References

AZARNIA, N., BERMAN, H. M. & ROSENSTEIN, R. D. (1972). Acta Cryst. B28, 2157–2167.

BARRAGAN, I., LÓPEZ-CASTRO, A. & MÁRQUEZ, R. (1977). Acta Cryst. B33, 2244–2248.

CONDE, A., LÓPEZ-CASTRO, A. & MÁRQUEZ, R. (1978). Rev. Iberoam. Crystallogr. Mineral. Metalogen. 1, 23–36.

CREMER, D. & POPLE, J. A. (1975). J. Am. Chem. Soc. 97, 1354–1358.

CRIADO, A., CONDE, A. & MÁRQUEZ, R. (1983). Acta Cryst. C39, 122–125.

FERNANDEZ-BOLAÑOS, J., FUENTES-MOTA, J. & FERNANDEZ-BOLAÑOS GUZMAN, J. M. (1983). An. Quím. 79, 345–349.

GALLOY, J. & WATSON, W. H. (1983). Acta Cryst. C39, 348–350.

International Tables for X-ray Crystallography (1962). Vol. III, pp. 202–207. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

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.

MAZHAR-UL-HAQUE, AHMED, J. & HORNE, W. (1983). Acta Cryst. C39, 383–385.

PÉREZ-GARRIDO, S., CONDE, A. & MÁRQUEZ, R. (1974). Acta Cryst. B30, 2348–2352.

STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY70 System. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Structure of (E)-3-(2-Methoxyphenyl)-1-phenyl-3-(1H-1,2,4-triazol-1-yl)-2-propen-1-one, $C_{18}H_{15}N_3O_2$

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Abstract. $M_r = 305.3$, $P2_12_12_1$, $a = 15.020$ (5), $b = 14.537$ (5), $c = 7.169$ (3) Å, $V = 1565.3$ Å^3, $Z = 4$, $D_m = 1.32$ (2), $D_x = 1.30$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 0.51$ cm^{-1}, $F(000) = 640$, room temperature, final $R = 0.064$ for 1092 reflections classed as observed. The α,β-unsaturated carbonyl function is nearly planar and adopts a cis conformation. The methoxyphenyl ring is twisted by 59 (1)° out of the plane containing the carbon–carbon double bond while the triazolyl ring, which shows significant delocalization, lies almost in that plane. The unsubstituted phenyl ring is almost coplanar with the carbonyl group. The exocyclic angles of the triazolyl ring are very asymmetric with C–N–C [129.9 (6)°] significantly larger than C–N–N [122.4 (6)°].

Introduction. The title compound is one of a series of γ-ketotriazoles (Balasubramanyan & Lewis, 1976) that are good pre-emergence herbicides. We have determined its crystal structure to establish its conformation in the solid state as part of a programme to study the relationship between conformation and biological activity in this class of herbicides. We have previously reported the crystal structure of an analogue of the title compound, (E)-4,4-dimethyl-1-(1-naphthyl)-1-(1H-1,2,4-triazol-1-yl)pent-1-en-3-one (Branch & Nowell, 1985).

Experimental. Recrystallization from methanol:petrol 40–60 (1:2); D_m by flotation in carbon tetrachloride:petrol 60–80; crystal, yellow hexagonal prism, approximate dimensions 0.50 × 0.40 × 0.40 mm, mounted about crystallographic c axis; Stoe Stadi-2 two-circle diffractometer; Lorentz, polarization but no absorption corrections applied; one standard reflection monitored for each layer collected, intensity variation < 3%; 2343 reflections up to $\sin\theta/\lambda = 0.70$ Å^{-1}, index ranges $h = 0$ to 20, $k = 0$ to 20, $l = 0$ to 9; 2331 independent reflections of which 1092 with $I > 3\sigma(I)$ considered observed; structure solved by multisolution direct methods; blocked-matrix least-squares refinement, minimizing $\sum w(|F_o| - |F_c|)^2$ with $w = 3.5939/[\sigma^2(F_o) + 0.0007(F_o)^2]$, of atomic coordinates and anisotropic temperature factors for all non-H atoms, isotropic temperature factors for all H atoms; H(5) located by difference synthesis but positional parameters not refined, remaining H atoms included in positions calculated from geometry of molecule (C–H = 1.08 Å); final $R = 0.0640$, $wR = 0.0880$; max. $(\Delta/\sigma) = 1.97$; min. $\Delta\rho = -0.24$, max. $\Delta\rho = 0.31$ e Å^{-3}; no correction for secondary extinction; scattering factors calculated from analytical approximation (International Tables for X-ray Crystallography, 1974); computer programs SHELX (Sheldrick, 1976) and PLUTO (Motherwell & Clegg, 1978). Final value of $U = 0.093$ (28) Å^2 refined for H(5) while common isotropic temperature factors applied to methyl, phenyl

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$C_{18}H_{15}N_3O_2$

and triazolyl H atoms refined to $U = 0.119$ (19), 0.059 (14) and 0.073 (20) Å^2, respectively.

