same side of the plane. In mitomycin A these atoms are located on the opposite sides. The magnitude of the deviations in the present compound, however, is significantly smaller than those in the two crystallographically independent molecules of mitomycin A and this is possible due to the exocyclic double bond.

The present analysis has revealed that the influence of the exocyclic double bond at C(9) is spread over the molecule. Since the extent is unexpectedly wide the mode of the biological activities of the title compound is possibly quite different from other members of mitomycin family with a carbamate group at C(9).

There is one intermolecular hydrogen bond between the N atom (x, y, z) in the aziridine ring and the hydroxyl group $(-\frac{1}{2}+x, \frac{1}{2}-y, 1-z)$. The hydrogen-bond geometries are as follows. $N(1)\cdots O(9a) = 3\cdot023$ (2), $N(1)\cdots H[O(9a)] = 1\cdot96$ (3) Å and $\angle N(1)\cdots H-O(9a) = 154$ (2)°. Other intermolecular interactions are within van der Waals contacts.

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X-ray Structure of the Pyrethroid Insecticide {1R-[1α(S*),2α]}-2-(2,2-Dichlorovinyl)-3,3-dimethylcyclopropanecarboxylic Acid Cyano(3-phenoxyphenyl)methyl Ester (RU 24501)

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Abstract. α-Cyano-3-phenoxybenzyl {1R-[1α(S*),2α]}-2-(2,2-dichlorovinyl)-3,3-dimethylcyclopropanecarboxylate. The crystal structure of one pyrethroid insecticide patented by Roussel UCLAF has been determined at 293 K by X-ray diffraction. The crystal is orthorhombic, $P2_12_12_1$, $C_{22}H_{19}Cl_2NO_3$ (RU 24501), $M_r = 416\cdot30$, $a = 9\cdot296$ (3), $b = 35\cdot853$ (9), $c = 6\cdot212$ (3) Å, $Z = 4$, $V = 2070\cdot4$ Å^3, $D_x = 1\cdot34$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot7107$ Å, $\mu = 0\cdot288$ mm^{-1}, $F(000) = 864$. The structure was refined from 1526 reflections with $I > 3\sigma(I)$ to $R = 0\cdot059$. All H atoms were found on a difference map. The conformation of the compound is compared with those of other known pyrethroid structures in the crystalline state.

Introduction. Considerable progress has been made in relating the structure of pyrethroids with their biological activity but improvement of such concepts

needs reliable information on molecular shape (configuration, bond lengths and angles and conformation). As part of this work the results of the structural determination of {1R-[1α(S*),2α]}-cyano-3-phenoxybenzyl 2-(2,2-dichlorovinyl)-3,3-dimethylcyclopropanecarboxylate, (RU 24501), will be compared to those of the isomorphous dibromo derivative (III) (Owen, 1975) and to two pyrethroid insecticides cis-3-phenoxybenzyl 2-(2,2-dibromovinyl)-3,3-dimethylcyclopropane carboxylate (I) and the 2-(2,2-dichlorovinyl) (II) analogue (Owen, 1976). The general formula of these compounds is depicted below.
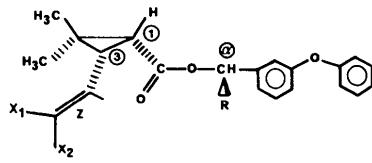
(I) $X_1 = X_2 =$ Br, $R =$ H; (II) $X_1 = X_2 =$ Cl, $R =$ H; (III) $X_1 = X_2 =$ Br, $R =$ CN; (IV) $X_1 = X_2 =$ Cl, $R =$ CN

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^3$)

