orientation seems to be governed by an attempt to minimize steric repulsion with (i) the carbonyl group and (ii) the triazolyl ring. Steric repulsion may also be responsible for the unusually large angle of 128·5 (7)° for C(31)—C(4)—C(5). The methoxy group [as defined by the plane containing atoms C(36), O(2) and C(21)] is inclined at an angle of 7·4 (10)° to its phenyl ring.

All four C—N distances in the triazolyl ring are shorter than the normal single bond as is the bond distance N(1)—N(2). The three atoms bonded to N(1) are coplanar with it and, taken together, these data indicate extensive delocalization in the heterocyclic ring. The most noteworthy feature of the triazolyl ring is the asymmetry of the exocyclic angles at N(1), C(4)—N(1)—C(1) being much larger than C(4)—N(1)—N(2). We have observed a similar pattern in related triazole systems (Nowell, Walker & Anderson, 1982; Branch & Nowell, 1985), which appears to be a function of the heterocyclic ring itself rather than the influence of any intra- or intermolecular interactions.

References

BALASUBRAMANYAN, S. & LEWIS, T. (1976). UK patent 1 580 535.
BRANCH, S. K. & NOWELL, I. W. (1985). Acta Cryst. C41, 594–597.
International Tables for X-ray Crystallography (1974). Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
MOTHERWELL, W. D. S. & CLEGG, W. (1978). PLUTO. Program for the production of crystal and molecular illustrations. Crystallographic Data Centre, Cambridge, England.
NOWELL, I. W., WALKER, P. E. & ANDERSON, N. H. (1982). Acta Cryst. B38, 1857–1859.
SHELDRICK, G. M. (1976). SHELX. Program for crystal structure determination. Univ. of Cambridge, England.
WILLIS, B. T. M. & PRYOR, A. W. (1975). Thermal Vibrations in Crystallography. Cambridge Univ. Press.

(1R,2R,3R,6S,10R)-1-Fluoro-3-(2-hydroxy-2-propyl)-6,10-dimethylbicyclo[4.4.0]decan-2-ol, C15H27FO2

BY R. G. PRITCHARD, R. RAMAGE AND I. A. SOUTHWELL

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England

Abstract. $M_r = 258·38$, orthorhombic, $P2_12_12_1$, $a = 7·458$ (6), $b = 13·436$ (6), $c = 14·736$ (5) Å, $V = 1476·6$ (23) Å^3, $Z = 4$, $D_x = 1·16$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 0·090$ mm^{-1}, $F(000) = 568$, $T = 293$ K, $R = 0·036$ for 1856 reflexions $[F > 3\sigma(F)]$. The cis-bridged bicyclodecane skeleton is in a slightly distorted double chair conformation with all the substituents, except the bulky 2-propanol group, in axial positions. The ring and isopropyl hydroxyl functions are linked by inter- and intramolecular hydrogen bonds.

Introduction. During the course of a study (Ramage & Southwell, 1984) of the rearrangement of the bio-genetically significant sesquiterpene rosifoliol (1) (Southwell, 1978), it was found that the corresponding β-epoxide (2) gave an unusual fluorinated product on treatment with boron trifluoride–diethyl ether. Owing to the importance of the rearrangement processes in biogenetic terms (MacSweeney, Ramage & Sattar,

1970), it was crucial to establish unambiguously the structure of the fluoroalcohol (3).

(1)   (2)   (3)
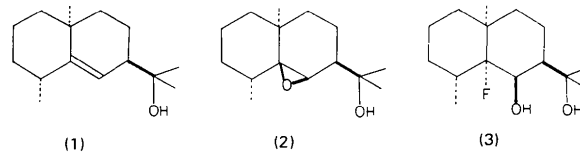
Experimental. Crystal: rectangular prism 0·2 × 0·2 × 0·4 mm, CAD-4 diffractometer, variation in 3 intensity standards <3%, unit-cell dimensions from 25 reflexions, 2658 reflexions measured, $0 \le h \le 8$, $0 \le k \le 15$, $-17 \le l \le 17$, $\theta < 25°$, 2084 unique (Friedel pairs not merged), $R_{Int} = 0·038$, 1856 structure amplitudes with $F > 3\sigma(F)$ considered observed; Lorentz and polarization but no absorption corrections; MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) used to solve structure; full-matrix

