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Octane-1,8-diylbis[3-ethyl-1*H*-1,2,4-triazol-5(4*H*)-one]

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.136
Data-to-parameter ratio = 16.5

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

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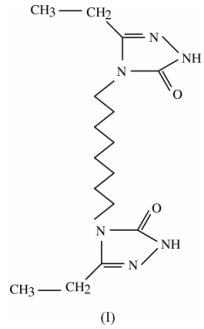
The title compound, $C_{16}H_{28}N_6O_2$, has a centre of symmetry. There are two planar 1,2,4-triazole rings, connected by an octane group. The crystal structure is stabilized by C—H···N, C—H···O and N—H···O intermolecular hydrogen bonds.

Comment

Schiff bases of 4-amino-1,2,4-triazole have received considerable attention over the past few decades (Kitaev *et al.*, 1971; Mazza *et al.*, 1976; Kargin *et al.*, 1988). It is of interest that some of them are anti-inflammatory agents (Gupta & Bhargava, 1978) and new concidiostatic drugs (Colauti *et al.*, 1971). Some other diverse pharmacological properties of 1,2,4-triazoles are analgesic, antiasthmatic, diuretic, fungicidal, bactericidal and pesticidal activities (Bennur *et al.*, 1976; Webb & Parsons, 1977; Heubach *et al.*, 1980; Mohamed *et al.*, 1993). Therefore, the structures of 1,2,4-triazole derivatives with different substituents have been the subject of much interest in our laboratory. Examples include 1-acetyl-3-(*p*-chlorobenzyl)-4-benzylidenamino-4,5-dihydro-1*H*-1,2,4-triazol-5-one, (II) (Çoruh, 2002), 1-acetyl-4-(*p*-chlorobenzylidenamino)-3-acetyl-4,5-dihydro-1*H*-1,2,4-triazol-5-one, (III) (Çoruh, Kahveci, Şaşmaz, Ağar & Kim, 2003), 1-acetyl-3-(*p*-chlorobenzyl)-4-(*p*-chlorobenzylidenamino)-4,5-dihydro-1*H*-1,2,4-triazol-5-one, (IV) (Ocak *et al.*, 2003), and C—H···O and C—H···π interactions in 1-acetyl-4-(*p*-chlorobenzylidenamino)-3-ethyl-4,5-dihydro-1*H*-1,2,4-triazol-5-one, (V) (Çoruh, Kahveci, Şaşmaz, Ağar, Kim & Erdönmez, 2003).

The molecular structure of (I) is shown in Fig. 1. The compound consists of two 1,2,4-triazole rings, each with an ethyl group on the C atom in the 3-position and an oxo O atom on the C atom at the 5-position, and linked by an octane chain attached to their N atoms at the 4-position. The molecule has a centre of symmetry in the middle of this connecting chain.



(I)

In the molecule, the placement of the ethyl group and the oxo O atom are very similar to a previously reported example

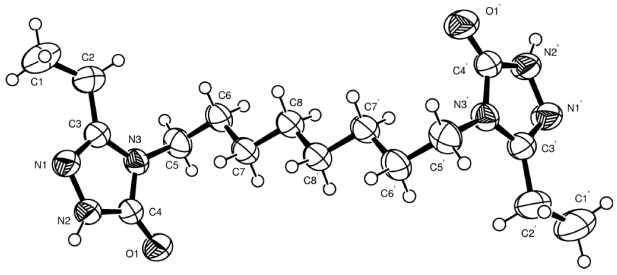


Figure 1
A view of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Primed atoms are related by the centre of symmetry [code (i) in Table 1].

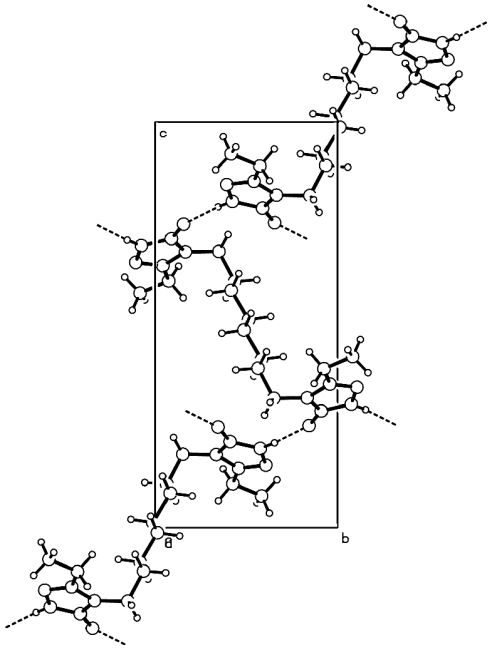


Figure 2
The strong hydrogen-bond network observed in (I), viewed along the *a* axis.