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

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

	x	y	z	$B_{eq}(\text{Å}^2)$
N(1)	656 (3)	7920 (3)	2532 (10)	4·1 (3)
N(2)	−214 (4)	7666 (4)	2275 (11)	5·3 (3)
N(3)	−30 (5)	8240 (5)	5134 (13)	6·6 (4)
O(1)	2221 (4)	7675 (4)	−2482 (10)	6·4 (3)
O(2)	1189 (3)	9684 (3)	1413 (9)	5·0 (3)
C(1)	729 (5)	8245 (6)	4200 (14)	5·4 (4)
C(2)	−567 (6)	7873 (6)	3854 (17)	5·9 (5)
C(21)	1032 (7)	10632 (5)	1810 (15)	6·0 (4)
C(31)	2108 (4)	8403 (4)	1428 (10)	3·7 (3)
C(32)	2936 (5)	7998 (6)	1563 (13)	5·1 (4)
C(33)	3688 (5)	8534 (7)	1920 (14)	6·1 (5)
C(34)	3587 (5)	9470 (8)	2106 (14)	6·5 (5)
C(35)	2753 (5)	9903 (6)	1982 (13)	5·4 (4)
C(36)	2033 (4)	9354 (5)	1607 (11)	4·1 (3)
C(4)	1302 (4)	7822 (4)	1131 (11)	3·9 (3)
C(5)	1137 (5)	7225 (5)	−250 (14)	4·9 (4)
C(6)	1684 (4)	7100 (5)	−1938 (13)	4·8 (4)
C(71)	1538 (4)	6230 (5)	−3086 (11)	4·2 (3)
C(72)	1890 (5)	6212 (5)	−4844 (14)	5·3 (4)
C(73)	1795 (5)	5443 (6)	−5954 (15)	6·3 (5)
C(74)	1323 (6)	4685 (5)	−5296 (16)	6·5 (5)
C(75)	996 (5)	4711 (5)	−3476 (16)	5·7 (4)
C(76)	1098 (5)	5483 (5)	−2405 (14)	5·2 (4)

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

N(1)–N(2)	1·371 (7)	C(36)–O(2)	1·363 (8)
N(2)–C(2)	1·286 (14)	O(2)–C(21)	1·426 (9)
C(2)–N(3)	1·334 (13)	C(4)–C(5)	1·340 (11)
N(3)–C(1)	1·321 (12)	C(5)–H(5)	1·06
C(1)–N(1)	1·290 (12)	C(5)–C(6)	1·474 (13)
N(1)–C(4)	1·405 (9)	C(6)–O(1)	1·225 (9)
C(4)–C(31)	1·491 (9)	C(6)–C(71)	1·525 (10)
C(31)–C(32)	1·378 (10)	C(71)–C(72)	1·367 (12)
C(32)–C(33)	1·397 (12)	C(72)–C(73)	1·379 (13)
C(33)–C(34)	1·375 (16)	C(73)–C(74)	1·393 (12)
C(34)–C(35)	1·404 (12)	C(74)–C(75)	1·394 (16)
C(35)–C(36)	1·371 (10)	C(75)–C(76)	1·369 (12)
C(36)–C(31)	1·393 (9)	C(76)–C(71)	1·362 (10)
C(1)–N(1)–N(2)	107·7 (6)	C(35)–C(36)–O(2)	123·3 (6)
C(1)–N(1)–C(4)	129·9 (6)	C(31)–C(36)–O(2)	114·6 (6)
N(2)–N(1)–C(4)	122·4 (6)	C(36)–O(2)–C(21)	118·2 (6)
N(1)–N(2)–C(2)	102·2 (7)	C(4)–C(5)–H(5)	120
N(2)–C(2)–N(3)	116·7 (8)	C(4)–C(5)–C(6)	125·7 (7)
C(2)–N(3)–C(1)	100·1 (9)	H(5)–C(5)–C(6)	114
N(3)–C(1)–N(1)	113·3 (7)	C(5)–C(6)–O(1)	123·0 (7)
N(1)–C(4)–C(31)	113·7 (6)	C(5)–C(6)–C(71)	117·7 (6)
N(1)–C(4)–C(5)	117·8 (6)	O(1)–C(6)–C(71)	119·2 (8)
C(31)–C(4)–C(5)	128·5 (7)	C(6)–C(71)–C(72)	117·3 (6)
C(4)–C(31)–C(32)	120·0 (6)	C(6)–C(71)–C(76)	122·5 (7)
C(4)–C(31)–C(36)	120·6 (6)	C(72)–C(71)–C(76)	120·2 (7)
C(32)–C(31)–C(36)	119·4 (6)	C(71)–C(72)–C(73)	120·5 (7)
C(31)–C(32)–C(33)	120·3 (8)	C(72)–C(73)–C(74)	119·9 (9)
C(32)–C(33)–C(34)	118·7 (7)	C(73)–C(74)–C(75)	118·4 (8)
C(33)–C(34)–C(35)	122·4 (8)	C(74)–C(75)–C(76)	120·5 (8)
C(34)–C(35)–C(36)	117·1 (8)	C(75)–C(76)–C(71)	120·5 (9)
C(35)–C(36)–C(31)	122·1 (6)		

