	39
	5		79	65	117
	6		88	89	148
	7		164	162	359
	8		169	173	16
	9		66	64	321
	12		51	46	325
	14		46	34	155
1	0	-3	302	325	180
	1		20	31	111
	2		25	19	97
	3		265	244	170
	4		233	259	182
	5		29	9	26
	6		78	101	11
	7		57	68	90
	8		32	40	200
	9		100	96	315
	10		109	119	341
	11		46	50	219
	12		31	35	148
	14		49	24	22
	15		38	40	128
1	0	4	167	192	0
	1		229	225	3
	2		185	212	232
	3		63	78	116
	4		42	50	312
	5		150	143	348
	6		60	71	177
	7		71	76	133
	8		148	150	68
	10		62	60	50
	11		46	39	166
	12		23	16	39
1	0	-4	94	83	0
	1		155	160	33
	2		39	27	355
	3		140	165	130
	4		63	49	21
	5		145	122	330
	6		92	115	174
	7		85	80	208
	8		30	41	35
	9		107	111	280
	10		63	70	138
	11		81	76	204
	12		51	51	10
	13		22	24	42
	16		27	10	293
1	0	5	122	106	180
	1		117	116	343
	2		33	31	29
	3		40	33	270
	4		74	67	151
	5		148	145	49
	6		116	96	326
	8		81	75	199
	9		56	54	111
	10		60	42	299
	11		32	27	212
	12		53	47	160
	14		23	30	23
1	0	-5	81	82	0
	1		71	80	199
	2		49	41	155
	3		93	73	322
	4		97	78	328
	5		55	41	165
	6		106	95	138
	7		69	63	315
	8		59	62	337
	9		27	17	290
	10		74	77	174
	11		44	37	313
1	0	6	122	125	0
	1		77	73	217
	2		108	127	350
	5		115	96	92
	6		69	59	147
	7		97	87	2
	8		66	63	194
	10		31	19	48
	11		31	31	329
1	0	-6	46	39	180
	1		67	65	177
	2		63	60	342
	3		35	31	269
	4		77	69	200
	5		68	62	140
	6		27	30	47
	7		75	69	313
	9		53	51	176
	10		36	38	291
1	0	7	70	76	0
	1		49	61	94
	5		30	23	232
	7		36	17	4
1	0	-7	23	15	180
	1		60	62	301
	2		49	40	35
	3		102	107	189
	4		30	32	110
	6		38	39	5
	9		21	6	155
	13		21	20	142
1	0	-8	12	17	0
	1		42	38	292
	2		42	36	173
	3		46	46	216
2	0	0	14	38	180
2	1	0	46	75	148
	2		320	414	351
	3		110	146	145
	4		115	97	176
	5		152	156	11
	6		52	54	329
	7		119	108	170
	8		42	38	184
	9		101	118	338
	10		24	28	253
	11		151	180	167
	12		31	33	259
	13		29	33	36
	15		38	40	172
	16		11	19	174
	17		21	37	333
2	0	1	109	83	180
	1		257	170	204
	2		224	229	3
	3		124	134	25
	4		139	78	160
	5		51	47	183
	6		103	133	336
	7		136	116	7
	8		115	125	206
	9		51	38	256
	10		94	94	289
	11		42	37	287
	12		116	118	210
	13		27	29	166
	14		47	43	337
2	0	-1	179	192	0
	1		300	285	281
	2		300	274	106
	3		223	222	193
	4		205	218	339
	5		87	73	20
	6		139	140	133
	7		74	71	106
	8		159	182	305
	9		94	90	15
	10		74	81	173
	11		28	34	120
	12		91	91	7
	13		23	22	49
	14		40	45	185
2	0	2	381	368	0
	1		88	59	131
	2		134	143	358
	3		195	174	33
	4		100	114	340
	5		90	108	226
	6		122	103	172
	7		130	116	340
	8		28	33	172
	9		122	122	220
	10		48	52	175
	11		92	90	321
