

3,3'-Dimethyl-4,4'-(hexane-1,6-diyl)-bis[1*H*-1,2,4-triazol-5(4*H*)-one]

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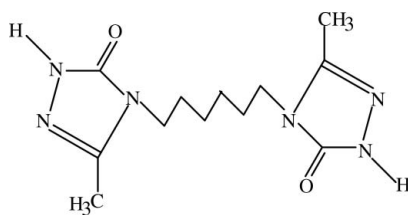
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Key indicators: single-crystal X-ray study; $T = 101$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.041; wR factor = 0.112; data-to-parameter ratio = 12.8.

The title compound, $\text{C}_{12}\text{H}_{20}\text{N}_6\text{O}_2$, has a centre of symmetry. The molecule consists of two triazole rings joined by an aliphatic $-(\text{CH}_2)_6-$ chain. The crystal structure is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds and by $\pi-\pi$ stacking interactions between the triazole rings of inversion-related molecules [centroid-centroid distance = $3.277(8)$ Å].

Related literature

For background information including pharmacological studies, see: Chiu & Huskey (1998); Clemons *et al.* (2004); Dalloul & Boyle (2006); Elliott *et al.* (1986); Griffin & Mannion (1986); Santen (2003); Tanaka (1974); Zamani *et al.* (2003). Related structures have been reported by Ustabaş *et al.* (2006, 2007, 2009); Ünver *et al.* (2008, 2009); Çoruh *et al.* (2003).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{20}\text{N}_6\text{O}_2$
 $M_r = 280.34$
Triclinic, $P\bar{1}$
 $a = 6.3641(2)$ Å

$b = 7.3034(2)$ Å
 $c = 7.7774(2)$ Å
 $\alpha = 93.299(2)^\circ$
 $\beta = 109.578(2)^\circ$

$\gamma = 94.707(2)^\circ$
 $V = 338.05(2)$ Å³
 $Z = 1$
Mo $K\alpha$ radiation

$\mu = 0.10$ mm⁻¹
 $T = 101$ K
 $0.40 \times 0.16 \times 0.12$ mm

Data collection

Bruker Kappa APEXII CCD
area-detector diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2007)
 $T_{\min} = 0.962$, $T_{\max} = 0.988$

6074 measured reflections
1673 independent reflections
1309 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.112$
 $S = 1.03$
1673 reflections

131 parameters
All H-atom parameters refined
 $\Delta\rho_{\text{max}} = 0.32$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N3}-\text{H3}\cdots\text{O1}^i$	0.90 (2)	1.89 (2)	2.7707 (15)	167 (2)

Symmetry code: (i) $-x + 1, -y, -z + 2$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2267).

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supporting information

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3,3'-Dimethyl-4,4'-(hexane-1,6-diyl)bis[1*H*-1,2,4-triazol-5(4*H*)-one]

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S1. Comment

The 1,2,4-triazole compounds possess important pharmacological activities that include antifungal and antiviral properties. Examples of compounds bearing the 1,2,4-triazole group are fluconazole, the powerful azole antifungal agent as well as the potent antiviral N- nucleoside ribavirin (Ünver *et al.*, 2008; Ünver *et al.*, 2009). Furthermore, various 1,2,4-triazole derivatives have been reported as fungicidal (Zamani *et al.*, 2003), insecticidal (Tanaka, 1974), antimicrobial (Griffin & Mannion, 1986), and some showed antitumor activity as well as having anticonvulsant (Dalloul & Boyle, 2006), antidepressant (Chiu & Huskey, 1998) and plant growth regulator anticoagulant activity (Elliott *et al.*, 1986). It was reported that compounds having triazole moieties, such as Vorozole, Anastrozole and Letrozole appear to be very effective aromatase inhibitors and can be useful for preventing breast cancer (Santen, 2003; Clemons *et al.*, 2004).

The molecular structure of the compound is shown in Fig.1. The molecule consists of two triazole rings, joined by an aliphatic $-(CH_2)_6-$ chain connected to nitrogen atoms of the rings. The molecule has an inversion center in the middle of the chain, that connects the triazole rings. The length of the N=C [N2=C5= 1.3031 (17) Å] bond in the triazole ring is close to the those similar structures in the literature [1.296 (3)Å in C₁₄H₁₆N₆O₂S (Ustabaş *et al.*, 2007); 1.288 (2)Å in C₁₆H₂₈N₆O₂ (Çoruh *et al.*, 2003)]. The bond length of O=C [O1=C1= 1.2421 (16) Å] is in conformity with the values mentioned before[1.218 (3)Å in C₁₆H₂₀N₆O₂S (Ustabaş *et al.*, 2006); 1.220 (2)Å in C₂₄H₂₀N₄O₂S (Ustabaş *et al.*, 2009)]. The triazole ring is very close to planarity, with a maximum deviation from the least-squares plane of -0.014 (13)Å for atom C1.