(Çoruh, Kahveci, Şaşmaz, Ağar & Kim, 2003). As the C—H···N hydrogen bond involves N1 as acceptor (Table 2), the N3═C1 bond length, 1.288 (3) Å, is a little longer than some values reported in the literature [1.272 (3) Å in $C_{13}H_{13}ClN_4O_2$ (Çoruh, Kahveci, Şaşmaz, Ağar, Kim & Erdönmez, 2003), 1.261 (4) Å in the 4-amino-3-methyl-1,2,4-triazole-5-thione derivative of *p*-nitrobenzaldehyde (Liu *et al.*, 1999), and 1.267 (2) Å in 4-(4-hydroxybenzylidenamino)-4*H*-1,2,4-triazole hemihydrate (Zhu *et al.*, 2000)]. However, it is close to other reported values (Puviarasan *et al.*, 1999; Çoruh, Kahveci, Şaşmaz, Ağar & Kim, 2003; Ocak *et al.*, 2003). In the 1,2,4-triazole ring, atoms N1 and N2 have no substituents, and the N1—N2 bond length, 1.372 (2) Å, is essentially identical to that [1.373 (2) Å] reported for a similar compound (Liu *et al.*, 1999). This is shorter than in compounds where at least one N atom has a substituent [1.394 (3) Å (Çoruh, Kahveci, Şaşmaz, Ağar, Kim & Erdönmez, 2003), 1.399 (2) Å (Çoruh, Kahveci, Şaşmaz, Ağar & Kim, 2003) and 1.404 (4) Å (Ocak *et al.*, 2003)]. In (I), the 1,2,4-triazole ring is planar, with a maximum deviation from the least-squares plane of 0.0017 (1) Å for atom N1. Atom O1 is also in the plane, with a deviation of only 0.0006 (1) Å.

In addition to van der Waals interactions, the molecular structure and crystal packing of (I) are stabilized by C—H···O, C—H···N and N—H···O intermolecular interactions (Fig. 2 and Table 2).

Experimental

1,8-Diaminooctane (1,44 g, 0.001 mol) was dissolved in water (100 ml) and ethyl propionate ethoxycarbonylhydrazone (3.76 g, 0.02 mol) was added. The reaction mixture was refluxed for 6 h and then cooled to room temperature. The precipitate was filtered off and washed with cold water. After drying *in vacuo*, the solid product was recrystallized from ethanol-water (1:2) to afford the desired compound, (I) (yield 2.82 g, 84%). M.p. 452–453 K. IR (KBr, cm^{-1}): ν(N—H) 3165, 3063; ν(C═O) 1680 and ν(C═N) 1560. ^{1}H NMR (p.p.m. in DMSO-d_6): 0.54, 1.86 (*m*, 18H, $6CH_2$, $2CH_3$); 2.54 (*q*, 4H, $2CH_2$); 3.44 (*t*, 2N—CH_2, 4H); 11.26 (*s*, 2N—H, 2H).

Crystal data

$C_{16}H_{28}N_6O_2$
M_r = 336.44
Monoclinic, $P2_1/c$
a = 6.7852 (10) Å
b = 7.829 (2) Å
c = 17.550 (3) Å
β = 95.894 (10)°
V = 927.4 (3) Å^3
Z = 2
D_x = 1.205 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 8.0–12.8°
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, colorless
0.45 × 0.35 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 MACH3 diffractometer
$2\theta/\omega$ scans
Absorption correction: none
1975 measured reflections
1817 independent reflections
1162 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 26.0°
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 9$
$l = -21 \rightarrow 21$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.136
S = 1.03
1817 reflections
110 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0712P)^2 + 0.0879P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.224 (2)	C4—N3	1.367 (2)
C1—C2	1.480 (3)	N3—C5	1.463 (2)
C2—C3	1.482 (3)	C5—C6	1.509 (3)
C3—N1	1.288 (2)	C6—C7	1.505 (3)
C3—N3	1.359 (2)	C7—C8	1.512 (3)
N1—N2	1.372 (2)	C8—C8^{i}	1.504 (4)
N2—C4	1.330 (2)		
C1—C2—C3	113.4 (2)	N2—C4—N3	103.79 (15)
N1—C3—N3	111.56 (15)	C3—N3—C4	107.73 (14)
N1—C3—C2	124.48 (17)	C3—N3—C5	128.06 (16)
N3—C3—C2	123.88 (17)	C4—N3—C5	124.16 (16)
C3—N1—N2	104.08 (14)	N3—C5—C6	113.17 (17)
C4—N2—N1	112.83 (14)	C7—C6—C5	114.30 (16)
O1—C4—N2	128.01 (18)	C6—C7—C8	112.91 (15)
O1—C4—N3	128.20 (18)	C8^{i}—C8—C7	113.70 (19)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.86	1.90	2.745 (3)	168
C2—H2B···O1^{ii}	0.97	2.80	3.719 (3)	158
C5—H5A···N2^{iii}	0.97	2.94	3.803 (3)	148
C7—H7A···O1^{iv}	0.97	2.68	3.547 (3)	149
C6—H6A···N1^{v}	0.97	2.74	3.685 (3)	165

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

The H atoms were positioned geometrically and refined using a riding model, with ethyl C—H = 0.97 Å, methyl C—H = 0.96 Å, and N—H = 0.86 Å; U_{iso}(H) was set to $1.2U_{eq}$ of the parent atom in each case.

Data collection: *CAD*-4-*PC Software* (Enraf–Nonius, 1992); cell refinement: *CAD*-4-*PC Software*; data reduction: *XCAD*4 (Harms, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*3 *for Windows* (Farrugia, 1997) and *PLATON* (Spek, 1997); software used to prepare material for publication: *SHELXL*97 and *WinGX* (Farrugia, 1999).

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