	12		46	42	7
	13		47	52	162
	14		21	21	241
	15		29	29	5
2	0	-2	72	58	0
	1		143	149	216
	2		132	155	153
	3		92	90	342
	4		50	51	249
	5		217	219	137
	6		74	107	148
	7		89	97	351
	8		116	116	330
	9		128	143	153
	10		65	64	297
	11		86	92	335
	12		73	74	341
	14		35	38	229
	15		43	46	354
2	0	3	119	87	0
	1		143	114	159
	2		146	147	137
	3		171	138	151
	4		108	105	18
	5		48	28	95
	6		73	59	189
	7		76	84	218
	8		112	116	310
	9		102	101	309
	10		62	61	216
	11		93	85	194
	12		50	53	355
	13		30	32	37
	14		36	35	155
	15		29	28	138
2	0	-3	167	153	180
	1		67	61	219
	2		186	224	349
	3		73	91	103
	4		149	132	122
	5		129	143	102
	6		96	110	340
	7		95	86	25
	8		66	72	124
	9		77	91	224
	10		34	34	325
	11		45	44	297
	12		31	28	172
	13		31	28	219
	14		25	31	348
	15		51	49	25
2	0	4	94	68	0
	1		80	84	250
	2		111	104	286
	3		163	163	192
	4		117	93	215
	5		62	62	271
	6		43	40	252
	7		69	69	170
	8		36	38	140
	9		119	117	352
	10		81	77	7
	11		56	51	137
	12		17	21	202
	13		40	47	22
	15		29	29	167
2	1	-4	74	89	346
	2		97	97	350
	3		62	75	168
	4		69	69	270
	5		137	138	20
	6		29	23	11
	7		32	28	109
	8		89	87	192
	9		87	105	344
	10		39	37	124
	12		31	33	207
	13		45	49	321
	14		40	35	332
	16		14	16	271
2	0	5	62	72	180
	3		51	43	208
	4		46	42	93
	5		93	89	210
	6		128	122	323
	8		67	61	138
	9		61	60	154
	10		26	22	12
	11		29	32	52
	12		57	56	156
	13		39	36	128
	14		26	25	43
2	0	-5	102	108	0
	1		62	65	50
	2		52	45	155
	3		49	39	172
	4		88	93	324
	5		104	107	52
	6		44	48	179
	8		61	59	278
	9		50	43	48
	10		67	79	195
	11		37	32	201
	14		21	32	171
2	0	6	28	27	0
	7		41	34	349
	9		57	52	160
	10		20	15	179
2	0	-6	117	124	0
	2		51	49	304
	3		42	40	4
	4		49	43	348
	5		45	44	79
	6		68	71	218
	7		88	89	359
	8		23	27	108
	9		56	60	166
2	0	7	45	56	0
2	0	-7	82	78	180
	1		61	61	243
	4		30	27	111
	5		36	38	94
	6		66	70	315
	7		59	57	38
2	0	-8	72	59	180
	1		37	39	66
	2		22	21	202
	3		25	25	64
	5		24	30	5
	6		35	37	11
	7		21	31	184
	10		18	23	26
2	2	-9	20	29	176
	3		22	33	151
	5		15	20	28
	8		8	23	44
3	0	0	129	173	180
	1		116	160	271
	2		250	242	355
	3		44	25	190
	4		211	225	218
	5		69	60	350
	6		82	72	343
	7		102	91	40
	8		141	135	180
	9		36	33	287
	10		72	76	56
	11		23	30	112
	12		64	75	99
	14		16	28	1
3	0	1	21	5	180
	1		338	259	168
	2		48	29	336
	3		221	196	356
	4		20	26	341
	5		126	116	142
	6		24	24	266
	7		119	118	345
	9		65	61	165
	10		43	47	177
	12		50	47	126
	13		54	55	180
3	0	-1	260	339	180
	1		288	280	351
	2		278	228	144
	3		157	188	187
	4		69	78	299
	5		133	122	32
	6		85	85	6
