Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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Received 14 April 2005
Accepted 5 May 2005
Online 14 May 2005

The title compound, $C_{24}H_{24}N_4O_3$, contains three aromatic rings and a triazole ring which are not coplanar. In the crystal structure the molecules are linked by C—H···N, C—H···O, N—H···N, C—H···π and π–π interactions.

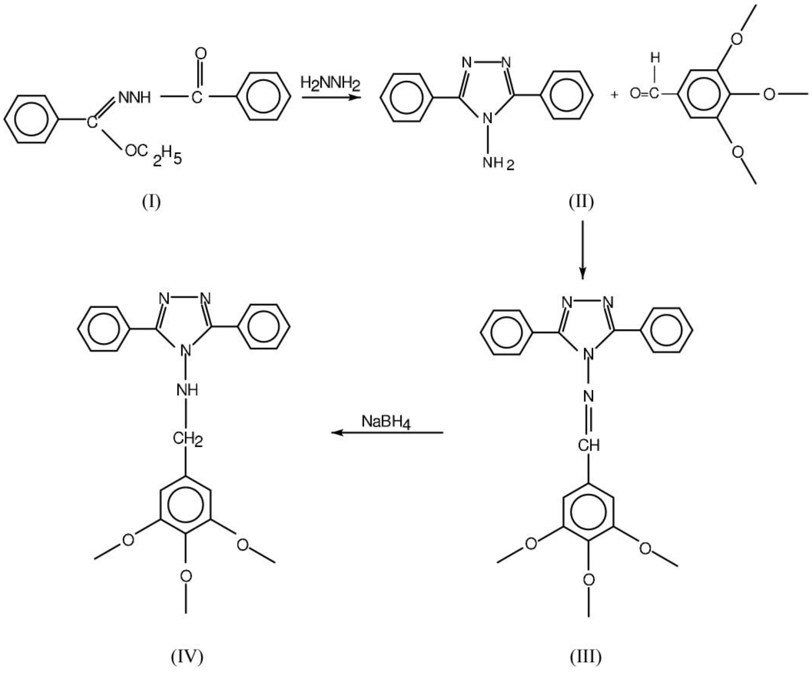
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.049
wR factor = 0.151
Data-to-parameter ratio = 21.1

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

Comment

Recently, the synthesis and biological activities of some 4-arylidenamino-4,5-dihydro-1*H*-1,2,4-triazol-5-ones have been reported (Kröger *et al.*, 1965; Kahveci & İkizler, 2000). Due to their structural similarity, 4-arylidenamino-4*H*-1,2,4-triazoles may be important as potential biologically compounds (Grammaticakis & Champetier, 1970). For this reason, the synthesis of the title compound, (IV), was carried out and its crystal structure is reported here.



In the title compound (Fig. 1), all the six-membered aromatic rings and the triazole ring are planar. The dihedral angles between these aromatic rings [C11–C16 (ring *A*), C21–C26 (ring *B*) and C31–C36 (ring *C*)] are 23.67 (5) (between rings *A*/*B*), 36.95 (5) (*A*/*C*) and 17.93 (5)° (*B*/*C*). The dihedral angles they form with the triazole ring are 40.13 (5), 29.45 (6) and 43.76 (4)°, respectively. The molecular conformation as well as bond lengths and angles are similar to those observed in the related derivative 3-phenyl-5-*p*-tolyl-4-(3,4,5-trimethoxybenzylamino)-4*H*-1,2,4-triazole (Isik *et al.*, 2003).

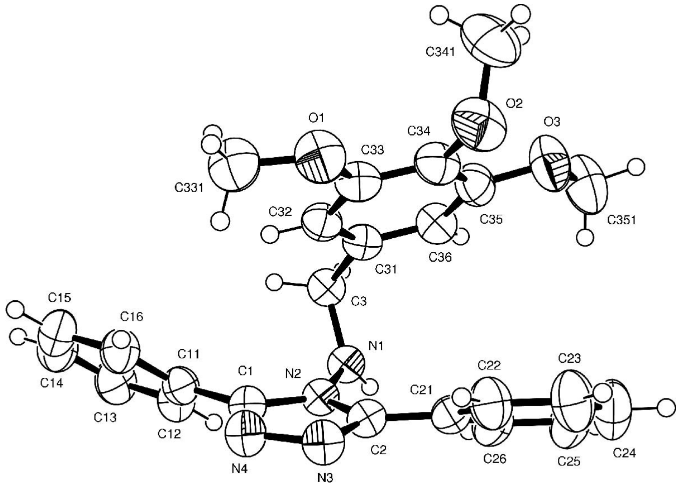


Figure 1
An *ORTEPIII* drawing (Burnett & Johnson, 1996) of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

The significant deviation from the ideal value observed for the bond angles O1—C33—C32 [123.57 (14)°], O1—C33—C34 [116.53 (12)°], O3—C35—C36 [124.21 (14)°] and O3—C35—C36 [115.95 (13)°] could be ascribed to the electronic repulsions occurring between atoms H33*A*/H32 and H35*B*/H36. The crystal packing is determined by the combination of C—H···O, C—H···N, N—H···N weak intra- and intermolecular hydrogen bonds, and C—H···π (Table 2) and π–π interactions. Weak intra- and intermolecular C—H···π interactions are present involving atoms C32 and C331 as donors and the triazole ring and benzene ring C. In addition to these interactions, the crystal structure is stabilized by intermolecular π–π stacking interactions occurring between the triazole ring and rings *A* and *B* of centrosymmetrically related molecules [$Cg1\cdots Cg1^{\rm v}$ = 3.356 (1) Å and $Cg2\cdots Cg3^{\rm v}$ = 3.991 (1) Å; symmetry code: (v) $2 - x, -y, -z$; *Cg*1, *Cg*2 and *Cg*3 are the centroids of the triazole ring and phenyl rings *A* and *B*, respectively].

