

3-Phenyl-5-*p*-tolyl-4-(3,4,5-trimethoxybenzylamino)-4*H*-1,2,4-triazole

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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$

Some non-H atoms missing

R factor = 0.070

wR factor = 0.224

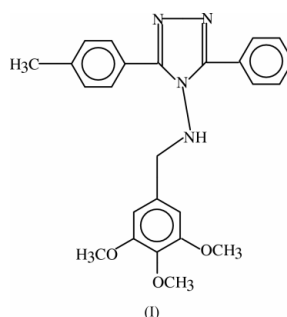
Data-to-parameter ratio = 21.7

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

In the title molecule, $\text{C}_{25}\text{H}_{25}\text{N}_4\text{O}_3$, the dihedral angles between the phenyl, *p*-tolyl and trimethoxybenzylamino rings and the triazole ring are $34.3(1)$, $52.9(1)$ and $59.5(1)^\circ$. The crystal structure is stabilized by $\text{N}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{N}$ interactions.

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 anti-convulsants, antidepressants, antifungal agents and antibacterial agents (Bradbury & Rivett, 1991; Hirota *et al.*, 1991). In view of this, we present here the crystal structure of the title compound, (I), and a conformational analysis of the molecule using X-ray structure determination methods.



The $\text{N}2=\text{C}2$ [$1.295(3)\text{ \AA}$] and $\text{N}3=\text{C}1$ [$1.300(3)\text{ \AA}$] bonds (Fig. 1) show double-bond character. The other $\text{N}-\text{C}$ bonds have an intermediate character, indicating delocalization of the electrons. In the molecule, all of the bond lengths and angles are consistent with the reported structures of similarly substituted triazoles (Chen *et al.*, 1998; Kahveci & İzkizler, 2000; Çoruh *et al.*, 2003; Işık *et al.*, 2003a,b).

All six-membered rings and the triazole ring are planar. The dihedral angles between these three benzene rings [C3–C8 (ring 1), C9–C14 (ring 2) and C17–C22 (ring III)] are $33.6(1)$ (between rings 1/2), $25.7(1)$ (1/3) and $29.7(1)^\circ$ (2/3). Also, the dihedral angles between the benzene rings (1/2/3) and the triazole ring are $34.3(1)$, $52.9(1)$ and $59.5(1)^\circ$, respectively.

In the crystal structure, the molecules are linked by intermolecular $\text{N}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{N}$ contacts, resulting in a network structure (Fig. 2). The geometry of these interactions is listed in Table 2.

Experimental

The title compound (0.005 mol) was dissolved in 50 ml of dried methanol and NaBH_4 (0.005 mol) was added in small portions to this

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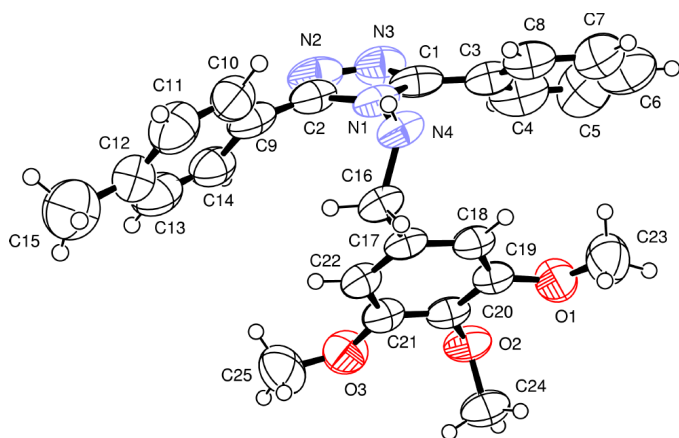


