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4-(2,4-Dichlorobenzylamino)-3-phenyl-5-*p*-tolyl-4*H*-1,2,4-triazole

Hande Petek,[a]* Ismet Şenel,[a] Olcay Bekircan,[b] Erbil Ağar[c] and Selami Şaşmaz[d]

[a]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139 Kurupelit-Samsun, Turkey, [b]Department of Chemistry, Giresun Art and Science Faculty, Karadeniz Teknik University, Giresun, Turkey, [c]Ondokuz Mayıs University, Art and Science Faculty, Department of Chemistry, 55139 Samsun, Turkey, and [d]Department of Chemistry, Rize Art and Science Faculty, Karadeniz Teknik University, Rize, Turkey

Correspondence e-mail: hpetek@omu.edu.tr

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The title compound, $C_{22}H_{18}Cl_2N_4$, displays the characteristic features of 1,2,4-triazole derivatives. There is an N—H···N intermolecular hydrogen bond.

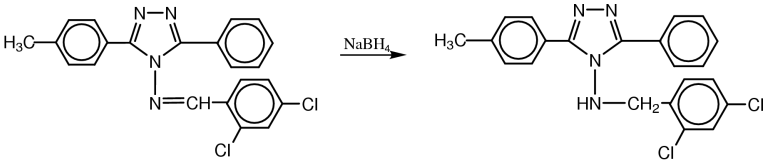
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.010 Å
R factor = 0.074
wR factor = 0.133
Data-to-parameter ratio = 14.4

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

Comment

1,2,4-Triazole and its derivatives are starting materials for the synthesis of many heterocycles (Desenko, 1995). Apart from its extensive chemical significance, the 1,2,4-triazole nucleus is also found to be associated with diverse medicinal properties, such as analgesic, anti-asthmatic, diuretic and antifungal activities (Mohamed *et al.*, 1993).



(I)

The 1,2,4-triazole ring (*A*: N1, N2, C9, N3 and C8) of the title compound, (I), is approximately planar, with a maximum deviation of 0.003 (7) Å for C8, and forms dihedral angles of 33.68 (17) and 32.86 (21)°, respectively, with the attached methylphenyl and phenyl rings. The dihedral angle between ring *B* (C2–C7) and ring *C* (C10–C15) is 12.1 (3)°. The other ring, *D* (C17–C22), is also essentially planar, with maximum deviations of 0.013 (6) Å for C17 and 0.010 (7) Å for C20. The N1=C8 and N2=C9 bonds display double-bond character, with bond distances of 1.320 (6) and 1.336 (6) Å, respectively. The bond lengths and angles of the triazole ring are comparable to reported values (Fun *et al.*, 1999; Zhu *et al.*, 2000). The Cl1—C18 and Cl2—C20 bond lengths [1.745 (6) and 1.725 (6) Å, respectively] are in agreement with those found

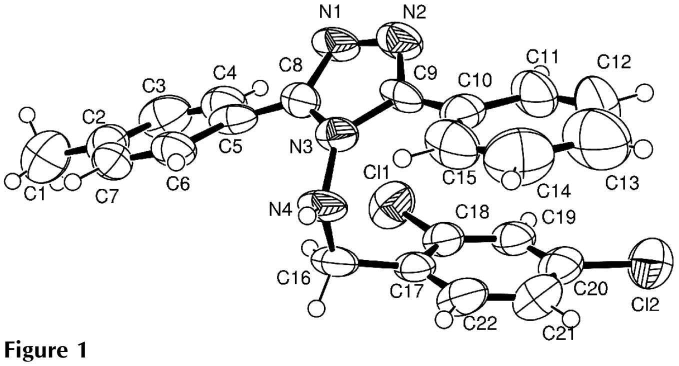


Figure 1
An *ORTEP*III (Burnett & Johnson, 1996) view of the title compound, showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level.

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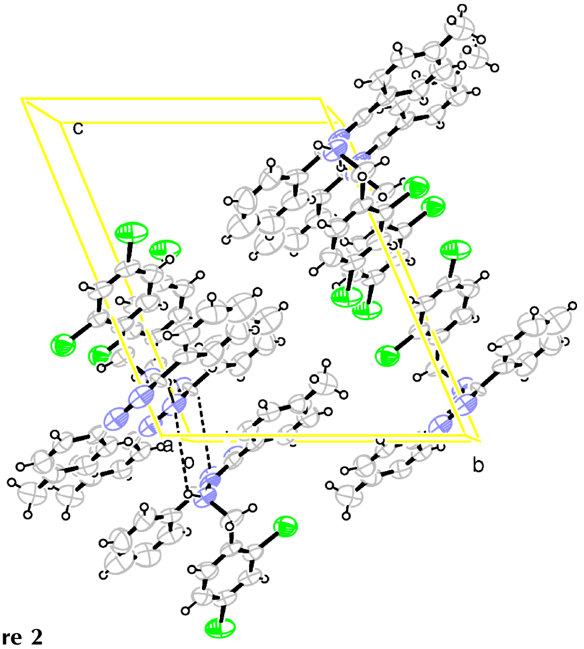

