Table 2. *Selected geometric parameters (Å, °)*

O(1)—C(10)	1.210 (3)	O(2)—C(11)	1.259 (4)
O(3)—C(11)	1.252 (3)	C(1)—C(2)	1.563 (4)
C(1)—C(9)	1.551 (3)	C(1)—C(10)	1.524 (3)
C(1)—C(11)	1.525 (3)	C(2)—C(3)	1.503 (3)
C(3)—C(4)	1.406 (3)	C(3)—C(12)	1.385 (3)
C(4)—C(5)	1.504 (3)	C(4)—C(15)	1.389 (4)
C(5)—C(6)	1.553 (5)	C(6)—C(7)	1.535 (3)
C(6)—C(10)	1.510 (3)	C(7)—C(8)	1.522 (4)
C(8)—C(9)	1.527 (4)	C(12)—C(13)	1.381 (4)
C(13)—C(14)	1.372 (5)	C(14)—C(15)	1.389 (5)
O(2)···O(3^{i})	2.647 (3)		
C(2)—C(1)—C(9)	111.0 (2)	C(2)—C(1)—C(10)	110.3 (2)
C(9)—C(1)—C(10)	107.9 (2)	C(2)—C(1)—C(11)	111.0 (2)
C(9)—C(1)—C(11)	110.5 (2)	C(10)—C(1)—C(11)	105.9 (2)
C(1)—C(2)—C(3)	115.9 (2)	C(2)—C(3)—C(4)	119.7 (2)
C(2)—C(3)—C(12)	120.9 (2)	C(4)—C(3)—C(12)	119.3 (2)
C(3)—C(4)—C(5)	119.2 (2)	C(3)—C(4)—C(15)	118.6 (2)
C(5)—C(4)—C(15)	122.2 (2)	C(4)—C(5)—C(6)	115.2 (2)
C(5)—C(6)—C(7)	112.7 (2)	C(5)—C(6)—C(10)	112.0 (2)
C(7)—C(6)—C(10)	107.9 (2)	C(6)—C(7)—C(8)	112.4 (2)
C(7)—C(8)—C(9)	111.5 (2)	C(1)—C(9)—C(8)	112.8 (2)
O(1)—C(10)—C(1)	121.8 (2)	O(1)—C(10)—C(6)	122.5 (2)
C(1)—C(10)—C(6)	115.6 (2)	O(2)—C(11)—O(3)	122.1 (2)
O(2)—C(11)—C(1)	118.5 (2)	O(3)—C(11)—C(1)	119.3 (3)
C(3)—C(12)—C(13)	121.6 (3)	C(12)—C(13)—C(14)	119.1 (3)
C(13)—C(14)—C(15)	120.5 (3)	C(4)—C(15)—C(14)	120.9 (3)
C(11)—O(2)···O(3^{i})	119.2 (2)	C(11)—O(3)···O(2^{i})	118.6 (2)

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
C(14)—H(14A)···O(1^{ii})	2.570 (4)	3.513 (4)	169.4 (1)

Symmetry codes: (i) $1 - x, -y, 2 - z$; (ii) $1 - x, -y, 1 - z$.

The structure was solved by direct methods. Integrated intensities were corrected for Lorentz and polarization effects. Full-matrix least-squares refinement using *SHELXTL/PC* (Sheldrick, 1990) was performed. All non-H atoms were refined anisotropically. Although most H atoms were found in difference maps, they were nonetheless replaced by H atoms in calculated positions and refined with a riding model, which gave a slightly better goodness-of-fit.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SZ1015). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C52, 687–689

Methyl 3-(*RS*)-Hydroxy-4-[(*RS*)-3,4,5,6-tetrahydro-2*H*-2-pyranyloxy]cyclohexanecarboxylate

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Abstract

In the title compound, $C_{13}H_{22}O_5$, an intramolecular hydrogen bond between the hydroxy group and the O atom of the tetrahydropyranyl moiety results in a seven-membered ring which displays a twist-chair conformation.

Comment

Interest in the structure of substituted cyclohexanes arises from their presence in immunosuppressant macrolides like FK 506 and rapamycin. An *ORTEP*II

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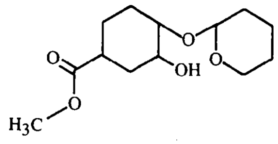
(Johnson, 1976) view of the title molecule, (I), showing the atom-numbering scheme and the intramolecular hydrogen bond, is presented in Fig. 1.