Figure 1
The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

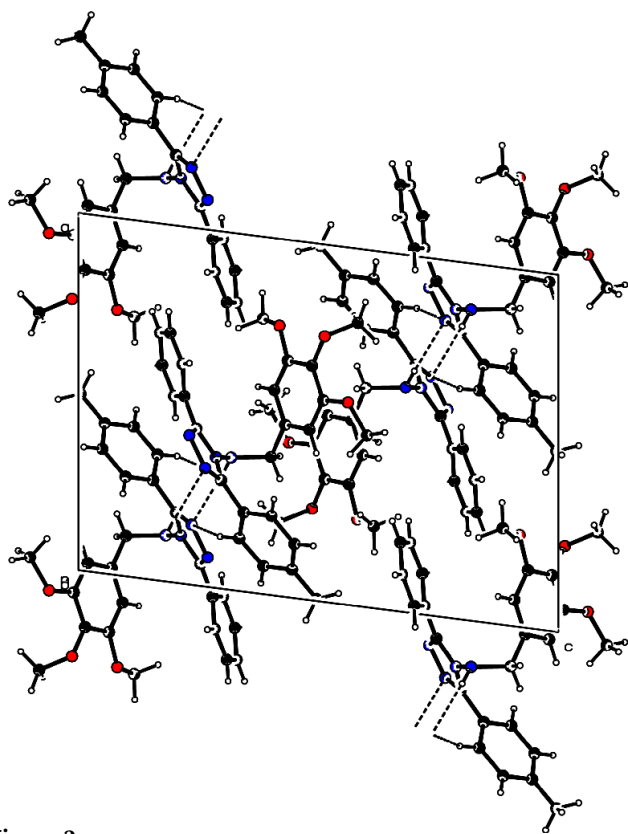


Figure 2
A crystal packing diagram of (I), viewed along the *b* axis. Intermolecular contacts are shown as dashed lines.

solution. The mixture was refluxed for 20 min and then allowed to cool. After evaporation at 298–303 K under reduced pressure, the resulting solid residue was washed with cold water. After drying *in vacuo*, the solid product was recrystallized from ethyl acetate to afford the desired compound, (I) (m.p = 417–418 K, yield = 95.8%). Calculated: C 69.75, H 6.09, N 13.01%; found: C 69.87, H 6.17, N 12.87%. IR data (KBr/ cm^{-1}): 3229 (N–H), 1592 (C=N), 693, 772, 822, 836 (aromatic). ^1H NMR (DMSO- d_6): δ 2.41 (CH₃, *s*, 3H), 3.60 (2OCH₃, *s*, 6H), 3.78 (OCH₃, *s*, 3H), 3.63 (CH₂, *d*, 2H), 5.82 (NH, *t*, 1H), 5.90 (aromatic H, *s*, 2H), 7.28 (aromatic H, *m*, 2H), 7.45 (aromatic H, *m*, 3H), 5.82 (aromatic H, *m*, 4H).

Crystal data

$\text{C}_{25}\text{H}_{25}\text{N}_4\text{O}_3$
 $M_r = 430.51$
Monoclinic, $P2_1/n$
 $a = 13.2141$ (9) Å
 $b = 9.8945$ (5) Å
 $c = 17.8861$ (12) Å
 $\beta = 97.336$ (5)°
 $V = 2319.4$ (3) Å³
 $Z = 4$

$D_x = 1.233$ Mg m⁻³
Mo K α radiation
Cell parameters from 22255 reflections
 $\theta = 1.8$ – 29.3°
 $\mu = 0.08$ mm⁻¹
 $T = 293$ (2) K
Prism, colourless
 $0.80 \times 0.51 \times 0.23$ mm

Data collection

Stoe IPDS 2 diffractometer
 ω rotation scans
Absorption correction: by integration (*X-RED32*; Stoe & Cie, 2002)
 $T_{\min} = 0.928$, $T_{\max} = 0.982$
6461 measured reflections

6461 independent reflections
2717 reflections with $I > 2\sigma(I)$
 $\theta_{\max} = 29.6^\circ$
 $h = -18 \rightarrow 18$
 $k = 0 \rightarrow 13$
 $l = 0 \rightarrow 24$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.070$
 $wR(F^2) = 0.224$
 $S = 0.83$
6461 reflections
298 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1262P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.88$ e Å⁻³
 $\Delta\rho_{\min} = -0.31$ e Å⁻³
Extinction correction: *SHELXL97*
Extinction coefficient: 0.013 (2)

Table 1

Selected geometric parameters (Å, °).

N4–N1	1.408 (2)	C2–N2	1.295 (3)
N4–C16	1.474 (3)	N2–N3	1.382 (4)
N1–C2	1.351 (3)	C1–N3	1.300 (3)
N1–C1	1.373 (3)		
N1–N4–C16–C17	56.6 (3)		

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H \cdots <i>A</i>	<i>D</i> –H	H \cdots <i>A</i>	<i>D</i> ⋯ <i>A</i>	<i>D</i> –H \cdots <i>A</i>
N4–H4N \cdots N2 ⁱ	0.81 (2)	2.47 (2)	3.277 (3)	176 (1)
C10–H10 \cdots N2 ⁱ	0.93	2.49	3.357 (4)	155

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

Atom H4N attached to N4 was located in a difference Fourier map and refined isotropically. The other H atoms were positioned geometrically and refined isotropically using a riding model (aromatic C–H = 0.93 Å, CH₂ = 0.97 Å and methyl C–H = 0.96 Å, with $U_{\text{iso}} = 1.2$ or 1.5 times U_{eq} of the parent atom. A relatively high maximum of residual electron density was observed at 0.38 Å from H6.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

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