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Structure of the 2*R*,3*R*/2*S*,3*S* Diastereoisomer of 1-(2-Fluorophenyl)-4,4-dimethyl-2-(1*H*-1,2,4-triazol-1-yl)-3-pentanol

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Abstract. $C_{15}H_{20}FN_3O$, $M_r = 277.3$, $P2_1/c$, $a = 9.500$ (4), $b = 12.389$ (5), $c = 13.019$ (5) Å, $\beta = 101.92$ (3)°, $V = 1499.2$ Å³, $Z = 4$, $D_m = 1.25$ (2), $D_x = 1.23$ g cm⁻³, $Mo K\alpha$, $\lambda = 0.71069$ Å, $\mu = 0.53$ cm⁻¹, $F(000) = 592$, room temperature, final $R = 0.047$ for 1364 observed reflections. The carbon atoms of the pentanol function adopt an extended 'W' conformation and the atoms are essentially coplanar. The triazolyl ring, which shows significant delocalization, and the fluorophenyl ring are twisted well out of the plane of the pentanol carbon chain. The exocyclic angles of the triazolyl ring are very asymmetric with C–N–C, 129.9 (3)°, significantly larger than C–N–N, 120.6 (3)°. There is intermolecular hydrogen bonding between the 4-aza N atom of the triazolyl ring and the hydroxyl group of a neighbouring molecule, $N \cdots H(O) = 1.88$ Å, such that molecules of the 2*R*,3*R* and 2*S*,3*S* configurations are linked alternately into chains.

Introduction. The title compound is an analogue of the systemic fungicide diclobutrazol (Balasubramanian & Shephard, 1975). The mode of action is believed to be the inhibition of fungal ergosterol biosynthesis and the *RR*,*SS* diastereoisomer is more active than the *RS*,*SR* diastereoisomer (Gadher, Mercer, Baldwin & Wiggins, 1983). Structure–activity relationships indicate that the triazolyl and hydroxyl groups play important roles while the substituents in the phenyl ring may have significant effects on molecular conformation and lipophilicity (Marchington, 1978). Thus, we have determined the crystal structure of the title compound to establish its solid-state conformation as part of a programme to study the relationship between conformation and biological activity in this series of fungicides. A preliminary description of this structure has already been published (Anderson, Branch, Loeffler, Mann, Nowell & Walker, 1984).

Experimental. Recrystallization from ethanol:water (2:1); D_m by flotation in hexane/ CCl_4 ; clear colourless

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crystal, approximate dimensions $0.40 \times 0.44 \times 0.18$ mm, mounted about crystallographic c axis; Stoe Stadi-2 two-circle diffractometer; Mo $K\alpha$ radiation; Lorentz, polarization but no absorption corrections applied; one standard reflection monitored for each layer collected, intensity variation $<3\%$; 3274 reflections up to $\theta = 27.5^\circ$; index ranges $h = -12$ to 12 , $k = 0$ to 16 , $l = 0$ to 15 ; 1364 with $I \geq 3.0\sigma(I)$ considered observed; structure solved by multiresolution direct methods; blocked full-matrix least-squares refinement of atomic coordinates and anisotropic temperature factors for all non-H atoms and isotropic temperature factors for all H atoms, minimizing $\sum w(F_o - F_c)^2$ with $w = 0.4220/[\sigma^2(F_o) + 0.0050(F_o)^2]$; H(O1) located by difference synthesis but positional parameters not refined, remaining H atoms included in positions calculated from geometry of molecule (C—H = 1.08 \AA); final $R = 0.047$, $wR = 0.069$; max. $(\Delta/\sigma) = 1.93$ [for U_{11} of C(5)], max Δ/σ for coordinates = 1.35 [for x of C(1)], mean $\Delta/\sigma = 0.35$; min. $\Delta\rho = -0.23$, max. $\Delta\rho = 0.17 \text{ e \AA}^{-3}$; no correction for secondary extinction; scattering factors from *International Tables for X-ray Crystallography* (1974); computer programs: *SHELX* (Sheldrick, 1976) and *PLUTO* (Motherwell & Clegg, 1978). Final refined values of $U = 0.069$ (13), 0.054 (9) and 0.057 (12) \AA^2 for H(O1), H(4) and H(5) respectively, common isotropic temperature factors applied to methyl, methylene, phenyl and triazolyl H atoms refined to $U = 0.084$ (5), 0.072 (9), 0.067 (6) and 0.106 (12) \AA^2 , respectively.