Figure 2
Packing diagram of the structure of (I), viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

previously (Ocak *et al.*, 2003; Puviarasan *et al.*, 1999). There are five π–π stacking interactions in (I). These involve ring *A* at (x, y, z) with ring *A* at $(-x, 2-y, -z)$, ring *B* at (x, y, z) with ring *C* at $(-x, 2-y, -z)$, ring *C* at (x, y, z) with ring *B* at $(-x, 2-y, -z)$, ring *C* at (x, y, z) with ring *D* at (x, y, z), and ring *D* at (x, y, z) with ring *D* at $(-x, 2-y, 1-z)$, with distances of 3.449 (4), 3.816 (4), 3.816 (4), 3.998 (5) and 3.797 (4) Å, respectively, between the centres of the rings.

Experimental

3-Phenyl-5-*p*-tolyl-4-(2,4-dichlorobenzilideneamino)-4*H*-1,2,4-triazole (0.005 mol) was dissolved in 50 ml of dried methanol and $NaBH_4$ (0.005 mol) was added in small portions to this solution. The mixture was refluxed for 20 minutes 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, yield 92%, m.p. 465–466 K. IR (KBr): 3242, 1598, 867, 818, 805, 765, 686; ^{1}H NMR ($CDCl_3$): δ 2.45 (*s*, 3H, CH_3),3.82 (*d*,2H,CH_2), 5.85 (*t*, 1H, NH), Ar–H; 6.55 (*m*, 1H), 6.95 (*m*, 2H), 7.30 (*d*,2H), 7.45 (*m*, 3H), 7.75 (*m*, 4H), ^{13}C NMR ($CDCl_3$). δ 155.38 (2C), 140.35, 135.01, 131.92, 130.61, 129.98, 129.48 (2C), 129.45, 129.30, 128.68 (2C), 127.96 (2C), 127.82 (2C), 127.07, 126.37, 123.48, 53.15, 21.46, UV [λ_{max}, nm ($\varepsilon \times 10^{-3}$)]: 260 (22.6), 213 (24.6). Analysis: calculated for ($C_{22}H_{18}N_4Cl_2$); C: 64.56, H:4.43, N:13.69; found: C: 64.75, H:4.41, N:13.58.

Crystal data

$C_{22}H_{18}Cl_2N_4$
$M_r = 409.30$
Triclinic, $P\overline{1}$
$a = 6.2604$ (12) Å
$b = 11.952$ (3) Å
$c = 14.775$ (3) Å
$\alpha = 113.152$ (16)°
$\beta = 91.984$ (15)°
$\gamma = 95.864$ (17)°
$V = 1007.8$ (4) Å^3
$Z = 2$
$D_x = 1.349$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4695 reflections
$\theta = 1.9$–26.7°
$\mu = 0.34$ mm^{-1}
$T = 293$ (2) K
Plate, colourless
0.18 × 0.14 × 0.07 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{min} = 0.924$, $T_{max} = 0.969$
13341 measured reflections
3654 independent reflections
1272 reflections with $I > 2\sigma(I)$
$R_{int} = 0.149$
$\theta_{max} = 25.3$°
$h = -7 \rightarrow 7$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.074$
$wR(F^2) = 0.133$
$S = 0.93$
3654 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0303P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C18	1.745 (6)	N1—C8	1.320 (6)
Cl2—C20	1.725 (6)	N1—N2	1.399 (6)
N3—C8	1.352 (6)	N2—C9	1.336 (6)
N3—C9	1.385 (6)		
N3—N4—C16	112.7 (4)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···N2^{i}	0.86	2.39	3.031 (6)	132

Symmetry code: (i) $x-1, y, z$.

The H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C—H distances at 0.93 Å, methylene C—H distances at 0.97 Å, methyl C—H distances at 0.96 Å and N—H distances at 0.86 Å. U_{iso}(H) values were set to $1.2U_{eq}$ ($1.5U_{eq}$ for the methyl group) of the parent atom. The high R_{int} value is due to the poor quality of the crysatl.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (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).

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