	7		122	103	173
	8		116	116	292
	9		43	34	49
	10		58	47	109
	11		43	39	214
	13		27	22	343
	14		24	24	85
3	0	2	74	43	0
	1		48	34	347
	2		162	170	191
	3		27	11	189
	4		152	118	334
	5		51	39	71
	6		113	125	173
	7		24	37	23
	8		65	60	357
	9		37	34	54
	10		109	111	164
	11		27	42	330
	12		35	35	348
3	1	-2	141	147	71
	2		207	224	209
	3		45	40	235
	4		113	121	22
	5		51	44	76
	6		77	69	170
	7		176	145	263
	8		187	207	42
	9		56	53	187
	10		68	68	123
	11		27	21	275
	12		58	60	6
	14		47	37	182
3	0	3	166	168	180
	1		175	145	328
	2		74	72	66
	3		160	148	152
	4		24	20	52
	5		129	119	18
	6		58	58	48
	7		59	63	121
	8		24	18	45
	9		68	75	331
	10		66	64	137
	11		52	45	232
	12		29	37	36
	13		33	42	356
3	0	-3	132	135	180
	1		180	216	188
	2		127	134	163
3	4	-3	97	109	63
	5		113	121	176
	6		51	52	336
	7		174	170	345
	8		43	49	105
	9		68	67	276
	11		64	71	347
	13		67	60	176
	14		40	34	319
3	0	4	138	123	180
	1		76	68	189
	2		47	42	41
	3		77	64	56
	4		127	125	183
	5		32	41	109
	6		82	85	43
	7		58	59	125
	8		37	24	159
	9		31	17	314
	10		89	90	2
	11		29	36	183
	13		31	38	354
3	0	-4	112	130	180
	1		74	92	108
	2		121	119	68
	3		18	19	124
	4		81	86	146
	5		67	69	237
	6		110	100	7
	7		32	36	264
	8		80	108	204
	10		56	56	342
	11		22	19	116
	12		43	41	169
	14		41	40	356
3	0	5	29	47	180
	3		74	76	358
	4		45	48	81
	5		45	47	183
	6		27	27	309
	7		68	70	47
3	0	-5	62	68	0
	1		109	111	329
	2		58	65	9
	3		101	115	195
	4		25	33	96
	5		52	58	357
	7		51	52	176
3	0	6	44	60	0
3	0	-6	58	46	0
	1		104	99	259
	2		78	91	192
	3		58	57	220
	4		64	68	1
	5		32	42	125
	6		71	76	230
3	0	7	21	2	0
3	0	-7	44	38	180
	1		71	80	181
	2		27	38	153
	4		36	42	22
	5		70	80	188
	6		45	55	323
3	0	8	17	24	0
3	2	-8	36	43	354
3	2	-9	33	30	88
	3		36	41	158
	4		13	14	73
4	0	0	104	140	180
	1		85	104	239
	2		66	73	21
	3		39	39	343
	4		41	33	239
	5		131	130	171
	6		114	105	318
	7		91	81	58
	8		84	92	232
	9		107	108	164
	11		61	60	8
	12		17	23	145
	13		30	35	137
	14		20	26	353
4	0	1	203	193	0
	1		133	145	240
	2		38	21	135
	3		56	48	298
	4		122	102	5
	5		75	60	149
	6		110	107	207
	7		85	79	21
	8		36	28	308
	9		36	33	163
	10		68	68	213
	11		28	28	345
	12		38	38	9
	13		23	24	151
	14		31	29	177
	15		34	13	17
4	0	-1	80	102	180
	1		91	114	36
	2		124	134	353
	3		115	100	222
	4		38	35	153
	5		25	17	53
	6		140	133	343
	7		112	110	205
	8		57	55	131
	9		40	39	72
	10		39	32	21
	11		28	25	92
	12		54	53	173
	13		29	37	335
	14		35	33	32
	15		23	18	79
4	0	2	199	174	0
	1		159	148	43
	2		83	64	107
	3		87	70	220
	4		82	89	25
	5		82	70	357