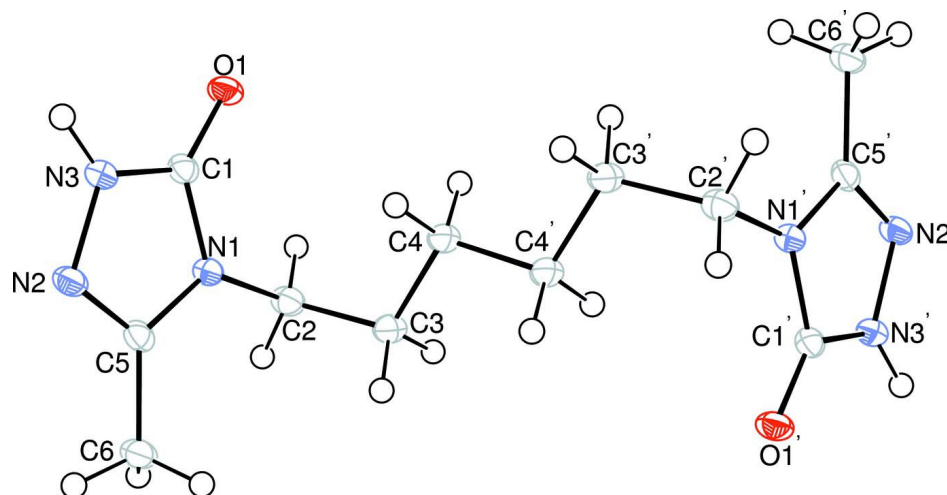
In the crystal structure of the compound, there is a strong intermolecular N3—H3···O1 hydrogen-bonding interaction (Table 1). The compound also exhibits π - π stacking interactions between triazole rings ($Cg1 \cdots Cg1 = 3.277$ (8) Å; symmetry code: $-X, 2-Y, -Z$).

S2. Experimental

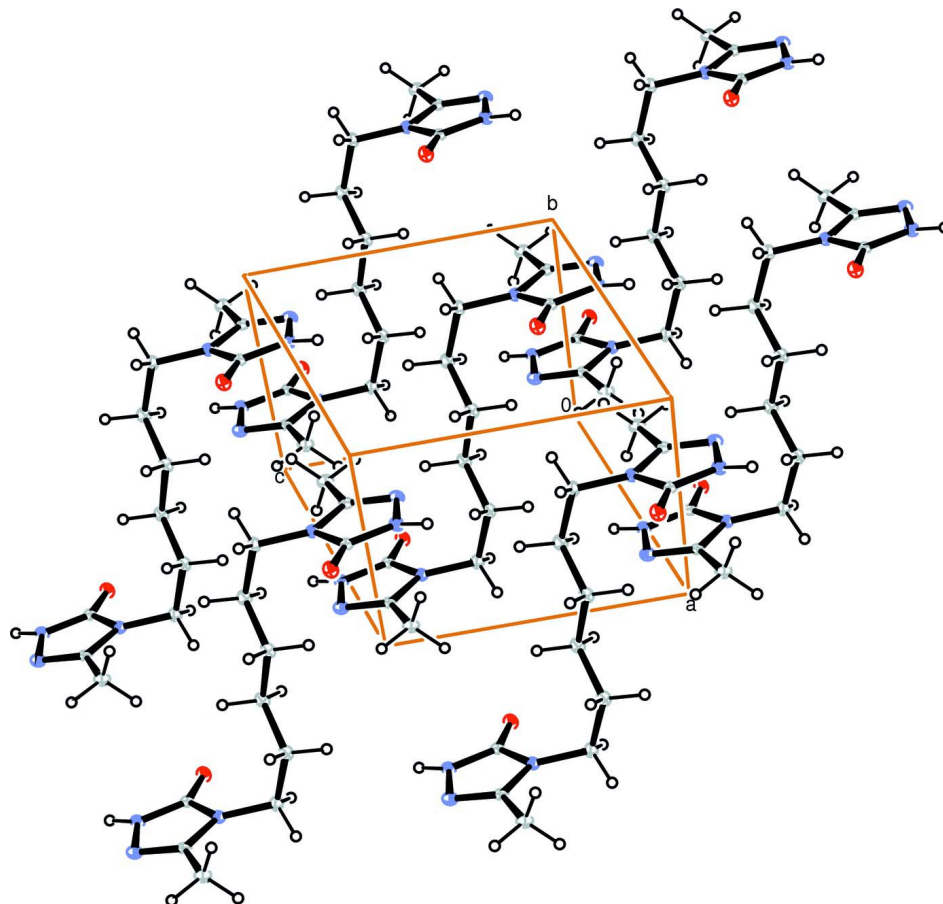
The synthesis of 4,4'-(hexane-1,6-diyl)bis (5-ethyl-2*H*-1,2,4-triazol-3(4*H*)-one) to a solution of ethyl 2 (1-ethoxyethylidene)hydrazinecarboxylate (0.02 mol) in 50 ml water hexane-1,6-diamine (0.01 mol) was added. Having refluxed this mixture for 4 h the precipitate formed was filtered off. The solid product was washed with water and crystallized from ethanol/water (1/3)(yield 73.25%) to afford the desired compound.

