Crystal and Molecular Structure of (1*S*,2*S*)-2-[(*S*)-Chlorofluoroacetoxy]-1-phenylcyclohexanol

By **M. Colapietro, R. Spagna, and L. Zambonelli,*** Laboratorio di Strutturistica Chimica, ' Giordano Giacomello,' C.N.R., Città Universitaria, 00100 Roma, Italy

The structure of the title compound has been determined by single-crystal *X*-ray analysis. Crystals are orthorhombic, $a = 5{\cdot}750$, $b = 14{\cdot}339$, $c = 17{\cdot}014$ Å, space group $P2_12_12_1$, $Z = 4$. The structure was solved by a combination of direct and Fourier methods, and refined by least-squares methods to *R* 0·054, by use of 1657 independent reflections collected by counter methods.

The structure contains discrete molecules linked into infinite chains by O–H · · · O hydrogen bonds. The absolute configuration of the molecule, established with >97·5% probability, corroborates the known chirality of the parent compound (−)-(1*S*,2*S*)-1,2-epoxy-1-phenylcyclohexane, and assigns the (*S*)-configuration to the asymmetric carbon atom of the (+)-chlorofluoroacetic acid.

CHLOROFLUOROACETIC acid is one of the simplest chiral molecules. It appeared interesting to determine its configuration by *X*-ray analysis of the crystalline product (1*S*,2*S*)-[(*S*)-chlorofluoroacetoxy]-1-phenylcyclohexanol, of the reaction of (+)-chlorofluoroacetic acid with (−)-(1*S*,2*S*)-1,2-epoxy-1-phenylcyclohexane.[1-3]

EXPERIMENTAL

Crystal Data.—$C_{14}H_{16}ClFO_3$, $M = 286{\cdot}73$. Orthorhombic, $a = 5{\cdot}750(3)$, $b = 14{\cdot}339(6)$, $c = 17{\cdot}014(4)$ Å, $U = 1402{\cdot}8$ Å^3, $D_m = 1{\cdot}36 \pm 0{\cdot}01$ (by flotation in $CaBr_2$ solution), $Z = 4$, $D_c = 1{\cdot}356$, $F(000) = 600{\cdot}60$. Space group $P2_12_12_1$, from systematic absences: $h00$ for h odd, $0k0$ for k odd, and $00l$ for l odd. Mo-K_α radiation, $\lambda = 0{\cdot}7107$ Å; μ(Mo-K_α) = 2·9 cm^{-1}.

Unit-cell parameters and their estimated standard deviations were determined from a least-squares refinement of the setting angles of 13 *X*-ray reflections which had been centred on a Siemens AED automatic diffractometer by use of Mo-K_α radiation.

Data Collection.—A crystal of dimensions 0·39 × 0·48 × 0·51 mm was mounted on a Siemens AED automatic single-crystal diffractometer, equipped with a scintillation counter and pulse-height analyser. The *a* axis (along the 0·51 mm dimension of the crystal) was coincident with the polar ϕ axis of the goniostat. Intensities were recorded by use of zirconium-filtered Mo-K_α radiation, and the θ—2θ scan technique, each reflection being scanned twice at a scan rate automatically chosen according to the peak intensity. After several test scans a symmetrical scan range of 0·80° from the computed Mo-K_α peak was used for reflections up to θ 26·0°, and of 0·70° for reflections from θ 26·0—32·0°. At each end of the scan, stationary-crystal–stationary-counter background counts were taken for a time equal to the scan time.

Since there were few reflections of significant intensity for values of $\theta > 32°$, only the 2764 independent reflections within $\theta < 32°$ were gathered. The intensities of three standard reflections, chosen in different regions of the reciprocal space and measured every 80 reflections, remained essentially constant throughout.

From the collected data the values of F_o^2 and $\sigma(F_o^2)$

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[1] G. Bellucci, G. Berti, A. Borraccini, and F. Macchia, *Tetrahedron*, 1969, **25**, 2979.

[2] G. Berti, B. Macchia, F. Macchia, and L. Monti, *J. Org. Chem.*, 1968, **33**, 4045.

[3] G. Bellucci, G. Berti, C. Bettoni, and F. Macchia, preceding paper.

were obtained with the procedure previously described.[4] A value of 0·05 was chosen for the constant p, which is used in the formula for calculating $\sigma(F_o^2)$, according to the variance of the standard reflections.