	6		49	62	150
	7		111	125	219
	8		44	35	296
	9		38	35	48
	10		27	31	241
	11		41	48	157
	13		21	18	336
4	0	-2	165	171	0
	1		75	80	18
	2		75	61	214
	3		137	137	182
	4		69	55	23
	5		102	87	5
	6		78	96	204
	7		48	42	221
	8		26	27	121
	9		91	90	350
	11		20	14	172
	12		36	40	313
4	13	-2	27	31	8
4	0	3	152	203	180
	1		50	56	21
	2		119	118	55
	3		143	139	182
	4		58	60	121
	5		26	11	352
	6		103	95	1
	7		33	28	255
	8		76	75	245
	9		57	50	335
	10		26	25	256
	11		21	16	331
	12		20	22	226
	14		32	28	331
4	0	-3	100	116	0
	1		75	85	135
	2		180	171	131
	3		110	118	44
	4		161	175	31
	5		35	35	145
	6		50	47	222
	7		61	71	18
	8		77	78	265
	9		26	28	238
	10		61	68	221
	11		45	49	273
	13		25	27	187
4	0	4	33	74	130
	1		85	77	173
	2		94	94	348
	3		53	39	12
	4		55	63	207
	5		63	75	209
	6		35	35	50
	7		66	63	324
	8		49	35	90
	9		48	47	228
	10		26	26	67
	11		19	21	338
	12		18	22	356
4	0	-4	30	21	0
	1		38	39	96
	2		128	135	3
	3		224	217	4
	4		38	39	125
	5		38	38	307
	6		31	31	303
	7		36	42	18
	8		52	59	173
	9		60	58	175
	11		24	17	342
	13		22	21	257
4	4	5	64	63	347
	5		56	60	210
	7		19	22	195
	8		66	53	328
	9		40	41	194
	10		18	18	190
	11		14	7	126
4	0	-5	88	92	180
	1		84	80	301
	2		48	48	322
	3		23	18	163
	4		41	40	211
	5		78	85	34
	6		63	66	329
	7		60	62	89
	8		26	22	205
	9		25	31	357
	10		21	12	35
	12		40	39	207
4	0	-6	29	23	180
	1		27	30	35
	2		77	75	161
	3		17	23	159
	4		71	76	353
	5		29	24	1
	6		41	38	338
	7		51	45	167
	8		24	24	310
	10		20	26	131
4	0	7	18	22	180
4	1	-7	30	28	174
	2		31	37	160
	3		25	22	339
	5		54	47	225
	8		31	28	15
4	0	-8	56	46	180
	4		37	32	159
	6		17	11	89
5	0	0	14	36	180
	1		56	93	140
	2		72	94	184
	3		81	113	335
	4		53	63	356
	5		77	89	205
	6		19	18	334
	7		54	49	284
	8		50	53	32
	9		60	68	139
	10		50	49	110
	11		47	45	32
	12		47	37	81
	13		18	15	138
	14		8	11	147
5	0	1	68	54	180
	1		79	65	310
	2		134	139	183
	3		57	56	152
	4		89	94	3
	5		47	55	34
	6		32	34	267
	7		58	56	89
	8		60	68	352
	9		18	18	356
	10		51	53	163
	11		23	36	205
5	0	-1	86	115	180
	1		54	71	192
	2		17	14	267
	3		41	46	237
	4		104	118	160
	5		88	87	195
	6		60	58	346
	8		60	61	130
	9		18	19	53
	10		75	80	352
	11		61	60	23
	12		23	27	149
5	0	2	40	30	0
	1		154	124	1
	2		82	65	296
	3		45	34	341
	4		49	49	196
	5		96	122	359
	7		30	28	168
	8		58	67	133
	9		18	22	20
5	10	2	42	34	17
	11		30	30	184
	12		27	22	190
	13		35	28	321
5	0	-2	52	53	180
	1		75	66	8
	2		43	57	126
	3		80	85	149
	4		63	63	117
	5		45	52	304
	6		38	37	34
	7		73	81	155
	8		23	28	178
	9		39	48	312
	13		30	28	2
