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Crystal structure of (*E*)-(2-methoxy-benzylidene)-(4-[1,2,4]triazol-1-yl-phenyl)-amine, C₁₆H₁₄N₄O

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Abstract

C₁₆H₁₄N₄O, triclinic, $P\bar{1}$ (no. 2), $a = 8.721(5)$ Å, $b = 12.116(5)$ Å, $c = 13.413(5)$ Å, $\alpha = 88.870(5)^\circ$, $\beta = 86.304(5)^\circ$, $\gamma = 80.568(5)^\circ$, $V = 1395.1(11)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0543$, $wR_{\text{ref}}(F^2) = 0.1645$, $T = 298(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.30 × 0.20 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	Bruker Smart Apex, ω
θ_{max} , completeness:	27.2°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10,575, 5516, 0.049
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4614
$N(\text{param})_{\text{refined}}$:	382
Programs:	Bruker [1], Olex2 [2], SHELX [3]

Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Source of material

All chemicals were purchased from commercial sources and used as received without further purification. Under stirring, 4-[1,2,4]triazol-1-yl-phenylamine (4.00 mmol,

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
O1	0.27522 (14)	−0.00568 (10)	0.97368 (9)	0.0592 (3)
N1	−0.19096 (13)	0.40970 (10)	0.52191 (8)	0.0399 (3)
N2	−0.10429 (16)	0.36964 (12)	0.43806 (9)	0.0561 (4)
N3	−0.30142 (17)	0.50571 (12)	0.39791 (10)	0.0542 (4)
N4	−0.05979 (15)	0.25301 (10)	0.91010 (9)	0.0459 (3)
C1	−0.1766 (2)	0.43079 (14)	0.36728 (12)	0.0554 (4)
H1	−0.143144	0.422569	0.300191	0.067*
C2	−0.30579 (18)	0.49041 (13)	0.49518 (11)	0.0486 (4)
H2	−0.379200	0.530468	0.539878	0.058*
C3	−0.15533 (16)	0.36630 (11)	0.61903 (10)	0.0374 (3)
C4	−0.00442 (17)	0.31836 (13)	0.63746 (11)	0.0467 (4)
H4	0.073667	0.313135	0.586365	0.056*
C5	0.03009 (17)	0.27809 (13)	0.73247 (11)	0.0474 (4)
H5	0.131223	0.245085	0.744640	0.057*
C6	−0.08605 (16)	0.28688 (11)	0.80966 (10)	0.0403 (3)
C7	−0.23677 (17)	0.33689 (12)	0.78968 (11)	0.0443 (3)
H7	−0.314487	0.344625	0.841076	0.053*
C8	−0.27283 (16)	0.37520 (12)	0.69484 (10)	0.0429 (3)
H8	−0.374386	0.406509	0.682006	0.052*
C9	0.04335 (17)	0.16986 (12)	0.92953 (11)	0.0434 (3)
H9	0.103346	0.131608	0.877325	0.052*
C10	0.06955 (17)	0.13305 (12)	1.03295 (10)	0.0423 (3)
C11	0.18528 (17)	0.04162 (12)	1.05393 (11)	0.0455 (3)
C12	0.2038 (2)	0.00462 (14)	1.15214 (12)	0.0572 (4)
H12	0.279977	−0.055990	1.165876	0.069*
C13	0.1090 (2)	0.05807 (15)	1.22906 (12)	0.0626 (5)
H13	0.121980	0.033074	1.294452	0.075*
C14	−0.0051 (2)	0.14824 (15)	1.21012 (12)	0.0598 (4)
H14	−0.068159	0.183912	1.262373	0.072*
C15	−0.0244 (2)	0.18481 (13)	1.11280 (12)	0.0512 (4)
H15	−0.101568	0.245152	1.100121	0.061*
C16	0.3811 (2)	−0.10682 (15)	0.99030 (16)	0.0699 (5)
H16A	0.433419	−0.132876	0.927947	0.105*
H16B	0.324385	−0.162707	1.018537	0.105*
H16C	0.456381	−0.092731	1.035575	0.105*
O2	0.76343 (14)	0.99833 (9)	0.51039 (8)	0.0572 (3)
N5	0.69815 (13)	0.58124 (10)	0.99306 (8)	0.0399 (3)
N6	0.61966 (16)	0.63438 (12)	1.07473 (9)	0.0536 (4)
N7	0.80753 (15)	0.49468 (11)	1.12173 (9)	0.0498 (3)
N8	0.57036 (14)	0.73578 (10)	0.60405 (9)	0.0434 (3)
C17	0.69104 (19)	0.57859 (14)	1.14853 (11)	0.0524 (4)
H17	0.662799	0.596012	1.215117	0.063*
C18	0.80763 (17)	0.49931 (12)	1.02367 (11)	0.0445 (3)
H18	0.875191	0.451688	0.981055	0.053*
C19	0.66554 (16)	0.61822 (11)	0.89346 (10)	0.0382 (3)
C20	0.51301 (16)	0.64523 (12)	0.86690 (11)	0.0443 (3)
H20	0.431246	0.638520	0.913478	0.053*
C21	0.48263 (17)	0.68229 (13)	0.77066 (11)	0.0459 (4)
H21	0.380355	0.699114	0.752247	0.055*
C22	0.60497 (16)	0.69440 (11)	0.70147 (10)	0.0392 (3)
C23	0.75728 (17)	0.66234 (13)	0.72837 (11)	0.0482 (4)
H23	0.839374	0.666248	0.681307	0.058*
C24	0.78847 (17)	0.62467 (13)	0.82426 (11)	0.0471 (4)
H24	0.890678	0.603978	0.841884	0.057*
C25	0.64573 (17)	0.81053 (12)	0.56838 (10)	0.0429 (3)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H25	0.713999	0.837717	0.608494	0.052*
C26	0.62945 (16)	0.85561 (12)	0.46666 (10)	0.0415 (3)
C27	0.69152 (16)	0.95216 (12)	0.43840 (11)	0.0438 (3)
C28	0.67620 (19)	0.99467 (14)	0.34148 (12)	0.0535 (4)
H28	0.717174	1.058592	0.322274	0.064*
C29	0.6003 (2)	0.94185 (15)	0.27416 (12)	0.0588 (4)
H29	0.589952	0.971102	0.209862	0.071*
C30	0.5393 (2)	0.84654 (15)	0.30033 (12)	0.0550 (4)
H30	0.488981	0.811300	0.254207	0.066*
C31	0.55441 (18)	0.80413 (13)	0.39658 (11)	0.0479 (4)
H31	0.513495	0.739874	0.414714	0.057*
C32	0.8181 (2)	1.10139 (15)	0.48802 (15)	0.0649 (5)
H32A	0.902209	1.089249	0.437583	0.097*
H32B	0.854169	1.129090	0.547328	0.097*
H32C	0.734787	1.155112	0.464111	0.097*