Experimental

3,5-Diphenyl-4-(3,4,5-trimethoxybenzylidenamino)-4*H*-1,2,4-triazole, (III) (0.005 mol), was dissolved in dried methanol (50 ml) and $NaBH_4$ (0.005 mol) was added in small portions. The mixture was refluxed for 20 min and then allowed to cool. After evaporation at 298–303 K under reduced pressure, the solid residue was washed with cold water. After drying *in vacuo*, the solid product was recrystallized from ethyl acetate to afford the desired compound (IV) (m.p. 428–429 K; yield 97.12%). IR: 3272 (NH), 1596 (CN), 698, 766, 819 (substituted benzenoid ring), ^{1}H NMR (DMSO-d$_6$): δ 3.60 (*s*, 6H, $2OCH_3$), 3.78 (*s*, 3H, OCH_3), 3.65 (*d*, 2H, CH_2), 5.65 (*t*, 1H, NH), 5.95 (*s*, 2H, aromatic H) 7.50 (*m*, 6H, aromatic H), 7.87 (*m*, 4H, aromatic H). ^{13}C NMR (DMSO-*d*$_6$): δ 55.79 (2C, $2OCH_3$), 60.68 (1C, OCH_3), 56.36 (2C, CH_2); Aryl C atoms: 152.99 (2C), 137.65, 130.06 (2C), 129.54, 128.79 (4C), 128.05 (4C), 126.84 (2C), 105.77 (2C), Triazole C-3 and C-5: 153.98 (2 C). UV: λ_{max} 259 (e 21.7), λ_{max} 213 (e 28.3). Analysis calculated for $C_{24}H_{24}N_4O_3$: C 69.21, H 5.81, N 13.45%; found: C 69.55, H 5.72, N 13.11%.

Crystal data

$C_{24}H_{24}N_4O_3$
M_r = 416.47
Monoclinic, $P2_1/n$
a = 14.4453 (12) Å
b = 6.3267 (5) Å
c = 24.4965 (19) Å
β = 104.517 (6)°
V = 2167.3 (3) Å^3
Z = 4

D_x = 1.276 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 15455 reflections
θ = 1.5–29.5°
μ = 0.09 mm^{-1}
T = 293 (2) K
Prism, colourless
0.58 × 0.49 × 0.34 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: by integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.931, T_{max} = 0.973
19928 measured reflections

5940 independent reflections
3823 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 29.4°
$h = -19 \rightarrow 19$
$k = -8 \rightarrow 8$
$l = -33 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.151
S = 1.01
5940 reflections
281 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0855P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.034 (3)

Table 1
Selected geometric parameters (Å, °).

C341—O2	1.416 (3)	N4—N3	1.3823 (17)
C331—O1	1.413 (2)	C3—N1	1.4673 (16)
O1—C33	1.3610 (17)	C1—N2	1.3711 (16)
O2—C34	1.3852 (17)	N3—C2	1.3161 (16)
C35—O3	1.3666 (19)	N2—C2	1.3677 (15)
O3—C351	1.417 (2)	N2—N1	1.4062 (13)
N4—C1	1.3138 (16)		
C33—O1—C331	117.68 (12)	O3—C35—C36	124.21 (14)
C34—O2—C341	114.09 (13)	O3—C35—C34	115.95 (13)
O1—C33—C34	116.53 (12)	C35—O3—C351	117.15 (14)
O1—C33—C32	123.57 (14)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C26—H26···N1	0.93	2.57	3.0572 (19)	113
C341—H34*C*···O3	0.96	2.56	3.085 (3)	115
N1—H1*A*···N3$^{\rm i}$	0.86	2.51	3.0226 (14)	119
C331—H33*A*···O1$^{\rm ii}$	0.96	2.42	3.197 (2)	138
C32—H32···*Cg*1	0.93	3.25	3.4488 (15)	94
C331—H33*C*···*Cg*4$^{\rm iii}$	0.96	2.86	3.670 (2)	143

Symmetry codes: (i) $x, 1 + y, z$; (ii) $1 - x, -1 - y, -z$; (iii) $1 - x, -y, -z$. *Cg*1 and *Cg*4 are the centroids of the triazole and C31–C36 benzene rings, respectively

The H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C—H distances at 0.93 Å, the methylene C—H distances at 0.97 Å, the methyl group C—H distances at 0.96 Å and the N—H distance at 0.86 Å, with U_{iso}(H) = 1.2U_{eq}(parent atom) or 1.5U_{eq}(methyl group).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular

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graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Grammaticakis, M. P. & Champetier, M. G. (1970). *C. R. Acad. Sci. Paris*, **271**, 940.

Isik, S., Olcay, B. C., Ağar, A. & Şaşmaz, S. (2003). *Acta Cryst.* E**59**, o1535–o1537.

Kahveci, B. & İkizler, A. A. (2000). *Acta Pol Pharm. Drug Res.* **57**, 119–122.

Kröger, C. F., Hummel, L., Mutscher, M. & Beyer, H. (1965). *Chem. Ber.* **98**, 3025.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.

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