$C_{15}H_{27}FO_2$

Table 1. *Fractional atomic coordinates* $(\times 10^4)$ *and equivalent isotropic thermal parameters* $(\text{Å}^2 \times 10^4)$ *for the heavier atoms*

$$U_{eq} = \tfrac{1}{3}\Sigma_i \Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	U_{eq}
O(1)	6608 (3)	7774 (1)	4109 (1)	410
O(2)	8254 (3)	6711 (1)	5420 (1)	460
F(1)	6235 (2)	6262 (1)	2101 (1)	517
C(1)	6607 (3)	7199 (1)	2509 (1)	361
C(2)	6528 (4)	6920 (1)	3533 (1)	350
C(3)	8009 (3)	6213 (1)	3819 (1)	381
C(4)	9793 (4)	6684 (2)	3586 (2)	488
C(5)	9907 (4)	6849 (3)	2562 (2)	567
C(6)	8451 (3)	7525 (2)	2164 (1)	436
C(7)	8755 (4)	8617 (2)	2425 (2)	574
C(8)	7295 (5)	9301 (2)	2085 (2)	664
C(9)	5499 (5)	8992 (2)	2450 (2)	601
C(10)	5064 (4)	7899 (2)	2262 (2)	475
C(11)	7851 (4)	5880 (2)	4828 (1)	424
C(12)	9200 (6)	5081 (2)	5052 (2)	610
C(13)	5998 (5)	5517 (2)	5058 (2)	556
C(14)	4340 (5)	7759 (3)	1296 (2)	679
C(15)	8616 (5)	7416 (3)	1125 (2)	589

least-squares refinement, F magnitudes, using *SHELX* (Sheldrick, 1976); H atoms from difference Fourier synthesis; final $R = 0.036$, $wR = 0.046$, $w = 1.0567/[\sigma^2(F) + 0.000851F^2]$; heavier atoms treated anisotropically, hydrogen vibration parameters isotropic; max. $\Delta/\sigma = 0.027$ [z of H(14C)]; fluctuations in final difference map < 0.25 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Computations carried out on the joint CDC 7600/ICL 1906A system of the University of Manchester Regional Computing Centre. The Cambridge Crystallographic Database was surveyed using the *CSSR* (1980) interactive system accessed *via* the UMIST link to the DEC10 at Edinburgh.

Discussion. Atomic parameters are given in Table 1.* The molecular geometry is presented in Table 2 and illustrated stereoscopically in Fig. 1.

In common with other *cis*-bridged bicyclodecanes (Hamor & Hamor, 1976; Gatilov, Dubovenko & Khan, 1979) the title molecule has assumed a double chair conformation, which places the substituents, with the exception of the bulky 2-propanol group, in axial positions. On one side of the ring plane the hydroxyl O(1) takes part in a 2,7; 2,9 interaction, which tends to flatten the molecule, particularly in the bridge region [O(1)$\cdots$H(7A) 2.58 (5), O(1)$\cdots$H(9B) 2.45 (5) Å] while on the reverse side there is congestion due to the C(14) and C(15) methyl groups and the fluorine atom [F(1)$\cdots$H(14C) 2.34 (4), F(1)$\cdots$H(15A) 2.40 (4), H(14B)$\cdots$H(15C) 2.17 (7) Å].

---

* Lists of structure amplitudes, anisotropic thermal parameters, torsion angles and H parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39989 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

A hydrogen bond bridges the two hydroxyl groups intramolecularly [O(2)$\cdots$O(1) 2.698 (2), H(O2)$\cdots$O(1) 1.98 (4) Å], while another links the molecules in infinite spirals parallel to **a** [O(1)$\cdots$O(2^i) 2.687 (2), H(O1)$\cdots$O(2^i) 1.83 (4) Å; (i) denotes equivalent position $-\tfrac{1}{2}+x$, $1\tfrac{1}{2}-y$, $1-z$].