0.64 g) was added to a solution of 2-methoxy-benzaldehyde (6.98 mmol, 0.95 g) in ethanol (30 mL) in a 150 mL round bottom flask. The mixture was refluxed for 5 h, and then the precipitate was collected by filtration and washed with ethanol. The product was recrystallized from methanol to afford white crystal. Yield: 0.86 g (77.34%). **m.p.:** 212.1–212.4 °C. **¹H-NMR** (CDCl₃, 400 MHz, ppm): δ 8.96 (s, 1H), 8.56 (s, 1H), 8.16 (dd, *J* = 7.72 Hz, 1.76 Hz, 1H), 8.12 (s, 1H), 7.69 (dt, *J* = 9.48 Hz, 2.81 Hz, 2H), 7.48 (td, *J* = 7.90 Hz, 1.76 Hz, 1H), 7.35 (dt, *J* = 9.52 Hz, 2.81 Hz, 2H), 7.07 (t, *J* = 7.56 Hz, 1H), 6.98 (d, *J* = 8.36 Hz, 1H), 3.92 (s, 3H).

Experimental details

All H-atoms bonded to C atoms were placed geometrically and refined using a riding model with common isotropic displacement factors $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}(\text{parent C-atom})$.

Comment

1,2,4-triazol-based compounds received notable interest because of their prominent photophysical and chemical properties, and excellent biological activities, and have wide applications in the fields of biology, agriculture, pharmacy, and functional materials, and so on [4–7]. The title compound was synthesized by a standard aldehyde-amine condensation reaction to give the Schiff base [8–10]. Furthermore, the title compound is an excellent N-containing ligand.

As shown in the figure, the title compound shows an E configuration in both crystallographically independent molecule with respect to the C=N bond, and the C=N bond lengths are 1.271 (N4=C9) and 1.272 (N8=C25) Å, which are

shorter than the normal C=N bond length (1.34–1.38 Å). The C9–C10 and C25–C26 bond lengths are 1.469 and 1.466 Å, respectively. The N4–C6 and N8–C22 bond lengths are 1.422 and 1.423 Å, respectively. According to the X-ray diffraction analysis, there is partially delocalized electron and significant conjugation in the title compound. All geometric parameters are in the expected ranges [11].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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