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4-[3-(1*H*-Imidazol-1-yl)propyl]-3-phenyl-1*H*-1,2,4-triazol-5(4*H*)-one

Hüseyin Kalkan,[a] Reşat Ustabaş,[b]* Kemal Sancak,[c] Yasemin Ünver[c] and Ezequiel M. Vázquez-López[d]

[a]Department of Elementary Education, Educational Faculty, Ondokuz Mayıs University, 55200 Atakum-Samsun, Turkey, [b]Anatolian Trade High School, Teacher of Physics, Merzifon 05300, Amasya, Turkey, [c]Department of Chemistry, Faculty of Arts and Sciences, Karadeniz Teknik University, 61080 Trabzon, Turkey, and [d]Departamento de Química Inorgánica, Facultade de Ciencias-Química, Universidade de Vigo, 36200-Vigo, Galicia, Spain

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

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.047
wR factor = 0.105
Data-to-parameter ratio = 8.2

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

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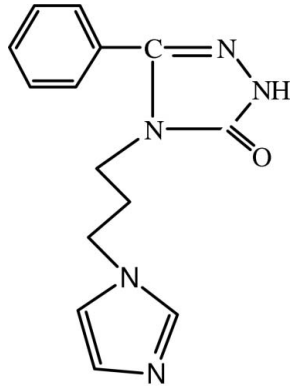
The title compound, $C_{14}H_{15}N_5O$, has two similar independent molecules in the asymmetric unit. The crystal structure is stabilized by C—H···O, C—H···N and N—H···N intermolecular hydrogen bonding.

Comment

The considerable biological importance of imidazoles and triazoles has stimulated much work on these heterocycles and there are a variety of methods available for the synthesis of 1,2,4-triazole (Ray & Hank, 1990; İkizler & Sancak, 1992; Mullican *et al.*, 1993). 1,2,4-Triazole and imidazole are the hetero-rings of choice and are essential structural features of many of the potent azole fungicides (Narayana *et al.*, 1993). It is also known that the triazole ring has been used instead of imidazole, which is found in the structure of some antagonist, anti-ulcer and antifungal drugs (Menozzi *et al.*, 2001). In a continuing search for pharmacologically active imidazole and 1,2,4-triazole compounds, it has been found that most azole fungicides have been developed for diseases of cereal crops; examples include fluconazole (Ichikawa *et al.*, 2001), ravuconazole (Ueda, 2003) and posaconazole (Kim *et al.*, 2003). Furthermore, in many compounds, the thiophene unit is associated with high anticancer and antifungal activity (Smith *et al.*, 2001; Nakib *et al.*, 1994). Spectroscopic and crystal structure data of some 1,2,4-triazoles have been reported previously (Çoruh *et al.*, 2003; Sancak *et al.*, 2005). In this paper, we present the synthesis, molecular and crystal structure of the title compound, (I).



(I)

In compound (I), two independent molecules with similar conformations, labelled a and b, exist in the asymmetric unit (Fig. 1). The bond lengths and angles (Table 1) are within normal ranges (Ocak *et al.*, 2003; Ünver *et al.*, 2006; Çoruh *et*

al., 2003; Ustabaş *et al.*, 2006). Atoms N2a and N2b have a trigonal configuration, the sums of the three bond angles around them being 360°.

The dihedral angles between the planes *A* (N2a/C2a/C1a/N1a/C3a), *B* (N3a/C7a/N4a/N5a/C8a), *C* (C9a–C14a) and *A*′ (N2b/C2b/C1b/N1b/C3b), *B*′ (N3b/C7b/N4b/N5b/C8b), *C*′ (C9b–C14b) are *A*/*B* = 60.78 (10)°, *A*/*C* = 34.22 (11)°, *B*/*C* = 56.37 (9)° and *A*′/*B*′ = 59.09 (12)°, *A*′/*C*′ = 29.19 (11)° and *B*′/*C*′ = 54.04 (10)°.

The crystal structure of (I) is stabilized by intermolecular hydrogen bonding (Table 2).

Experimental

Ethyl benzoate ethoxycarbonyl hydrazone (0.01 mol) and *N*-(3-aminopropyl)imidazole (0.01 mol) were heated at 433–438 K in an oil bath for 3 h. After cooling to room temperature, a solid appeared. It was recrystallized from acetone/petroleum ether (1:2) (yield 72%) to afford the desired compound (I).

Crystal data

$C_{14}H_{15}N_5O$
M_r = 269.31
Orthorhombic, $P2_12_12_1$
a = 10.073 (5) Å
b = 15.594 (8) Å
c = 17.669 (9) Å
V = 2775 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.50 × 0.45 × 0.40 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
13657 measured reflections
2953 independent reflections
2237 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.105
S = 1.00
2953 reflections
362 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N2b—C3b	1.337 (3)	N2a—C3a	1.338 (3)
N2b—C2b	1.361 (4)	N2a—C2a	1.357 (4)
N2b—C4b	1.458 (3)	N2a—C4a	1.459 (3)
N3b—C8b	1.374 (3)	N3a—C8a	1.380 (3)
N5b—N4b	1.370 (4)	N4a—N5a	1.378 (4)
N1b—C1b	1.363 (4)	N1a—C1a	1.362 (4)
O1b—C7b	1.218 (4)	O1a—C7a	1.219 (3)
C3b—N2b—C2b	106.1 (2)	C3a—N2a—C2a	106.7 (2)
C3b—N2b—C4b	127.2 (3)	C3a—N2a—C4a	126.6 (3)
C2b—N2b—C4b	126.7 (2)	C2a—N2a—C4a	126.7 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4a—H7a···N1a^{i}	0.86	2.01	2.845 (4)	164
N4b—H7b···N1b^{ii}	0.86	1.99	2.825 (4)	163
C2b—H2b···N1a^{iii}	0.93	2.59	3.518 (4)	172
C3a—H3a···O1b	0.93	2.57	3.343 (4)	140

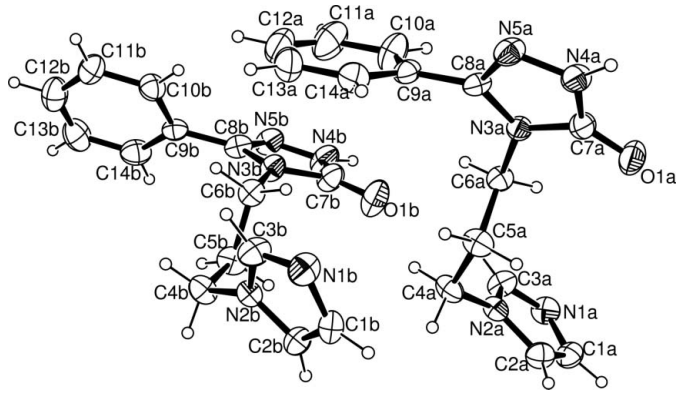


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3b—H3b···O1a^{iv}	0.93	2.44	3.233 (4)	143
C4a—H4a2···O1b	0.97	2.48	3.365 (4)	152
C4b—H4b2···O1a^{iv}	0.97	2.49	3.335 (4)	146

Symmetry codes: (i) $-x+\frac{3}{2}, -y+1, z+\frac{1}{2}$; (ii) $-x+\frac{1}{2}, -y+1, z-\frac{1}{2}$; (iii) $x-\frac{1}{2}, -y+\frac{1}{2}, -z$; (iv) $x-1, y, z$.

The crystal was of poor quality. All H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H = 0.93 (aromatic) and 0.97 Å (methylene), N—H = 0.86 Å, and U_{iso}(H) = 1.2U_{eq}(C,N). In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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