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3,5-Diphenyl-4-(3,4,5-trimethoxybenzylideneamino)-4*H*-1,2,4-triazole

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.097
Data-to-parameter ratio = 11.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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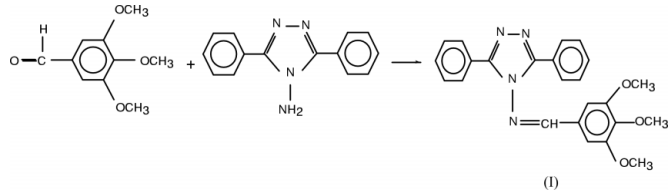
The title compound, $C_{24}H_{22}N_4O_3$, prepared by the reaction of 3,4,5-trimethoxybenzaldehyde and 3,5-diphenyl-4-amino-4*H*-1,2,4-triazole, crystallizes in a monoclinic space group, with one molecule in the asymmetric unit. The crystal structure is stabilized mainly by van der Waals interactions, although a short intermolecular C···O contact of 3.156 (3) Å is also found.

Comment

Based on the promising pharmacological activities found in various 1,2,4-triazoles and 4,5-dihydro-1*H*-1,2,4-triazol-5-ones, a study of the syntheses and biological activities of some 4-arylidenamino-4,5-dihydro-1*H*-1,2,4-triazol-5-ones has been undertaken recently (Kahveci & Ikizler, 2000*a*,*b*).



(I)

We report here the crystal structure of the title compound, (I), which contains one 1,2,4-triazole ring and three benzene rings (Fig. 1). The bond lengths are normal (Table 1) and comparable with reported values (Çoruh *et al.*, 2003; Ocak, Çoruh *et al.*, 2003; Ocak, Kahveci *et al.*, 2003; McCarrick *et al.*, 1999, Prabakaran *et al.*, 2001). The dihedral angles between the planes of rings C1–C6 (*A*), C7/C8/N1–N3 (*B*), C9–C14 (*C*) and C16–C21 (*D*) are *A*/*B* = 40.81 (7)°, *A*/*C* = 15.50 (7)°, *A*/*D* = 75.25 (7)°, *B*/*C* = 46.72 (8)°, *B*/*D* = 53.74 (7)° and *C*/*D* = 66.51 (9)°. The triazole ring *B* is planar, with a maximum deviation of 0.0039 (2) Å for atom C8. The crystal structure is stabilized mainly by van der Waals interactions, although there is also a short intermolecular C—H···O contact (Table 2).

Experimental

3,4,5-Trimethoxybenzaldehyde (0.005 mol) was added to a solution of 3,5-diphenyl-4-amino-4*H*-1,2,4-triazole (0.005 mol) in glacial acetic acid (20 ml) and the mixture was refluxed for 4 h. After cooling, the mixture was poured into a beaker containing ice water (100 ml). The resulting precipitate was filtered off. After drying *in vacuo*, the product was recrystallized from ethanol to give 3,5-diphenyl-4-(3,4,5-trimethoxybenzylidenamino)-4*H*-1,2,4-triazole (m.p. 458–459 K, yield 91.75%). IR: 1613, 1576 (CN), 696, 763, 854 (substituted benzenoid ring). ^{1}H NMR (DMSO-d_6): 3.53 (*s*, 3H, OCH_3), 3.68 (*s*, 6H, $2OCH_3$), 7.20 (*s*, 2H, aromatic H), 7.52 (*m*, 6H, aromatic H), 7.88 (*m*, 4H, aromatic H), 8.58 (*s*, 1H, CH). ^{13}C NMR (DMSO-d_6): 55.53 (2C, $2OCH_3$), 60.10 (1C, $2OCH_3$), Aryl C atoms: 152.83 (2C), 141.48,

129.32 (2C), 128.42 (4C), 127.70 (4C), 126.14, 125.87 (2C), 106.15 (2C), triazole C-3 and C-5: 149.64 (2C), 170.35 (1C, CH). UV: I_{max} 258 (*e* 11.1), I_{max} 205 (*e* 23.5). Analysis calculated for $C_{24}H_{22}N_4O_3$: C 69.55, H 5.35, N 13.52%; found: C 69.83, H 5.17, N 13.64%.