Of the 2764 observations, 1107 had $F_o^2 < 3\sigma(F_o^2)$ and were discarded.

Neither absorption nor extinction corrections were applied.

Solution and Refinement of the Structure.—Since a three-dimensional Patterson function proved difficult to interpret, direct methods were employed to solve the structure. While the three-dimensional structure ($P2_12_12_1$) is non-centrosymmetric, the projections along the crystallographic axes (pgg) are centrosymmetric: therefore we decided to solve the structure in projection along the a axis, which is the shortest in the cell. Normalized structure factors E were calculated from the observed values of 263 structure amplitudes of the kind $F(0kl)$. A total of 104 reflections having $E > 1{\cdot}0$ could be phased by the iterative application of Sayre's equation,[5] and an E map was obtained which showed a projection of the structure almost completely resolved.

Approximate values for the x co-ordinates of some atoms were obtained by a computer-programmed systematic trial. The structure was then completely solved by means of subsequent three-dimensional Fourier syntheses.

Least-squares isotropic refinement lowered R from 0·388 to 0·160. At this point the atoms were allowed to vibrate anisotropically and convergence was reached at R 0·084. A difference-Fourier synthesis computed at this stage showed some regions of the electron-density map with values $\geqslant 3\sigma(\rho)$ where $\sigma(\rho) = 0{\cdot}13$ eÅ^{-3}.

All the hydrogen atoms were located and their contribution introduced in a structure-factor calculation reduced R to 0·066.

Refinement was completed anisotropically for the non-hydrogen atoms and isotropically for the hydrogen atoms. The final R was 0·054 (R' 0·074) and $\sigma(\rho)$ on the final difference-Fourier synthesis was 0·07 eÅ^{-3}. The refinement was carried out with a two-block approximation of the normal-equations matrix, using one block for the positional co-ordinates and one block for the temperature factors and the scale factor. The function minimized during the refinement was $\Sigma w(|F_o| - |F_c|)^2$ with weights w equal to $4F_o^2/\sigma^2(F_o^2)$.

The molecular configuration assumed during the refinement was derived to conform with the absolute configuration of the asymmetric carbon atoms C(1) and C(2) of the cyclohexane part of the molecule known from other methods.[2] Independent evidence for the chirality was obtained by changing the value of the imaginary part, $\Delta f_{\text{Cl}}''$, of the chlorine anomalous scattering factor in the last three cycles of the refinement. The experiment gave the following R' values, which are reported with several decimal digits for better comparison among the models used:

$\Delta f_{\text{Cl}}''$	R'
0·19	0·0744
0	0·0749
−0·19	0·0758

Hamilton's ratio test [6] applied to these results shows that the absolute configuration depicted in Figure 1 is correct with a probability >97·5%.

Table 1 gives the final positional and anisotropic thermal parameters for the non-hydrogen atoms. Co-ordinates and isotropic temperature factor for the hydrogen atoms are given in Table 2. Observed and calculated structure factors are listed in Supplementary Publication No. SUP 20568 (2 pp., 1 microfiche).*

Atomic scattering factors were taken from ref. 7 for chlorine, fluorine, oxygen, and carbon, and from ref. 8 for hydrogen. The effects of anomalous dispersion were included in the structure factors, the values $\Delta f'$ and $\Delta f''$ for chlorine being taken from ref. 9.

Calculations were carried out on the UNIVAC 1108 computer of Rome University and were performed with the system of programmes developed in this Laboratory,[10] with extensive contributions from Dr. J. R. Carruthers. Figure 1 was produced by Johnson's ORTEP programme.[11]

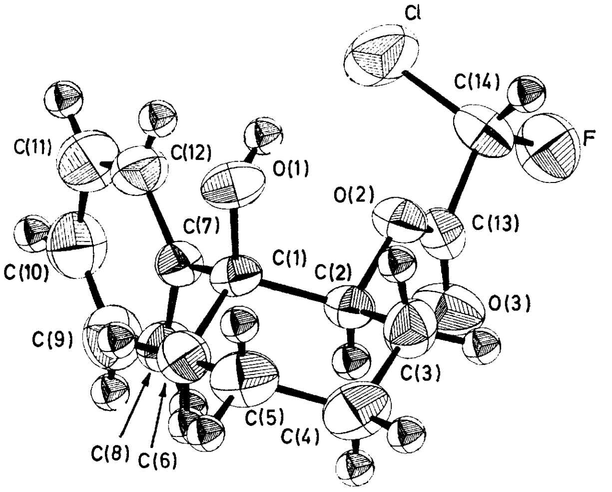

FIGURE 1 A perspective view of the molecule showing the 50% probability thermal vibration ellipsoids

