Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
8184 measured reflections
3048 independent reflections
2243 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\rm max} = 28.27°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.135$
$S = 1.119$
3048 reflections
212 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.3154P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.014 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C2	1.224 (2)	C5—C6	1.536 (2)
O2—C6	1.212 (2)	C5—C14	1.558 (2)
C1—C13	1.337 (2)	C6—C7	1.482 (2)
C1—C2	1.465 (3)	C7—C12	1.396 (2)
C2—C3	1.515 (3)	C12—C13	1.463 (2)
C3—C4	1.538 (3)	C14—C15	1.463 (3)
C4—C5	1.530 (2)	C15—C16	1.178 (3)
C5—C13	1.516 (2)		
C13—C1—C2—C3	11.6 (3)	C4—C5—C13—C1	−35.5 (2)
C1—C2—C3—C4	10.7 (3)	C13—C5—C14—C15	50.3 (2)
C2—C3—C4—C5	−43.4 (3)	C4—C5—C14—C15	172.57 (16)
C3—C4—C5—C13	53.7 (2)	C6—C5—C14—C15	−60.9 (2)
C2—C1—C13—C5	1.6 (3)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16···O1$^{\rm i}$	0.94 (3)	2.37 (3)	3.252 (3)	156 (2)
C14—H14B···O2$^{\rm ii}$	0.99 (2)	2.48 (2)	3.423 (2)	160 (2)
C11—H11···O1$^{\rm iii}$	1.01 (2)	2.57 (2)	3.554 (2)	162 (2)

Symmetry codes: (i) $2 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $1 + x, y, z$; (iii) $2 - x, 1 - y, 1 - z$.

All H atoms were located from a difference map and were refined isotropically.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1252). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1136–1138

4-(4-Methylphenyl)-3-phenyl-4*H*-1,2,4-triazole

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Abstract

In the title compound, $C_{15}H_{13}N_3$, the triazole ring is planar and forms dihedral angles of 35.05 (5) and 65.44 (5)°, respectively, with the attached phenyl and methylphenyl rings. The crystal structure is stabilized by a number of C—H···π interactions and weak C—H···N short contacts.

Comment

Generally, 1,2,4-triazole derivatives are found to be associated with diverse pharmacological activity. Recently, some new triazole derivatives have been synthesized as possible anticonvulsants, antidepressants, tranquilizers and plant-growth regulators (Bradbury & Rivett,

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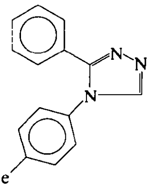
1991; Hirota *et al.*, 1991; Walser *et al.*, 1991). Extensive studies have also been carried out on the substituted 1,2,4-triazole ligands (Cornelissen *et al.*, 1992; Gupta & Bhargava, 1978; Kunkeler *et al.*, 1996). It is of interest that some iron(II) complexes containing substituted 1,2,4-triazole ligands are spin-crossover materials which could be used as molecular-based memory devices, displays and optical switches (Garcia *et al.*, 1997; Kahn & Martinez, 1998). The X-ray structure determination of the title compound, (I), a triazole derivative, was carried out in order to elucidate the molecular conformation.



(I)

The bond lengths and angles in the structure of (I) are comparable with those observed in a related structure (Rogers *et al.*, 1990). The N1—C2 [1.315 (2) Å] and N2—C1 [1.297 (2) Å] bonds show double-bond character. The other N—C bonds have an intermediate character, indicating delocalization of the electron cloud. The triazole ring is planar, with the phenyl and methylphenyl substituents twisted with respect to the plane by 35.05 (5) and 65.44 (5)°, respectively. The dihedral angle between the planes of the phenyl and methylphenyl rings is 66.71 (4)°. The screw-related molecules have C11—H11···N2[i] short contacts along the *b* direction, whereas the glide-related molecules have C15—H15*F*···N1[ii] short contacts along [101] [symmetry codes: (i) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (ii) $x-\frac{1}{2}, \frac{1}{2}-y, z+\frac{1}{2}$]. The crystal structure is further stabilized by a number of C—H···π interactions involving phenyl rings *B* and *C* (Table 2).

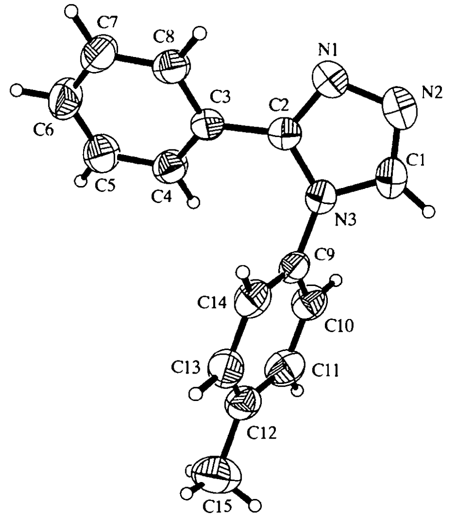


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Only one orientation of the disordered methyl group is shown.