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

	x	y	z	U_{eq}
Cl(1)	−9238 (1)	2536·6 (6)	−13274 (2)	75 (2)
Cl(2)	−12177 (1)	2502·7 (6)	−11914 (2)	75 (2)
C(19)	−9285 (6)	−1012 (1)	−7575 (8)	45 (7)
O(10)	−10608 (3)	759·6 (9)	−11029 (5)	39 (4)
O(11)	−9534 (5)	508 (1)	−11851 (8)	42 (7)
O(9)	−8928 (3)	1214·1 (8)	−11091 (6)	57 (5)
O(18)	−9191 (4)	−624·4 (9)	−7705 (5)	55 (5)
C(3)	−11311 (5)	1758 (1)	−9681 (7)	43 (7)
C(20)	−8913 (5)	−1236 (1)	−9233 (8)	53 (8)
C(5)	−12073 (5)	1566 (1)	−5842 (7)	52 (8)
C(8)	−10120 (5)	1112 (1)	−10611 (8)	39 (7)
C(4)	−10922 (5)	1571 (1)	−7570 (7)	36 (6)
C(16)	−11668 (5)	−390 (1)	−12107 (8)	43 (7)
C(7)	−11249 (4)	1330 (1)	−9527 (8)	36 (6)
C(14)	−9884 (5)	−445 (1)	−9403 (9)	45 (7)
C(15)	−11031 (5)	−597 (1)	−10485 (8)	45 (7)
C(13)	−9388 (5)	−87 (1)	−9861 (8)	33 (7)
C(1)	−10518 (5)	2294 (1)	−11848 (7)	42 (7)
C(21)	−8936 (6)	−1622 (1)	−8917 (8)	50 (8)
C(25)	−9359 (6)	580 (1)	−14207 (9)	56 (8)
C(24)	−9641 (5)	−1158 (2)	−5588 (9)	60 (8)
C(22)	−9302 (6)	−1773 (1)	−7009 (9)	64 (9)
N(26)	−9212 (6)	629 (1)	−16017 (7)	85 (9)
C(6)	−9404 (5)	1604 (1)	−6736 (7)	47 (7)
C(23)	−9637 (5)	−1542 (2)	−5339 (8)	75 (9)
C(12)	−10046 (5)	114 (1)	−11470 (8)	38 (7)
C(2)	−10255 (5)	1972 (1)	−10924 (8)	42 (7)
C(17)	−11165 (5)	−30 (1)	−12589 (7)	46 (7)
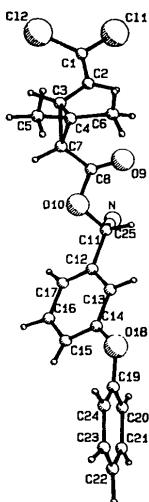
Fig. 1. Perspective view of the molecule and key to the numbering of the molecule.

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

Cl(1)—Cl(2)	2·863 (2)	C(25)—N(26)	1·146 (7)
Cl(1)—C(1)	1·719 (5)	C(24)—C(23)	1·385 (8)
Cl(2)—C(1)	1·715 (5)	C(22)—C(23)	1·364 (8)
C(19)—O(18)	1·394 (5)	C(12)—C(17)	1·355 (6)
C(19)—C(20)	1·352 (7)	C(19)—HC(21)	1·412 (5)
C(19)—C(24)	1·382 (7)	C(11)—HC(11)	0·980 (5)
O(10)—C(11)	1·439 (5)	C(3)—HC(3)	1·067 (5)
O(10)—C(8)	1·367 (5)	C(20)—HC(20)	0·927 (5)
C(11)—C(25)	1·495 (8)	C(20)—HC(21)	1·288 (5)
C(11)—C(12)	1·509 (6)	C(5)—H'C(5)	0·943 (5)
O(9)—C(8)	1·204 (6)	C(5)—HC(5)	0·956 (6)
O(18)—C(14)	1·393 (6)	C(5)—H''C(5)	1·003 (4)
C(3)—C(4)	1·515 (6)	C(16)—HC(16)	1·013 (5)
C(3)—C(7)	1·537 (6)	C(7)—HC(7)	1·007 (4)
C(3)—C(2)	1·467 (6)	C(15)—HC(15)	0·904 (5)
C(20)—C(21)	1·396 (7)	C(15)—HC(21)	1·872 (5)
C(5)—C(4)	1·516 (6)	C(13)—HC(13)	1·022 (5)
C(8)—C(7)	1·472 (6)	C(1)—HC(2)	1·930 (5)
C(4)—C(7)	1·523 (6)	C(21)—HC(21)	0·935 (6)
C(4)—C(6)	1·507 (6)	C(22)—HC(22)	1·111 (6)
C(16)—C(15)	1·383 (7)	C(6)—H''C(6)	0·975 (5)
C(16)—C(17)	1·406 (6)	C(6)—H'C(6)	0·974 (5)
C(14)—C(15)	1·373 (7)	C(6)—HC(6)	0·891 (5)
C(14)—C(13)	1·395 (6)	C(23)—HC(23)	1·001 (5)
C(13)—C(12)	1·377 (6)	C(2)—HC(2)	1·008 (4)
C(1)—C(2)	1·312 (6)	C(17)—HC(17)	0·996 (5)