RESULTS AND DISCUSSION

The X-ray analysis of the ester corroborates the chirality of the epoxycyclohexane previously determined by different methods,[2] and shows that the chiral carbon atom of the (+)-chlorofluoroacetic acid has the (S)-configuration. A perspective view of the molecule of (1S,2S)-2-[(S)-chlorofluoroacetoxy]-1-phenylcyclohexanol is shown in Figure 1. Intramolecular bond lengths and angles are given in Table 3 for the non-hydrogen atoms and in Table 4 for the hydrogen atoms. Relevant torsion angles are reported in Table 5.

The cyclohexane ring of the molecule is in the chair

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* For details see Notice to Authors No. 7 in *J. Chem. Soc.* (*A*), 1970, Issue No. 20 (items less than 10 pp. are sent as full size copies).

[4] S. Cerrini, M. Colapietro, R. Spagna, and L. Zambonelli, *J. Chem. Soc.* (*A*), 1971, 1375.

[5] D. Sayre, *Acta Cryst.*, 1952, **5**, 60.

[6] W. C. Hamilton, *Acta Cryst.*, 1965, **18**, 502.

[7] D. T. Cromer and J. B. Mann, *Acta Cryst.*, 1968, *A*, **24**, 321.

[8] H. P. Hanson, F. Herman, J. Lea, and S. Skillman, *Acta Cryst.*, 1964, **17**, 1040.

[9] D. T. Cromer, *Acta Cryst.*, 1965, **18**, 17.

[10] A. Domenicano, R. Spagna, and A. Vaciago, *Atti. Accad. naz. Lincei, Rend. Classe Sci. fis. mat. nat.*, 1969, **47**, 331.

[11] C. K. Johnson, 'ORTEP: A Fortran Thermal Ellipsoids Plot Program for Crystal Structure Illustrations,' Oak Ridge National Laboratory Report ORNL 3794, 1965.

TABLE 1

Final positional and anisotropic * thermal parameters (all $\times 10^4$) for the non-hydrogen atoms, with estimated standard deviations in parentheses

	x	y	z	b_{11}	b_{12}	b_{13}	b_{22}	b_{23}	b_{33}
Cl	−273(3)	5806(1)	2745(1)	557(6)	−195(5)	−100(3)	134(1)	30(1)	42(0)
F	3362(6)	6788(2)	2642(1)	547(12)	−83(8)	36(6)	94(2)	37(2)	39(1)
O(1)	−2536(4)	5502(2)	5360(2)	193(6)	40(6)	−2(4)	66(1)	−10(2)	40(1)
O(2)	1018(5)	6267(2)	4470(1)	255(7)	41(5)	−8(4)	48(1)	9(2)	30(1)
O(3)	4557(5)	5955(2)	3976(2)	295(9)	100(8)	33(5)	95(2)	26(2)	39(1)
C(1)	−241(5)	5122(2)	5415(2)	190(9)	7(7)	−8(5)	48(2)	0(2)	25(1)
C(2)	1559(6)	5895(2)	5259(2)	220(9)	20(7)	−1(5)	43(1)	5(2)	26(1)
C(3)	1451(9)	6697(3)	5835(2)	430(17)	−47(10)	−14(8)	49(2)	−11(3)	37(1)
C(4)	1764(10)	6343(4)	6671(2)	438(18)	−22(13)	−30(8)	76(3)	−25(3)	34(1)
C(5)	−92(9)	5607(3)	6861(2)	376(15)	19(12)	12(8)	80(3)	−8(3)	29(1)
C(6)	29(8)	4800(3)	6279(2)	321(13)	−37(10)	11(7)	59(2)	12(2)	28(1)
C(7)	142(6)	4312(2)	4849(2)	232(9)	−5(7)	−4(6)	41(2)	4(2)	28(1)
C(8)	2199(7)	3796(3)	4871(2)	287(12)	15(9)	−18(7)	51(2)	−6(3)	42(1)
C(9)	2565(10)	3054(3)	4368(3)	419(17)	28(11)	30(11)	51(2)	−12(3)	53(2)
C(10)	909(10)	2827(3)	3809(3)	483(18)	5(12)	20(10)	51(2)	−19(3)	47(2)
C(11)	−1153(11)	3327(3)	3778(3)	561(22)	−47(14)	−73(10)	63(2)	−17(3)	41(2)
C(12)	−1554(8)	4062(3)	4301(2)	330(13)	1(9)	−55(7)	51(2)	−8(3)	40(1)
C(13)	2633(7)	6228(2)	3920(2)	344(12)	−4(8)	0(7)	35(1)	4(2)	27(1)
C(14)	1594(8)	6618(3)	3159(2)	411(15)	57(10)	−6(8)	56(2)	23(3)	31(1)