Table 2. *Bond lengths* (Å) *and angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

C(2)–O(1)	1.428 (2)	C(11)–O(2)	1.449 (3)
C(1)–F(1)	1.422 (2)	C(2)–C(1)	1.556 (3)
C(6)–C(1)	1.530 (3)	C(10)–C(1)	1.530 (4)
C(3)–C(2)	1.517 (3)	C(4)–C(3)	1.513 (4)
C(11)–C(3)	1.557 (3)	C(5)–C(4)	1.527 (4)
C(6)–C(5)	1.533 (4)	C(7)–C(6)	1.534 (4)
C(15)–C(6)	1.543 (3)	C(8)–C(7)	1.509 (5)
C(9)–C(8)	1.502 (5)	C(10)–C(9)	1.529 (4)
C(14)–C(10)	1.534 (4)	C(12)–C(11)	1.508 (4)
C(13)–C(11)	1.504 (4)		

C(6)–C(1)–F(1)	106.8 (2)	C(2)–C(1)–F(1)	100.9 (1)
C(10)–C(1)–F(1)	107.3 (2)	C(6)–C(1)–C(2)	115.1 (2)
C(10)–C(1)–C(6)	114.9 (2)	C(10)–C(1)–C(2)	110.5 (2)
C(3)–C(2)–O(1)	107.9 (2)	C(1)–C(2)–O(1)	112.4 (2)
C(4)–C(3)–C(2)	108.4 (2)	C(3)–C(2)–C(1)	113.1 (2)
C(11)–C(3)–C(4)	113.8 (2)	C(11)–C(3)–C(2)	113.0 (2)
C(6)–C(5)–C(4)	115.1 (2)	C(5)–C(4)–C(3)	109.5 (2)
C(7)–C(6)–C(1)	108.9 (2)	C(5)–C(6)–C(1)	109.9 (2)
C(15)–C(6)–C(1)	112.0 (2)	C(7)–C(6)–C(5)	111.5 (2)
C(15)–C(6)–C(7)	109.1 (2)	C(15)–C(6)–C(5)	105.5 (2)
C(9)–C(8)–C(7)	110.9 (2)	C(8)–C(7)–C(6)	113.1 (2)
C(9)–C(10)–C(1)	112.8 (2)	C(10)–C(9)–C(8)	112.9 (3)
C(14)–C(10)–C(9)	111.1 (3)	C(14)–C(10)–C(1)	114.2 (2)
C(12)–C(11)–O(2)	106.1 (2)	C(3)–C(11)–O(2)	109.8 (2)
C(13)–C(11)–O(2)	107.8 (2)	C(12)–C(11)–C(3)	111.3 (2)
C(13)–C(11)–C(12)	109.4 (2)	C(13)–C(11)–C(3)	112.2 (2)

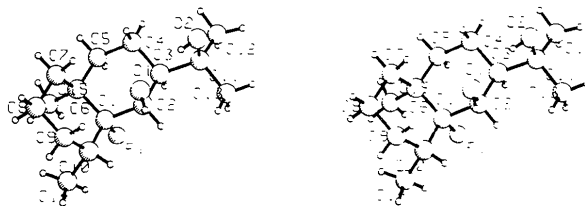| C(2)–C(1)–C(6)–C(5)	42.1 (4)
C(6)–C(1)–C(2)–C(3)	−48.6 (4)
C(6)–C(1)–C(10)–C(9)	−47.2 (4)
C(10)–C(1)–C(6)–C(7)	49.9 (4)
O(1)–C(2)–C(3)–C(11)	58.8 (4)
C(1)–C(2)–C(3)–C(4)	56.8 (4)
C(2)–C(3)–C(4)–C(5)	−61.3 (4)
C(2)–C(3)–C(11)–O(2)	−70.0 (4)
C(3)–C(4)–C(5)–C(6)	59.7 (4)
C(4)–C(5)–C(6)–C(1)	−48.5 (4)
C(1)–C(6)–C(7)–C(8)	−55.7 (4)
C(6)–C(7)–C(8)–C(9)	58.7 (4)
C(7)–C(8)–C(9)–C(10)	−53.7 (4)
C(8)–C(9)–C(10)–C(1)	48.1 (4)

Fig. 1. Stereoscopic view of the molecule produced using *PLUTO* (Motherwell & Clegg, 1978). Hydrogen atoms bear in the text labels related to those of the atoms to which they are attached with an additional final letter (*A*, *B*, *C*) where necessary.

References

CSSR (1980). *Crystal Structure Search and Retrieval. Instruction Manual.* Daresbury Laboratory, SERC, England.

GATILOV, YU. V., DUBOVENKO, ZH. V. & KHAN, V. A. (1979). *Zh. Strukt. Khim.* **20**, 509–513.

HAMOR, M. J. & HAMOR, T. A. (1976). *J. Chem. Soc. Perkin Trans.* 2, pp. 383–387.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

MACSWEENEY, D. F., RAMAGE, R. & SATTAR, A. (1970). *Tetrahedron Lett.* pp. 557–560.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO. Program for plotting molecular and crystal structures.* Univ. of Cambridge, England.