Crystal data

$C_{24}H_{22}N_4O_3$
M_r = 414.46
Monoclinic, $P2_1/c$
a = 12.3359 (10) Å
b = 12.5288 (12) Å
c = 13.9855 (11) Å
β = 96.272 (6)°
V = 2148.6 (3) Å^3
Z = 4
D_x = 1.281 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8342 reflections
θ = 1.6–30.4°
μ = 0.09 mm^{-1}
T = 293 (2) K
Prism, white
0.50 × 0.30 × 0.22 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
T_{min} = 0.985, T_{max} = 0.997
3749 measured reflections
3749 independent reflections
1919 reflections with $I > 2\sigma(I)$
R_{int} = 0.134
θ_{max} = 25.0°
$h = -14 \rightarrow 14$
$k = -18 \rightarrow 17$
$l = -20 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.097
S = 0.74
3749 reflections
336 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0074 (7)

Table 1
Selected geometric parameters (Å).

C4—C7	1.465 (3)	N3—N2	1.386 (3)
O1—C20	1.363 (2)	C18—O3	1.361 (2)
O1—C24	1.422 (3)	C15—N4	1.267 (3)
O2—C19	1.367 (2)	N1—C8	1.371 (2)
O2—C23	1.401 (3)	N1—N4	1.407 (2)
C7—N3	1.316 (2)	O3—C22	1.434 (3)
C7—N1	1.365 (2)	N2—C8	1.313 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C15—H11···O2^{i}	0.96 (2)	2.58 (2)	3.156 (3)	119.2 (15)

Symmetry code: (i) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$.

H atoms were positioned geometrically and refined using a riding model, fixing methyl group C—H distances at 0.96 Å.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1997) and *PARST* (Nardelli, 1995).

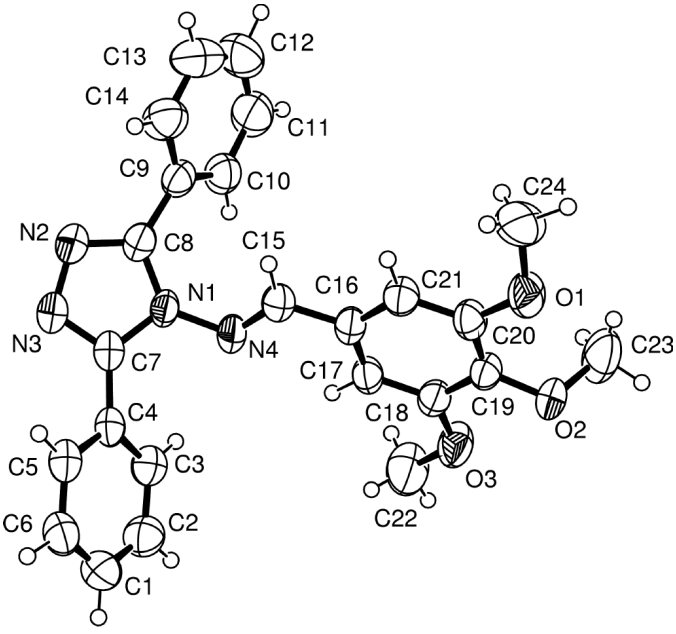


Figure 1
An *ORTEP*III (Farrugia, 1997) view of the title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

References

Burnett, M. N. & Johnson, C. K. (1996). *ORTEP*III. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Çoruh, U., Kahveci, B., Şaşmaz, S., Ağar, E., Kim, Y. & Erdönmez, A. (2003). *Acta Cryst.* C**59**, o176–o178.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Kahveci, B. & Ikizler, A. A. (2000*a*). *Acta Pol. Pharm. Drug Res.* **57**, 119.
Kahveci, B. & Ikizler, A. A. (2000*b*). *Turk. J. Chem*, **24**, 343.
McCarrick, R. M., Squattrito, P. J., Singh, R. N., Handa, R. N. & Dubey S. N. (1999). *Acta Cryst.* C**55**, 2111–2114.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Ocak, N., Çoruh,U., Kahveci, B., Şaşmaz, S., Ağar, E., Vázquez-López, E. M. & Erdönmez, A. (2003). *Acta Cryst.* E**59**, o750–o752.
Ocak, N., Kahveci, B., Şaşmaz, S., Ağar, E., & Erdönmez, A. (2003). *Acta Cryst.* E**59**, o1137–o1138.
Prabakaran, P., Robert, J. J., Muthiah, P. T., Bocelli, G. & Righi, L. (2001). *Acta Cryst.* C**57**, 459–461.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Stoe & Cie (2002). *X-AREA* (Version 1.118) and *X-RED*32 (Version 1.04). Stoe & Cie, Darmstadt, Germany.