	12		19	14	207
	11		28	37	178
	10		57	49	10
5	0	3	31	56	180
	1		95	68	65
	2		99	76	67
	3		80	70	0
	4		56	53	104
	5		39	37	58
	6		57	67	11
	7		32	30	320
	8		64	59	201
	10		20	22	335
	9		55	34	168
	11		31	24	331
	12		38	40	156
5	0	-3	49	44	0
	1		28	27	8
	2		64	53	145
	3		42	40	345
	4		94	85	11
	5		41	48	308
	6		43	40	162
	7		47	45	181
	9		21	34	14
	10		67	77	153
	11		25	27	214
	13		29	30	2
5	3	4	30	18	22
	5		44	46	132
	6		44	39	180
	7		64	55	1
	8		16	9	351
	10		23	23	167
	11		32	38	344
5	0	-4	16	0	0
	1		46	47	167
	2		73	51	259
	3		92	84	339
	4		60	49	272
	6		40	38	214
	5		47	46	214
	9		26	36	131
	10		44	36	182
	11		26	27	20
5	0	-5	46	45	180
	1		39	35	155
	2		28	34	14
	3		23	11	158
	4		36	23	238
	5		52	44	215
	6		58	59	341
	7		28	16	325
	10		13	16	4
5	1	-6	59	36	17
	2		28	28	36
	3		22	29	224
	4		19	16	198
	5		62	60	272
	6		51	13	302
5	0	-7	20	9	180
	2		40	34	142
	3		11	18	188
	5		25	15	209
	6		20	30	138
6	0	0	94	115	0
	1		19	24	95
	2		36	41	189
	3		31	35	304
	4		31	40	324
	5		27	36	225
	6		52	79	188
	7		31	42	220
	8		25	35	318
	9		22	29	86
	10		12	25	212
	12		7	38	356
6	0	1	30	33	0
6	0	-1	32	7	0
	1		59	54	170
	2		32	23	86
	3		71	63	38
	4		74	65	95
	5		74	65	180
	6		30	8	43
	7		30	32	35
	8		28	16	22
	9		69	55	198
	10		70	51	287
	11		48	35	329
6	0	2	22	17	180
6	0	-2	38	37	180
	1		44	17	62
	2		84	85	12
	3		45	72	18
	4		42	44	152
	5		41	46	312
	6		40	48	35
	7		39	13	120
6	0	3	35	42	0
6	0	-3	29	27	180
	9		70	61	20
6	0	4	41	55	0
6	0	-4	84	78	0
6	0	5	18	25	180
6	0	-5	28	25	0
6	0	-6	72	50	180
7	0	0	9	6	180
	1		40	52	15
	2		61	31	200
	3		70	56	182
	4		7	9	319
	5		16	16	333
	6		26	23	120
	7		49	57	182
7	0	-1	31	36	0
	1		24	29	176
	2		46	44	204
	3		23	23	348
	6		49	44	150
7	0	-2	45	46	180
7	0	4	21	24	0
8	0	0	10	17	0
	1		25	39	23
	2		16	26	343

The standard deviations in the positional co-ordinates given in Table 5 were calculated directly from the least-squares output by standard procedures. The molecular dimensions, also with their appropriate standard deviations, are given in Tables 6 and 7, with some non-bonded intramolecular distances in Table 8.

The packing of the molecules in the crystal can only be fully studied by means of models, but the projections of the structure shown in Figs. 5 and 6 indicate the general arrangement. No unusual approach distances occur, the shortest being 3·5 Å between carbon atoms of adjoining molecules. A sample of the intermolecular distances is given in Table 9.

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