* Coefficients b_{ij} are defined by the expression: $T = \exp(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{13}hl + b_{23}kl)$.

TABLE 2

Final positional ($\times 10^3$) and isotropic thermal parameters for the hydrogen atoms with estimated standard deviations in parentheses

	x	y	z	B/Å^2
H(1)[C(2)]	298(6)	565(2)	527(2)	0·2(6)
H(2)[C(3)]	254(11)	718(4)	573(3)	3·6(11)
H(3)[C(3)]	−32(10)	688(3)	583(3)	3·4(10)
H(4)[C(4)]	198(9)	692(3)	707(3)	3·0(10)
H(5)[C(4)]	317(9)	608(3)	673(3)	2·9(10)
H(6)[C(5)]	13(9)	533(3)	740(2)	2·6(8)
H(7)[C(5)]	−152(11)	582(4)	684(3)	4·3(12)
H(8)[C(6)]	−95(7)	432(3)	634(2)	1·2(7)
H(9)[C(6)]	174(8)	454(3)	634(2)	2·2(8)
H(10)[C(8)]	331(8)	393(3)	522(2)	2·4(9)
H(11)[C(9)]	407(9)	273(3)	431(2)	2·5(9)
H(12)[C(10)]	104(7)	226(3)	343(2)	1·7(7)
H(13)[C(11)]	−225(9)	317(3)	341(3)	2·7(9)
H(14)[C(12)]	−255(8)	439(2)	417(2)	1·1(7)
H(15)[O(1)]	−275(10)	570(3)	488(3)	4·3(13)
H(16)[C(14)]	77(8)	722(3)	322(2)	2·2(8)

TABLE 3

Intramolecular bond lengths (Å) and angles (°) for the non-hydrogen atoms

(*a*) Distances

C(1)–C(2)	1·539(5)	C(9)–C(10)	1·385(7)
C(2)–C(3)	1·512(5)	C(10)–C(11)	1·387(8)
C(3)–C(4)	1·522(6)	C(11)–C(12)	1·399(7)
C(4)–C(5)	1·535(7)	C(12)–C(7)	1·396(5)
C(5)–C(6)	1·526(6)	C(2)–O(2)	1·478(4)
C(6)–C(1)	1·548(4)	O(2)–C(13)	1·319(5)
C(1)–O(1)	1·431(4)	C(13)–O(3)	1·177(5)
C(1)–C(7)	1·526(4)	C(13)–C(14)	1·533(5)
C(7)–C(8)	1·395(5)	C(14)–Cl	1·733(5)
C(8)–C(9)	1·382(6)	C(14)–F	1·366(5)

(*b*) Angles

C(1)–C(2)–C(3)	114·1(3)	C(8)–C(9)–C(10)	120·2(5)
C(2)–C(3)–C(4)	110·3(4)	C(9)–C(10)–C(11)	119·5(4)
C(3)–C(4)–C(5)	110·1(4)	C(10)–C(11)–C(12)	120·4(5)
C(4)–C(5)–C(6)	110·7(4)	C(11)–C(12)–C(7)	120·2(4)
C(5)–C(6)–C(1)	112·7(3)	C(12)–C(7)–C(8)	118·3(3)
C(6)–C(1)–C(2)	108·1(4)	C(1)–C(2)–O(2)	106·0(3)
C(2)–C(1)–O(1)	109·5(3)	C(3)–C(2)–O(2)	107·7(3)
C(2)–C(1)–C(7)	110·0(3)	C(2)–O(2)–C(13)	118·8(3)
C(6)–C(1)–O(1)	105·6(3)	O(2)–C(13)–O(3)	128·2(3)
C(6)–C(1)–C(7)	111·0(3)	O(2)–C(13)–C(14)	108·1(3)
O(1)–C(1)–C(7)	112·4(3)	O(3)–C(13)–C(14)	123·7(4)
C(1)–C(7)–C(8)	120·6(3)	C(13)–C(14)–Cl	109·9(3)
C(1)–C(7)–C(12)	121·2(3)	C(13)–C(14)–F	108·6(4)
C(7)–C(8)–C(9)	121·4(4)	Cl–C(14)–F	108·6(3)