Discussion. The final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* bond distances and angles in Table 2 and the atomic numbering scheme in Fig. 1. The molecule adopts a conformation such that the carbon chain C(31)—C(4)—C(5)—C(6)—C(71) is extended and the atoms essentially coplanar (Fig. 1). Adoption of this 'W' conformation appears to minimize steric repulsion between the bulky *tert*-butyl and fluorophenyl groups. The triazolyl and fluorophenyl groups are inclined at an angle of 58.9 (10) $^\circ$ to each other and at angles of 79.3 (10) and 69.1 (10) $^\circ$ to the plane of the carbon chain, respectively. The triazolyl ring is orientated such that N(2) almost eclipses H(5), the torsion angle N(2)—N(1)—C(5)—H(5) being 1.7° . This orientation may be necessary to allow the close contact between N(3) and H(O1) of neighbouring molecules, which is revealed by inspection of intermolecular distances. The distance between these two atoms is 1.88 \AA , which is

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42490 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

considerably less than the sum of their respective van der Waals radii (1.5 and 1.15 \AA) and reflects the presence of strong hydrogen bonding. These intermolecular interactions link molecules of the $2R,3R$ and $2S,3S$ configurations alternately into chains (Fig. 2).

Table 1. Fractional positional parameters ($\times 10^4$) and equivalent isotropic thermal parameters with *e.s.d.*'s in parentheses

B_{eq} values are calculated by reference to Willis & Pryor (1975).

	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
F(72)	-2010 (3)	6836 (2)	1151 (3)	6.1 (1)
N(1)	2099 (3)	6465 (2)	468 (2)	3.0 (1)
N(2)	1651 (3)	5702 (3)	-274 (3)	4.2 (1)
N(3)	3354 (3)	6748 (3)	-730 (3)	4.4 (1)
O(1)	3694 (2)	7050 (2)	2513 (2)	3.6 (1)
C(1)	3104 (4)	7079 (3)	176 (3)	3.8 (1)
C(2)	2450 (4)	5907 (4)	-957 (3)	4.7 (2)
C(31)	3094 (4)	5134 (3)	2575 (3)	3.7 (1)
C(32)	1828 (4)	4335 (3)	2351 (4)	4.7 (1)
C(33)	3856 (5)	5029 (4)	3732 (4)	5.3 (2)
C(34)	4181 (4)	4859 (3)	1893 (4)	4.3 (2)
C(4)	2523 (3)	6306 (3)	2408 (3)	3.0 (1)
C(5)	1452 (3)	6545 (3)	1385 (3)	3.0 (1)
C(6)	776 (4)	7672 (3)	1438 (3)	3.7 (1)
C(71)	-377 (3)	7913 (3)	487 (3)	3.6 (1)
C(72)	-1741 (4)	7481 (3)	373 (3)	4.1 (2)
C(73)	-2846 (4)	7662 (4)	-470 (4)	5.1 (2)
C(74)	-2567 (5)	8313 (4)	-1264 (4)	6.0 (2)
C(75)	-1230 (5)	8769 (4)	-1193 (4)	5.4 (2)
C(76)	-147 (4)	8569 (3)	-315 (4)	4.7 (2)

Table 2. Bond lengths (\AA) and bond angles ($^\circ$) with *e.s.d.*'s in parentheses