S3. Refinement

All H atoms were located in a difference synthesis and refined [N—H = 0.902 (19) Å; ethylene C—H = 0.945 (18) Å-1.017 (18) Å; and methylene C—H = 0.952 Å-1.00 (2) Å].

**Figure 1**

An ellipsoid plot of the title compound, with the atom numbering scheme. Atoms with primed labels are related via an inversion center ($1-x, 1-y, 1-z$). Displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

A packing diagram, viewed along *b*.

3,3'-Dimethyl-4,4'-(hexane-1,6-diyl)bis[1*H*-1,2,4-triazol- 5(4*H*)-one]*Crystal data*C₁₂H₂₀N₆O₂ $M_r = 280.34$ Triclinic, $P\bar{1}$

Hall symbol: -P 1

 $a = 6.3641 (2) \text{ \AA}$ $b = 7.3034 (2) \text{ \AA}$ $c = 7.7774 (2) \text{ \AA}$ $\alpha = 93.299 (2)^\circ$ $\beta = 109.578 (2)^\circ$ $\gamma = 94.707 (2)^\circ$ $V = 338.05 (2) \text{ \AA}^3$ $Z = 1$ $F(000) = 150$ $D_x = 1.377 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1309 reflections

 $\theta = 2.8\text{--}28.3^\circ$ $\mu = 0.10 \text{ mm}^{-1}$ $T = 101 \text{ K}$

Rod-shaped, colorless

 $0.40 \times 0.16 \times 0.12 \text{ mm}$ *Data collection*Bruker Kappa APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2007)

 $T_{\min} = 0.962$, $T_{\max} = 0.988$

6074 measured reflections

1673 independent reflections

1309 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.033$ $\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 2.8^\circ$ $h = -8 \rightarrow 8$ $k = -9 \rightarrow 9$ $l = -10 \rightarrow 10$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.041$ $wR(F^2) = 0.112$ $S = 1.03$

1673 reflections

131 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

All H-atom parameters refined

 $w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.0594P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} < 0.001$ $\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$ $\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$ *Special details*

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.49741 (15)	0.02539 (13)	0.76514 (13)	0.0199 (3)
N1	0.84928 (18)	0.18555 (15)	0.81036 (15)	0.0159 (3)
N2	0.98018 (18)	0.22436 (15)	1.11442 (15)	0.0183 (3)

N3	0.76344 (18)	0.13460 (15)	1.04956 (15)	0.0173 (3)
C1	0.6815 (2)	0.10507 (17)	0.86549 (18)	0.0162 (3)
C2	0.8354 (2)	0.1878 (2)	0.61861 (18)	0.0189 (3)
C3	0.7642 (2)	0.36813 (19)	0.54057 (19)	0.0202 (3)
C4	0.5280 (2)	0.40487 (19)	0.53108 (19)	0.0193 (3)
C5	1.0248 (2)	0.25341 (17)	0.96590 (18)	0.0166 (3)
C6	1.2408 (2)	0.3456 (2)	0.9639 (2)	0.0207 (3)
H21	0.981 (3)	0.163 (2)	0.609 (2)	0.019 (4)*
H61	1.220 (3)	0.461 (3)	0.914 (2)	0.030 (4)*
H32	0.772 (3)	0.361 (2)	0.415 (2)	0.025 (4)*
H41	0.512 (3)	0.397 (2)	0.655 (2)	0.018 (4)*
H31	0.876 (3)	0.476 (2)	0.614 (2)	0.028 (4)*
H42	0.414 (3)	0.309 (2)	0.446 (2)	0.022 (4)*
H22	0.732 (3)	0.087 (2)	0.551 (2)	0.025 (4)*
H3	0.696 (3)	0.089 (2)	1.125 (3)	0.036 (5)*
H62	1.310 (3)	0.273 (3)	0.887 (3)	0.035 (5)*
H63	1.348 (3)	0.368 (3)	1.093 (3)	0.041 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0140 (5)	0.0240 (5)	0.0184 (5)	-0.0042 (4)	0.0030 (4)	0.0004 (4)
N1	0.0126 (6)	0.0162 (6)	0.0181 (6)	0.0005 (4)	0.0043 (5)	0.0024 (4)
N2	0.0123 (6)	0.0186 (6)	0.0213 (6)	-0.0005 (4)	0.0028 (5)	0.0012 (4)
N3	0.0127 (6)	0.0192 (6)	0.0183 (6)	-0.0006 (4)	0.0035 (5)	0.0026 (4)
C1	0.0134 (6)	0.0150 (6)	0.0197 (7)	0.0028 (5)	0.0047 (5)	0.0025 (5)
C2	0.0156 (7)	0.0230 (7)	0.0169 (7)	-0.0009 (6)	0.0049 (5)	-0.0001 (5)
C3	0.0163 (7)	0.0237 (7)	0.0202 (7)	-0.0017 (6)	0.0065 (6)	0.0039 (6)
C4	0.0160 (7)	0.0213 (7)	0.0184 (7)	-0.0029 (5)	0.0039 (6)	0.0035 (5)
C5	0.0130 (6)	0.0153 (6)	0.0198 (7)	0.0033 (5)	0.0028 (5)	0.0018 (5)
C6	0.0131 (7)	0.0208 (7)	0.0261 (8)	-0.0005 (5)	0.0045 (6)	0.0021 (6)