(I)

The molecule of (I) contains a tetrahydropyranyl group attached equatorially to the substituted cyclohexane ring. Both the cyclohexane and tetrahydropyranyl rings are in chair conformations. The equatorial methyl ester group is in a synperiplanar arrangement as the carbonyl O(1) atom is twisted away from the C(3) eclipsed position [O(1)—C(7)—C(4)—C(3) 29.1 (4)°]. The equatorial hydroxy group [O(3)] facilitates the formation of an intramolecular O—H···O hydrogen bond [O(3)···O(5) 2.766 (3), H(3*A*)···O(5) 1.939 (3) Å, O(3)—H(3*A*)···O(5) 163.7 (2)°] with the adjacent tetrahydropyranyl ring which results in a pseudo-seven-membered ring. This pseudo-seven-membered ring exhibits a twist-chair conformation as shown by the ring-puckering parameters (Cremer & Pople, 1975): $\varphi_2 = 176.5\,(14)$, $\varphi_3 = -125.7\,(2)°$, $q_2 = 0.107\,(2)$, $q_3 = 0.655\,(3)$ Å, $\theta_2 = 9.3\,(2)°$, $q_T = 0.664\,(3)$ Å. The crystal packing consists of discrete molecules separated by normal van der Waals interactions.

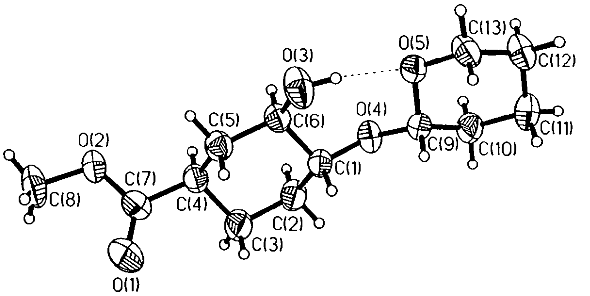


Fig 1. A view of the molecule with the atomic numbering scheme and the intramolecular hydrogen bond. Anisotropic displacement parameters are at the 50% probability level and H atoms are drawn as circles of arbitrary radii.

Experimental

The compound was synthesized by a similar route to that reported by Corey & Huang (1989) and crystals suitable for X-ray work were obtained by recrystallization from a mixture of ethyl acetate and petroleum ether (9:1).

Crystal data

$C_{13}H_{22}O_5$
$M_r = 258.3$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å

Triclinic
$P\bar{1}$
$a = 9.458\,(1)$ Å
$b = 9.644\,(1)$ Å
$c = 9.889\,(1)$ Å
$\alpha = 94.81\,(2)°$
$\beta = 117.76\,(2)°$
$\gamma = 114.16\,(2)°$
$V = 684.9\,(3)$ Å^3
$Z = 2$
$D_x = 1.253$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 8–13°$
$\mu = 0.089$ mm^{-1}
$T = 293$ K
Block
0.16 × 0.15 × 0.14 mm
Colourless

Data collection

Siemens *R*3*m*/*V* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1979 measured reflections
1937 independent reflections
1519 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int} = 0.019$
$\theta_{\rm max} = 22.5°$
$h = 0 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 13$
2 standard reflections monitored every 98 reflections
intensity decay: <1%

Refinement

Refinement on *F*
$R = 0.053$
$wR = 0.060$
$S = 0.861$
1519 reflections
163 parameters
$w = 1/[\sigma^2(F) + 0.0721F^2]$

$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.23$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1991)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	*y*	*z*	$U_{\rm eq}$
O(1)	−0.0429 (3)	1.2225 (3)	0.1778 (3)	0.062 (1)
O(2)	0.2412 (2)	1.3793 (2)	0.2390 (2)	0.058 (1)
O(3)	0.4204 (3)	1.1236 (2)	0.7119 (2)	0.065 (1)
O(4)	0.2711 (2)	0.7894 (2)	0.4992 (3)	0.045 (1)
O(5)	0.4631 (2)	0.8645 (2)	0.7749 (2)	0.047 (1)
C(1)	0.1950 (3)	0.8938 (3)	0.4551 (3)	0.041 (1)
C(2)	0.1023 (3)	0.8500 (3)	0.2727 (3)	0.050 (1)
C(3)	0.0204 (3)	0.9560 (3)	0.2077 (3)	0.055 (1)
C(4)	0.1784 (3)	1.1340 (3)	0.2913 (3)	0.042 (1)
C(5)	0.2684 (3)	1.1800 (3)	0.4750 (3)	0.045 (1)
C(6)	0.3450 (3)	1.0726 (3)	0.5425 (2)	0.042 (1)
C(7)	0.1089 (3)	1.2455 (3)	0.2289 (3)	0.049 (1)
C(8)	0.1919 (4)	1.4945 (3)	0.1810 (3)	0.072 (1)
C(9)	0.2857 (3)	0.7438 (2)	0.6344 (3)	0.039 (1)
C(10)	0.2743 (3)	0.5813 (3)	0.6105 (3)	0.052 (1)
C(11)	0.3056 (3)	0.5342 (3)	0.7609 (3)	0.058 (1)
C(12)	0.4861 (4)	0.6673 (3)	0.9118 (3)	0.057 (1)
C(13)	0.4909 (3)	0.8272 (3)	0.9206 (3)	0.053 (1)