N(1)—N(2)	1.355 (4)	O(1)—H(O1)	0.97
N(2)—C(2)	1.308 (6)	C(4)—C(5)	1.528 (4)
C(2)—N(3)	1.344 (5)	C(5)—C(6)	1.545 (5)
N(3)—C(1)	1.317 (6)	C(6)—C(71)	1.504 (5)
C(1)—N(1)	1.335 (5)	C(71)—C(72)	1.381 (5)
N(1)—C(5)	1.455 (5)	C(72)—F(72)	1.355 (5)
C(31)—C(32)	1.538 (5)	C(72)—C(73)	1.372 (6)
C(31)—C(33)	1.536 (6)	C(73)—C(74)	1.379 (8)
C(31)—C(34)	1.532 (6)	C(74)—C(75)	1.375 (7)
C(31)—C(4)	1.550 (5)	C(75)—C(76)	1.393 (6)
C(4)—O(1)	1.429 (4)	C(76)—C(71)	1.376 (6)
		O(1)...N(3)*	2.804 (5)
		H(O1)...N(3)	1.88
C(1)—N(1)—N(2)	109.4 (3)	O(1)—C(4)—C(5)	108.9 (3)
C(1)—N(1)—C(5)	129.9 (3)	C(4)—C(5)—N(1)	112.6 (2)
N(2)—N(1)—C(5)	120.6 (3)	C(4)—C(5)—C(6)	110.1 (3)
N(1)—N(2)—C(2)	102.0 (3)	N(1)—C(5)—C(6)	110.4 (3)
N(2)—C(2)—N(3)	115.8 (4)	C(5)—C(6)—C(71)	112.4 (3)
C(2)—N(3)—C(1)	102.3 (4)	C(6)—C(71)—C(72)	121.2 (4)
N(3)—C(1)—N(1)	110.4 (3)	C(6)—C(71)—C(76)	122.7 (3)
C(32)—C(31)—C(33)	108.9 (4)	C(72)—C(71)—C(76)	116.1 (3)
C(32)—C(31)—C(34)	110.0 (3)	C(71)—C(72)—F(72)	117.8 (3)
C(33)—C(31)—C(34)	108.1 (3)	C(71)—C(72)—C(73)	124.6 (4)
C(32)—C(31)—C(4)	109.8 (3)	F(72)—C(72)—C(73)	117.6 (4)
C(33)—C(31)—C(4)	107.3 (3)	C(72)—C(73)—C(74)	117.5 (4)
C(34)—C(31)—C(4)	112.6 (3)	C(73)—C(74)—C(75)	120.5 (4)
C(31)—C(4)—O(1)	110.3 (2)	C(74)—C(75)—C(76)	119.8 (5)
C(4)—O(1)—H(O1)	113	C(75)—C(76)—C(71)	121.5 (4)
C(31)—C(4)—C(5)	117.0 (3)	O(1)—H(O1)...N(3)*	158

* Intermolecular contacts; symmetry code: (i) $x, 1\frac{1}{2} - y, \frac{1}{2} + z$.

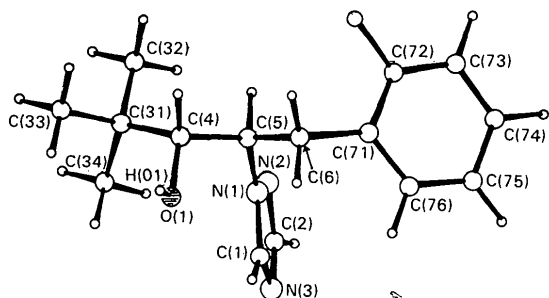


Fig. 1. The conformation of the molecule (*2R,3R* enantiomer depicted) showing the atom numbering.

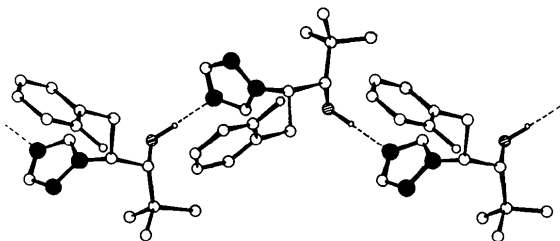


Fig. 2. Linking of molecules of *2R,3R* and *2S,3S* configurations by hydrogen bonding into chains running parallel to the *c* axis.

All four C–N and the N–N bond distances in the triazolyl ring are intermediate between the expected single- and double-bond lengths. The three atoms bonded to N(1) are coplanar with it and, taken together, these data indicate extensive delocalization in the heterocyclic ring. The exocyclic angles at N(1) show considerable asymmetry, C(5)–N(1)–C(1) being

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The Structure of Triphenyl[α -(phenylseleno)phenacylidene]phosphorane

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Abstract. C₃₂H₂₅OPSe, *M_r* = 535.48, monoclinic, *P2₁/c*, *a* = 10.943 (5), *b* = 13.576 (6), *c* = 17.494 (6) Å, β = 96.63 (3)°, *V* = 2581.6 Å³, *Z* = 4, *D_x* = 1.378 Mg m⁻³, *Mo K α* , λ = 0.71069 Å, μ = 1.521 mm⁻¹, *F*(000) = 1096, *T* = 296 K, final *R* = 0.053 for 3038 unique observed reflections. The

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significantly larger than C(5)–N(1)–N(2), which, as previously noted (Branch & Nowell, 1985), appears to be a characteristic of the triazolyl ring itself rather than the influence of any intra- or intermolecular interactions.

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molecule is essentially a phosphorus ylide with a phenacylidene and a phenylseleno substituent at the ylide carbon atom, C(1). The central P(1)=C(1)–C(2)=O group is nearly planar and *cis* relative to C(1)–C(2). Corresponding bond lengths are P–C(1) = 1.746 (4), C(1)–C(2) = 1.406 (6) and

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