Experimental

A solution of 4-(4-methylphenyl)-5-phenyl-1,2,4-triazole-3-thione (0.3 g, 0.001 mol) in absolute methanol (150 ml) was flushed with nitrogen for 30 min and irradiated for 1 h at 254 nm using a thin film reactor. After completion of the reaction (checked by TLC), the solvent was removed under reduced pressure and the mixture was chromatographed over a column of silica gel. Elution with petroleum ether–ethyl acetate (6:4) gave the title compound, (I) (yield 0.13 g, 50%; m.p. 405–407 K). Single crystals were grown by slow evaporation of a solution of (I) in a methanol–chloroform (1:1) solvent system.

Crystal data

$C_{15}H_{13}N_3$
M_r = 235.28
Monoclinic
$P2_1/n$
a = 9.2544 (1) Å
b = 14.5543 (4) Å
c = 9.6994 (2) Å
β = 105.435 (1)°
V = 1259.31 (5) Å^3
Z = 4
D_x = 1.241 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 4055 reflections
θ = 2.59–28.27°
μ = 0.076 mm^{-1}
T = 293 (2) K
Parallelepiped
0.44 × 0.26 × 0.24 mm
Colourless

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
8485 measured reflections
3109 independent reflections
2140 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.029
$\theta_{\rm max}$ = 28.27°
$h = -12 \rightarrow 11$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 12$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.120
S = 1.043
3109 reflections
210 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0540P)^2 + 0.1057P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.15 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.024 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N1—C2	1.315 (2)	N3—C1	1.360 (2)
N1—N2	1.391 (2)	N3—C2	1.371 (2)
N2—C1	1.297 (2)	N3—C9	1.437 (2)
N1—C2—C3—C8	−34.4 (2)	C1—N3—C9—C10	−67.3 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11···N2^{i}	0.96 (2)	2.60 (2)	3.519 (2)	160 (1)
C15—H15*F*···N1^{ii}	0.96	2.60	3.544 (3)	170
C6—H6···π(Ring *C*)^{iii}	0.98 (2)	2.76 (2)	3.591 (3)	143 (1)
C10—H10···π(Ring *B*)^{iv}	0.98 (2)	2.73 (2)	3.552 (3)	143 (1)
C13—H13···π(Ring *B*)^{ii}	0.96 (2)	2.83 (2)	3.645 (3)	143 (1)
C15—H15*C*···π(Ring *C*)^{v}	0.96	3.15 (2)	4.056 (3)	158

Symmetry codes: (i) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$; (iii) $1 + x, y, z$; (iv) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (v) $1 - x, 1 - y, 2 - z$.

All H atoms were located from a difference map. The methyl group was found to be disordered, with two equally occupied orientations rotated from each other by 60°, and it was treated as an idealized disordered methyl group. The remaining H atoms were refined isotropically.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

The authors would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. KC thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1079). Services for accessing these data are described at the back of the journal.

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Methyl (5*SR*,6*SR*)-1,3-dichloro-8-diethylamino-5,6-dihydro-5-hydroxy-7-nitro-6-phenylisoquinoline-5-carboxylate

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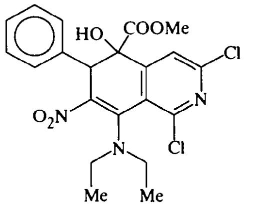
Abstract

In the title compound, $C_{21}H_{21}Cl_2N_3O_5$, the fused cyclohexadiene ring adopts a skew-boat conformation, with the methoxycarbonyl and phenyl groups attached in axial positions and the nitro and diethylamino groups attached equatorially. The mean plane through the cyclohexadiene ring forms dihedral angles of 23.30 (7) and 87.72 (8)° with the pyridine and phenyl rings, respectively. In the crystal, the inversion-related molecules exist as O—H···O hydrogen-bonded dimers.

Comment

The development of the tandem reaction process (Bunce, 1995), also called the domino reaction (Tietze, 1996), is a rapidly growing area of synthetic organic chemistry which allows the interlinking of several transformations in a single synthetic step. In conjunction with our work in this area, we have now uncovered a tandem Hamaguchi–Ibata and Diels–Alder reaction involving a crystalline diazoacetic ester (Sarkar *et al.*, 1999), and β-nitrostyrene in the presence of 1 mol% $Rh_2(OAc)_4$ in benzene under reflux conditions to give a substituted isoquinoline in high yield. The X-ray structure determination of the title compound, (I), was undertaken in order to elucidate the molecular conformation.



(I)

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