O(18)—C(14)—C(15)	123·2 (4)	C(11)—O(10)—C(8)	114·6 (3)
O(18)—C(14)—C(13)	115·2 (4)	O(10)—C(11)—C(25)	108·4 (4)
C(15)—C(14)—C(13)	121·4 (4)	O(10)—C(11)—C(12)	108·2 (4)
C(16)—C(15)—C(14)	118·5 (4)	C(25)—C(11)—C(12)	110·4 (4)
C(14)—C(13)—C(12)	119·0 (4)	C(19)—O(18)—C(14)	118·3 (4)
Cl(1)—C(1)—Cl(2)	112·9 (2)	C(4)—C(3)—C(7)	59·9 (3)
Cl(1)—C(1)—C(2)	122·7 (4)	C(4)—C(3)—C(2)	121·8 (4)
Cl(2)—lC(1)—C(2)	124·2 (4)	C(7)—C(3)—C(2)	122·1 (4)
C(20)—C(21)—C(22)	121·8 (5)	C(19)—C(20)—C(21)	118·6 (5)
C(11)—C(25)—N(26)	178·8 (6)	O(10)—C(8)—O(9)	122·7 (4)
C(19)—C(24)—C(23)	118·5 (5)	O(10)—C(8)—C(7)	109·9 (4)
C(21)—C(22)—C(23)	118·8 (5)	O(9)—C(8)—C(7)	127·4 (4)
C(24)—C(23)—C(22)	121·3 (5)	C(3)—C(4)—C(5)	116·7 (4)
C(11)—C(12)—C(13)	117·8 (4)	C(3)—C(4)—C(7)	60·8 (3)
C(11)—C(12)—C(17)	121·4 (4)	C(3)—C(4)—C(6)	119·1 (4)
C(13)—C(12)—C(17)	120·8 (4)	C(5)—C(4)—C(7)	114·7 (4)
C(3)—C(2)—C(1)	124·5 (4)	C(5)—C(4)—C(6)	114·7 (4)
C(16)—C(17)—C(12)	119·9 (4)	C(7)—C(4)—C(6)	120·4 (4)
Cl(2)—Cl(1)—C(1)	33·5 (1)	C(15)—C(16)—C(17)	120·3 (4)
Cl(1)—Cl(2)—C(1)	33·6 (1)	C(3)—C(7)—C(8)	121·8 (4)
O(18)—C(19)—C(20)	122·2 (4)	C(3)—C(7)—C(4)	59·4 (3)
O(18)—C(19)—C(24)	116·5 (4)	C(8)—C(7)—C(4)	121·6 (4)
C(20)—C(19)—C(24)	121·0 (5)		

Experimental. A needle-shaped crystal of dimensions $0.18 \times 0.34 \times 0.61$ mm was mounted on a Philips four-circle diffractometer. Intensities measured by $\omega/2\theta$ scan technique using Mo $K\alpha$ radiation, scan width = 1·4°, scan speed 0·020° s^{-1} and $0 < h < 13$, $0 < k < 50$, $0 < l < 8$. Lattice parameters from 25 reflexions ($20 < \theta < 27$). Three standard reflexions monitored every two hours, no significant variation was observed. Lp correction. Absorption and extinc-

tion ignored. The structure was determined by considering 1526 reflections with $I > 3\sigma(I)$.