TABLE 4

Bond lengths (Å) and angles (°) concerning the hydrogen atoms

(*a*) Cyclohexane ring (mean values)

C–H 0·95(3), C–C–H 109(1), H–C–H 107(3)

(*b*) Benzene ring (mean values)

C–H 0·92(5), C–C–H 119(1)

(*c*) Hydroxy-group

O(1)–H(15) 0·88(5), C(1)–O(1)–H(15) 108(4)

(*d*) Chlorofluoroacetoxy-group

C(14)–H(16) 0·99(4), C(13)–C(14)–H(16) 114(2), Cl–C(14)–H(16) 109(3), F–C(14)–H(16) 106(3)

TABLE 5

Internal rotation angles * with standard deviations in parentheses

C(1)–C(2)–C(3)–C(4)	57·2(5)
C(2)–C(3)–C(4)–C(5)	−57·1(5)
C(3)–C(4)–C(5)–C(6)	57·6(5)
C(4)–C(5)–C(6)–C(1)	−56·9(5)
C(5)–C(6)–C(1)–C(2)	53·3(4)
C(6)–C(1)–C(2)–C(3)	−54·0(4)
O(1)–C(1)–C(2)–C(3)	60·7(4)
O(1)–C(1)–C(2)–O(2)	−57·8(3)
C(7)–C(1)–C(2)–C(3)	−175·3(3)
C(7)–C(1)–C(2)–O(2)	66·3(3)
C(6)–C(1)–C(2)–O(2)	−172·3(3)
O(1)–C(1)–C(6)–C(5)	−63·8(4)
C(7)–C(1)–C(6)–C(5)	174·1(3)
O(2)–C(2)–C(3)–C(4)	174·6(4)
C(2)–O(2)–C(13)–C(14)	177·6(3)
C(2)–O(2)–C(13)–O(3)	−3·0(5)
O(2)–C(13)–C(14)–Cl	−75·5(3)
O(2)–C(13)–C(14)–F	165·8(3)
O(2)–C(13)–C(14)–H(16)	47·9(29)
O(3)–C(13)–C(14)–Cl	105·0(4)
O(3)–C(13)–C(14)–F	−13·6(5)
O(3)–C(13)–C(14)–H(16)	−131·6(29)

* The convention of Klyne and Prelog is adopted (W. Klyne and V. Prelog, *Experientia*, 1960, **16**, 521).

form. The endocyclic bond distances, angles and torsion angles have fairly regular values,[12,13] their weighted mean

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[12] M. Davis and O. Hassel, *Acta Chem. Scand.*, 1963, **17**, 1181.

[13] S. T. Rao and M. Sundaralingam, *Acta Cryst.*, 1969, *B*, **25**, 2509.

values being 1·532(6) Å, 111·2(10)°, and 55·7(8)° respectively; the maximum deviations relate to the carbon atoms involved in substituent groups. The phenyl and chlorofluoroacetoxy-groups are equatorially substituted. The plane of the benzene ring [plane (2)], and the plane of the chlorofluoroacetoxy-group [plane (3)] make with the plane of the cyclohexane ring [plane (1)],* dihedral angles of 86·7 and 84·7° respectively. The three planes are defined as the least-squares planes: (1) through the six carbon atoms of the cyclohexane ring, (2) through the six carbon atoms of the phenyl group, and (3) through the atoms C(13), C(14), O(2), and O(3) of the chlorofluoroacetoxy-group. Their equations, referred to the crystallographic axes, are:

$$(1)\ 0{\cdot}903x - 0{\cdot}423y + 0{\cdot}076z + 2{\cdot}313 = 0$$

$$(2)\ 0{\cdot}415x + 0{\cdot}630y - 0{\cdot}656z + 1{\cdot}492 = 0$$

$$(3)\ 0{\cdot}307x + 0{\cdot}918y + 0{\cdot}252z - 10{\cdot}346 = 0$$

The phenyl group is almost exactly planar and the endocyclic bond lengths and angles, with weighted mean values of 1·391(3) Å and 119·7(5)° respectively, are consistent with the data from the literature.[14] The bond C(1)–C(7) [1·526(4) Å] is significantly longer than a $C(sp^3)$–$C(sp^2)$ single bond (1·504 Å).[14]