Geometric parameters (Å, °)

O1—C1	1.2421 (16)	C3—C4	1.5271 (19)
N1—C5	1.3751 (17)	C3—H32	0.991 (16)
N1—C1	1.3794 (16)	C3—H31	1.017 (18)
N1—C2	1.4653 (16)	C4—C4 ⁱ	1.528 (3)
N2—C5	1.3031 (17)	C4—H41	1.007 (15)
N2—N3	1.3907 (15)	C4—H42	1.000 (17)
N3—C1	1.3467 (18)	C5—C6	1.4856 (19)
N3—H3	0.902 (19)	C6—H61	0.952 (18)
C2—C3	1.521 (2)	C6—H62	1.006 (18)
C2—H21	0.985 (15)	C6—H63	1.00 (2)
C2—H22	0.945 (18)		
C5—N1—C1	107.39 (11)	C2—C3—H31	110.3 (10)
C5—N1—C2	128.60 (11)	C4—C3—H31	109.3 (9)

C1—N1—C2	123.98 (11)	H32—C3—H31	107.2 (13)
C5—N2—N3	103.79 (11)	C3—C4—C4 ⁱ	112.27 (14)
C1—N3—N2	112.63 (11)	C3—C4—H41	110.2 (9)
C1—N3—H3	124.9 (12)	C4 ⁱ —C4—H41	108.2 (8)
N2—N3—H3	122.0 (12)	C3—C4—H42	110.5 (9)
O1—C1—N3	128.98 (12)	C4 ⁱ —C4—H42	109.0 (9)
O1—C1—N1	126.86 (12)	H41—C4—H42	106.5 (13)
N3—C1—N1	104.16 (11)	N2—C5—N1	111.99 (11)
N1—C2—C3	112.31 (11)	N2—C5—C6	124.26 (13)
N1—C2—H21	108.9 (9)	N1—C5—C6	123.74 (12)
C3—C2—H21	111.3 (9)	C5—C6—H61	110.6 (10)
N1—C2—H22	107.6 (10)	C5—C6—H62	113.5 (10)
C3—C2—H22	110.7 (10)	H61—C6—H62	105.9 (14)
H21—C2—H22	105.8 (13)	C5—C6—H63	108.9 (11)
C2—C3—C4	114.14 (11)	H61—C6—H63	108.3 (15)
C2—C3—H32	106.5 (9)	H62—C6—H63	109.6 (14)
C4—C3—H32	109.1 (9)		
C5—N2—N3—C1	1.87 (14)	N1—C2—C3—C4	-64.04 (15)
N2—N3—C1—O1	178.11 (12)	C2—C3—C4—C4 ⁱ	174.64 (14)
N2—N3—C1—N1	-2.33 (14)	N3—N2—C5—N1	-0.58 (14)
C5—N1—C1—O1	-178.57 (12)	N3—N2—C5—C6	-179.47 (12)
C2—N1—C1—O1	-0.5 (2)	C1—N1—C5—N2	-0.81 (15)
C5—N1—C1—N3	1.86 (13)	C2—N1—C5—N2	-178.77 (12)
C2—N1—C1—N3	179.93 (11)	C1—N1—C5—C6	178.09 (12)
C5—N1—C2—C3	-84.62 (16)	C2—N1—C5—C6	0.1 (2)
C1—N1—C2—C3	97.73 (14)		

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3 \cdots O1 ⁱⁱ	0.90 (2)	1.89 (2)	2.7707 (15)	167 (2)

Symmetry code: (ii) $-x+1, -y, -z+2$.