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 carbonyl group and unsaturated bond C(4)=C(5) are *cis* orientated and nearly coplanar. The dihedral angle between the two groups [as defined by the planes containing atoms C(5), C(6), O(1), C(71) and N(1), C(31), C(4), C(5), C(6)] is 23·4 (10)°. This arrangement minimizes steric interactions between the methoxyphenyl ring and the phenyl ring at C(6) in comparison with the *trans* conformation. The C(71)–C(76) ring is inclined to the plane containing atoms C(5), C(6), O(1) and C(71) at an angle of 14·1 (10)° while the triazolyl ring, which is effectively planar, is orientated such that N(2) is directed towards H(5). This allows the heterocyclic ring to lie almost in the plane containing atoms N(1), C(31), C(4), C(5) and C(6), the dihedral angle between the latter plane and the triazolyl ring being 18·2 (10)°. This conformation could facilitate conjugation between the phenyl ring at C(6), the α,β-unsaturated ketone function and the heterocyclic ring; however, this is not reflected in the bond distances. Although N(1)–C(4) is significantly shorter than the expected single-bond distance, C(5)–C(6) and C(6)–C(71) are not, nor is C(4)–C(5) longer than the expected double-bond distance. The methoxyphenyl ring at C(4) is inclined at an angle of 59·1 (10)° to the plane containing atoms N(1), C(31), C(4), C(5), C(6) and at 65·5 (10)° to the triazolyl ring. This
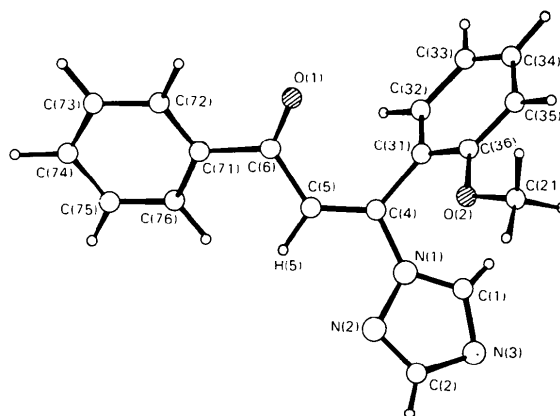
Fig. 1. The conformation of the molecule showing the atom numbering: a view perpendicular to the plane containing atoms N(1), C(31), C(4), C(5) and C(6).

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

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Abstract. $M_r = 258 \cdot 38$, orthorhombic, $P2_1 2_1 2_1$, $a = 7 \cdot 458$ (6), $b = 13 \cdot 436$ (6), $c = 14 \cdot 736$ (5) Å, $V = 1476 \cdot 6$ (23) Å³, $Z = 4$, $D_x = 1 \cdot 16$ Mg m⁻³, Mo Kα, $\lambda = 0 \cdot 71069$ Å, $\mu = 0 \cdot 090$ mm⁻¹, $F(000) = 568$, $T = 293$ K, $R = 0 \cdot 036$ 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)
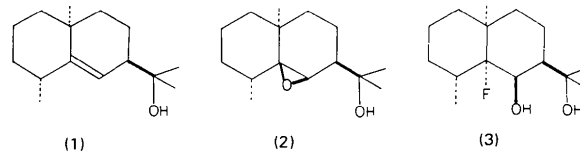
Experimental. Crystal: rectangular prism 0·2 × 0·2 × 0·4 mm, CAD-4 diffractometer, variation in 3 intensity standards <3%, unit-cell dimensions from 25 reflexions, 2658 reflexions measured, $0 \le h \le 8$, $0 \le k \le 15$, $-17 \le l \le 17$, $\theta < 25°$, 2084 unique (Friedel pairs not merged), $R_{int} = 0 \cdot 038$, 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