Direct methods using 499 reflections with normalized structure factors $E > 1.55$ in *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All H atoms obtained from a difference Fourier map. The non-H and H atoms refined anisotropically and isotropically respectively, R factor converged to 5·9% for all reflections with $I > 3\sigma(I)$, $wR = 5.9\%$, $S = 5.6$, $w = 1$. $\Delta/\sigma_{max} = 0.014$ and $|\Delta\rho|_{max} = 0.28$ e Å^{-3}. Scattering factor curves for the Cl, C, O and N atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV) while the factors for H were those of Stewart, Davidson & Simpson (1965).

Table 3. *Torsion angles* (°) *and dihedral angles* (°) *between planes*

Torsion angles

	(I)	(II)	(III)	(IV)
C(1)—C(2)—C(3)—C(4)	−148 (1)	−146 (2)	−90 (5)	−137·0 (6)
C(1)—C(2)—C(3)—C(7)	141 (2)	141 (2)	−161 (4)	151·0 (6)
C(3)—C(7)—C(8)—O(9)	41 (2)	36 (3)	39 (6)	26·2 (4)
C(4)—C(7)—C(8)—O(9)	−30 (3)	−36 (3)	−36 (6)	−45·0 (5)
O(9)—C(8)—O(10)—C(11)	5 (2)	6 (2)	7 (5)	7·0 (4)
C(8)—O(10)—C(11)—C(12)	93 (2)	95 (2)	104 (3)	159·6 (5)
O(10)—C(11)—C(12)—C(13)	7 (2)	3 (2)	−106 (3)	−106·2 (5)
C(13)—C(14)—O(18)—C(19)	−30 (2)	−27 (3)	−136 (3)	−160·0 (6)
C(14)—O(18)—C(19)—C(24)	−55 (2)	−56 (2)	21 (5)	−132·3 (6)

Dihedral angles between planes

Planes*	(I)	(II)	(III)	(IV)
(1)—(2)	114	116	35	48
(1)—(3)	137	134	90	54
(1)—(4)	115	116	92	64
(2)—(3)	81	80	78	75
(2)—(4)	11	10	126	61
(3)—(4)	73	72	124	118

* These planes are defined in the deposited material.

The resulting atomic coordinates appear in Table 1* and the atom numbering in Fig. 1. Table 2 shows the bond lengths and angles. The absolute configuration of (IV) has not been established for this structure determination but is known to be *cis* (1*R*,2*R*) for the cyclopropane and *S* for the benzylic *α*-C atom.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53356 (27pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. According to the substituents (*X* = Br, Cl and *R* = CN, H), we have four different pyrethroid molecules. Planes and torsion angles are defined as in Owen (1976). From Table 3, it is seen that two molecular conformations dominate in these four molecules. (I) is similar to (II) but different from (III) and (IV). The conformation of the molecule is certainly more dependent on the *R* substituent (CN, H) than the *X* one (Cl, Br).

Molecule (IV) (*X* = Cl, *R* = CN, our study) is more elongated than (III) where the Br atoms are pushed far away from the CN group due to electronic repulsion [torsion angles C(1)—C(2)—C(3)—C(4) and C(1)—C(2)—C(3)—C(7) are −90 (III), −137 (IV), −161 (III), 151° (IV)]. The crystal structures of the pyrethroids evoked here show that there is a certain degree of flexibility at each end of the molecule with the ester linkage in the middle forming a fairly rigid entity.

No intermolecular distances between molecules are less than the sum of the van der Waals radii of the atoms involved.

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Structure of 5-Methoxy-3-(1-methylethoxy)-1-phenyl-*N*-(1*H*-tetrazol-5-yl)-1*H*-indole-2-carboxamide–Diethylamine, a Potential Anti-Allergy Agent

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Abstract. $C_{24}H_{31}N_7O_3$, $M_r = 465·56$, monoclinic, $P2_1/n$, $a = 14·439$ (3), $b = 9·147$ (2), $c = 19·207$ (5) Å, $\beta = 90·89$ (2)°, $V = 2536·4$ Å³, $Z = 4$, $D_x = 1·219$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0·71073$ Å, $\mu = 0·078$ mm⁻¹, $F(000) = 992$, $T = 293$ K, final $R = 0·052$ for 2731 observed reflections with $I > 3\sigma(I)$. The indole moiety is essentially planar, with the phenyl ring inclined at 68·03 (8)° to it. The tetrazole

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