The C(13), C(14), O(2), and O(3) atoms of the chlorofluoroacetoxy-group lie exactly in a plane (the maximum deviation from the least-squares plane through them is 0·003 Å). The C(2) atom of the cyclohexane ring almost lies on this plane, deviating only 0·06 Å from it. A projection of the C(2)·O(2)·C(13)[:O(3)]·C(14) part of the molecule, on the best plane through C(13), C(14), O(2), and O(3), is shown in Figure 2.

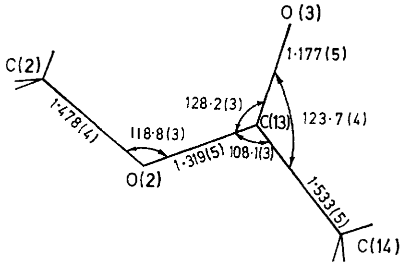
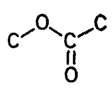
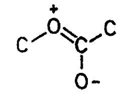


FIGURE 2 Schematic projection on the best plane through C(13), C(14), O(2), and O(3) of the C·O·C(:O)·C part of the molecule, showing relevant bond-lengths and angles

The geometry and dimensions of the group C·O·C(:O)·C compare well with the few data available in the literature.[15-17] The C(2)–O(2) bond [1·478(4) Å] is considerably longer than a paraffinic C–O bond, which is reported[18] to be 1·426 and 1·427 Å when calculated from the sum of the covalent radii.[14,19] On the other hand the O(2)–C(13) bond [1·319(5) Å] is much shorter even than the sum of the $C(sp^2)$ and oxygen single-bond radii, 1·397 Å.[14,19] Similar anomalous values of C–O bond lengths are generally observed in molecules containing the C·O·C(:O)·C group. A satisfactory explanation is given in ref. 17. The values of the C(2)–O(2)–C(13) angle [118·8(3)°] and of the O(2)–C(13) bond length [1·319(5) Å] suggest a significant contribution of the canonical form (B) to the ground state of the system. This contribution does not seem to affect the C(13)–O(3) double-bond whose length [1·177(5) Å] is in good agreement with the value for a pure C=O double bond; this contradiction is only apparent since it is known that the variation in bond length, due to variations in bond-order, is more marked for single than for double bonds.



(A)



(B)

Furthermore the contribution of form (B) lowers the amount of the ionic contribution, due to the electronegativity difference, in the C(2)–O(2) bond, which therefore becomes longer than the sum of the covalent radii corrected for the difference in electronegativity.[14,19]

Introduction into a molecule of an electronegative atom makes adjacent bonds shorter; the evidence for this has been summarized.[20] Accordingly, the expected value for the length of the C(13)–C(14) bond is less than the sum of the $C(sp^3)$ and $C(sp^2)$ single-bond radii (1·504 Å), since C(14) is substituted by the strongly electronegative fluorine and chlorine atoms. However the experimental value of this bond length [1·533(5) Å] is appreciably higher even than a pure $C(sp^3)$–$C(sp^2)$ single-bond. The reason for this can be inferred by noting the geometry around the C(13) atom. As a matter of fact the values of the angles around C(13) show that this sp^2-hybridized carbon atom does not have a perfectly symmetrical trigonal hybridization and that its orbital in the C(13)–O(3) double-bond direction has greater *s* character than the other two.[21] Therefore it can be expected that a greater *p* character in the orbital directed along C(13)–C(14) increases the actual radius of C(13) in this direction causing a lengthening of the C(13)–C(14) bond.

Both the C(14)–Cl [1·733(5) Å] and C(14)–F [1·366(5) Å] bonds are shorter than those observed in monohalogenoacetyl-derivatives (C–Cl 1·779 in monochloroacetylalanine,[22] C–F 1·406 Å in monofluoroacetamide[23]). Electronegative substituents affect the adjacent bond

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* Plane (1) defines the general orientation of the cyclohexane ring with respect to the rest of the molecule and it is used to help describe the geometry of the molecule.