RAMAGE, R. & SOUTHWELL, I. A. (1984). *J. Chem. Soc. Perkin Trans.* 1, pp. 1323–1328.

SHELDRICK, G. M. (1976). *SHELX76. Program for crystal structure determination.* Univ. of Cambridge, England.

SOUTHWELL, I. A. (1978). *Aust. J. Chem.* pp. 2527–2538.

Structure of [2.2.2.2]Paracyclophane,* $C_{32}H_{32}$

BY ANNE RENAULT AND CLAUDINE COHEN-ADDAD

Université Scientifique et Médicale de Grenoble, Laboratoire de Spectrométrie Physique, Boîte postale n° 87, 38402 Saint-Martin-d'Hères CEDEX, France

Abstract. $M_r = 416 \cdot 6$, monoclinic, $P2_1/c$, $a = 12 \cdot 026$ (3), $b = 9 \cdot 731$ (3), $c = 20 \cdot 978$ (6) Å, $\beta = 92 \cdot 35$ (5)°, $V = 2455$ Å^3, $Z = 4$, $D_x = 1 \cdot 13$ Mg m^{-3}, Ni-filtered Cu $K\alpha$ radiation, $\lambda = 1 \cdot 5418$ Å, $\mu = 0 \cdot 482$ mm^{-1}, $F(000) = 896$, $T = 293$ K, $R = 0 \cdot 061$ for 2763 observed reflexions. The observed *gauche* conformation of the 4-PCP molecule is in agreement with previous NMR studies in solution. The four benzene rings have a non-planar arrangement analogous to that observed in the homologous compound, 4-PCP–TCNE.

Introduction. This work is part of a series of structural studies on paracyclophanes and their complexes with metal ions or electron-acceptor molecules such as tetracyanoethylene, TCNE (Pierre, Baret, Chautemps & Armand, 1981; Cohen-Addad, Baret, Chautemps & Pierre, 1983; Cohen-Addad, Lebars, Renault & Baret, 1984). We have previously studied the complex [2.2.2.2]paracyclophane–TCNE. For comparison, the structure determination of [2.2.2.2]paracyclophane (4-PCP) was undertaken.

Experimental. Compound prepared by P. Baret and P. Chautemps (Laboratoire d'Etudes Dynamiques et Structurales de la Sélectivité, Grenoble). Colourless $0 \cdot 35 \times 0 \cdot 40 \times 0 \cdot 45$ mm crystal obtained by slow evaporation at 277 K from a solution in chloroform.

Measurements with a Siemens diffractometer,† Ni-filtered Cu $K\alpha$ radiation, five-points method (Troughton, 1969), ω-scan, no absorption correction. Lattice parameters refined with 15 reflexions. 2763 reflexions with $I > 3\sigma(I)$ (4205 measured), $\sin\theta/\lambda < 0 \cdot 58$ Å^{-1} (hkl, $\bar{h}kl$). Standard reflexions $\bar{4}08$, $\bar{4}25$, $\bar{2}38$ (3% variation). Multisolution direct method (Germain, Main & Woolfson, 1971), least-squares refinement minimizing $\sum w |F_o - F_c|^2$ (Busing & Levy, 1962). Positional and anisotropic parameters refined for C atoms, H-atom positional parameters calculated (not refined), isotropic temperature factors for H estimated (not refined). Weighting scheme approximately linear, obtained empirically by plotting $(F_o - F_c)^2$ as a function of F_o (Rollett, 1965). Final $R = 0 \cdot 061$, $wR = 0 \cdot 084$ for 2763 reflexions. $(\Delta/\sigma)_{max} = 0 \cdot 8$, final $\Delta\rho < 0 \cdot 25$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The atomic parameters are listed in Table 1; interatomic distances, valence and torsion angles in Table 2.‡

---

* *Chemical Abstracts* name: pentacyclo[20.2.2.2^{4,7}.2^{10,13}.2^{16,19}]-dotriaconta-4,6,10,12,16,18,22,24,25,27,29,31-dodecaene.

† Measurements performed at Laue–Langevin Institute, Grenoble, France.

‡ Lists of structure factors, anisotropic thermal parameters, calculated H-atom parameters and mean-plane equations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42031 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.