Table 2. *Selected geometric parameters (Å, °)*

O(1)—C(7)	1.188 (4)	O(2)—C(7)	1.334 (3)
O(2)—C(8)	1.437 (4)	O(3)—C(6)	1.421 (3)
O(4)—C(1)	1.448 (4)	O(4)—C(9)	1.406 (3)
O(5)—C(9)	1.435 (2)	O(5)—C(13)	1.444 (3)
C(4)—C(7)	1.510 (4)	C(9)—C(10)	1.517 (4)
C(7)—O(2)—C(8)	117.3 (2)	C(1)—O(4)—C(9)	118.2 (2)
C(9)—O(5)—C(13)	111.3 (2)	O(4)—C(1)—C(2)	105.4 (2)
O(4)—C(1)—C(6)	112.0 (2)	C(3)—C(4)—C(5)	109.4 (2)
C(3)—C(4)—C(7)	111.5 (2)	C(5)—C(4)—C(7)	110.2 (2)

O(3)—C(6)—C(1)	113.0 (2)	O(3)—C(6)—C(5)	106.4 (2)
O(1)—C(7)—O(2)	122.2 (3)	O(1)—C(7)—C(4)	126.1 (3)
O(2)—C(7)—C(4)	111.8 (2)	O(4)—C(9)—O(5)	107.3 (2)
O(4)—C(9)—C(10)	108.4 (2)	O(5)—C(9)—C(10)	110.1 (2)
O(5)—C(13)—C(12)	111.5 (2)		

H atoms were located from difference Fourier maps, positioned geometrically and included as riding atoms with fixed isotropic displacement parameters in the structure-factor calculations.

Data collection: *R3m/V* diffractometer software. Cell refinement: *R3m/V* diffractometer software. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1053). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A New Pyrethroid Insecticide, RU41414

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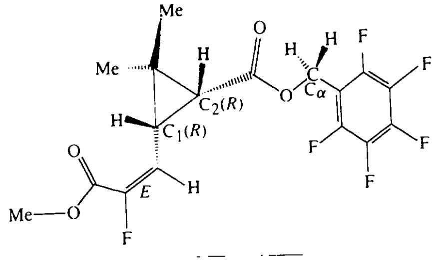
(*Received 28 July 1995; accepted 21 September 1995*)

Abstract

An X-ray diffraction study of RU41414, methyl 3-[2,2-dimethyl-3-(pentafluorophenylmethyloxycarbonyl)-cyclopropyl]-2-fluoropropenoate, $C_{17}H_{14}F_6O_4$, establishes the molecular structure, the configuration (*R*) of the asymmetric C atoms C12 and C8 of the cyclopropane ring and the stereochemistry of the propenoate C5═C7 double bond.

Comment

Biological activity in pyrethroids is related to molecular structure and depends strongly on the stereochemistry at the three centres C_1, C_2 and C_α. We report here the structure of RU41414, a useful pyrethroid insecticide.



RU41414

The cyclopropane ring has a mean bond length of 1.524 (10) Å, which is in the expected range as found from earlier studies (Hamzaoui, Lamiot & Baert, 1993; Baert, Guelzim & Germain, 1991). The average C—F bond length in the pentafluorophenyl ring is 1.337 (12) Å and the C—C distances in this ring vary between 1.318 (17) and 1.384 (12) Å. Knowledge of the stereochemistry of the C4═C5 double bond allows chemists to predict precisely the activity of the insecticide concerned (Tessier, Teche & Demoute, 1982).

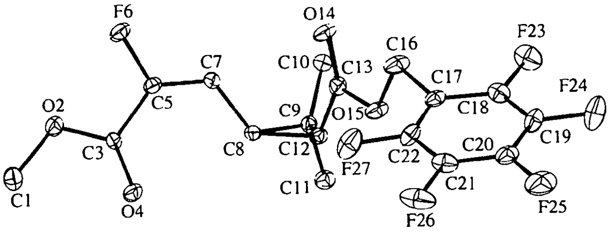


Fig. 1. An *ORTEP*II (Johnson, 1976) view of RU41414 showing the atom-numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

Single crystals were grown at room temperature by slow evaporation of an aqueous solution of RU41414.

Crystal data

$C_{17}H_{14}F_6O_4$
$M_r = 396.29$
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

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