[14] O. Bastiansen and P. N. Skancke, *Adv. Chem. Phys.*, 1961, **3**, 323.

[15] R. F. Curl, jun., *J. Chem. Phys.*, 1959, **30**, 1529.

[16] B. H. Bracher and R. W. H. Small, *Acta Cryst.*, 1967, **23**, 410.

[17] S. Merlino, *Acta Cryst.*, 1971, *B*, **27**, 2491.

[18] *Chem. Soc. Special Publ.*, No. 18, 1965.

[19] L. Pauling, 'The Nature of the Chemical Bond,' 3rd edn., Cornell University Press, Ithaca, New York, 1960, p. 228.

[20] H. A. Bent, *J. Chem. Phys.*, 1960, **33**, 1259, 1260.

[21] C. A. Coulson, A. Domenicano, and A. Vaciago, unpublished observations.

[22] F. E. Cole, *Acta Cryst.*, 1970, *B*, **26**, 622.

[23] D. O. Hughes and R. W. H. Small, *Acta Cryst.*, 1962, **15**, 933.

lengths.[20] Therefore it can be expected that in the system Cl–C–F of the present compound, the electronegative fluorine and chlorine atoms mutually shorten their bonds to the carbon atom.

The hydroxy-group is in an axial position: the C(1)–O(1) bond length [1·431(4) Å] agrees well with the sum of the covalent radii (1·427 Å)[14,19] and with the literature value of 1·426 Å.[18] The bond O(1)–H(15) has a length of 0·88(5) Å and the angle C(1)–O(1)–H(15) is 108(4)°. The hydroxy-group takes part in a hydrogen bond with the O(3) atom of the chlorofluoroacetoxy-group of the molecule at $x - 1, y, z$ [O(1) ··· O(3)′ 2·960, H(15) ··· O(3)′ 2·21 Å, and O(1)–H(15) ··· O(3)′ 143°]. Thus, the molecules in the crystal are stacked in infinite columns parallel to the *a* axis.

No intermolecular contacts other than the O–H ··· O hydrogen bonds are observed.

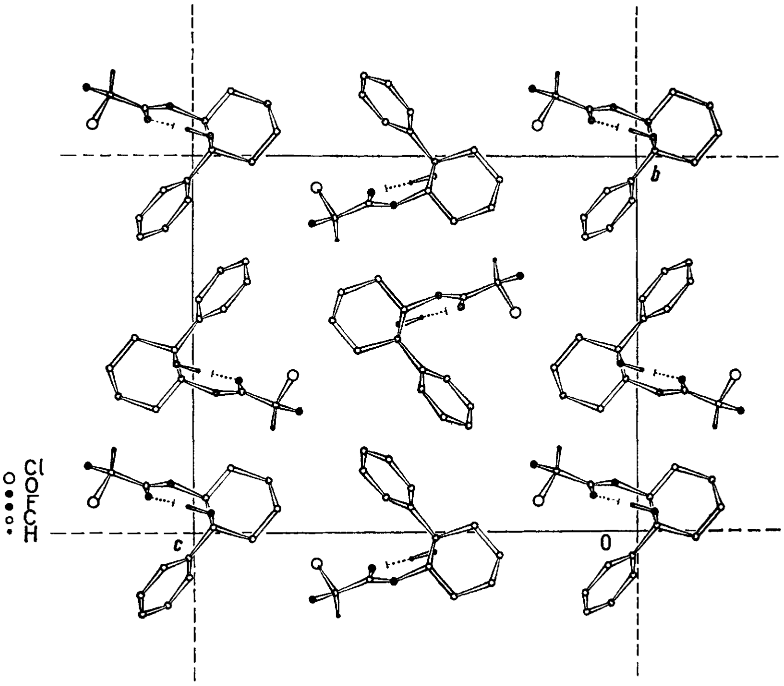

FIGURE 3 Projection of the structure on the crystallographic *bc* plane, with all hydrogen atoms except H(15) and H(16) omitted. Hydrogen bonds are shown by broken lines which are interrupted indicating that the hydrogen bond is to an atom which is not visible on the plane of the projection, being located at $x - 1$, directly beneath the atom shown. The co-ordinates given in Table 1 refer to the molecule at the centre of the Figure

A projection of the structure along the *a* axis is shown in Figure 3.

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