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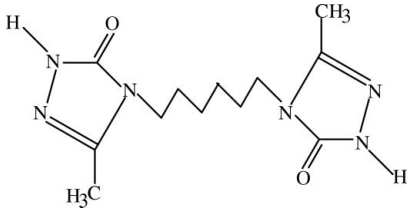
Key indicators: single-crystal X-ray study; $T$ = 101 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.041; $wR$ factor = 0.112; data-to-parameter ratio = 12.8.

The title compound, $C_{12}H_{20}N_6O_2$, has a centre of symmetry. The molecule consists of two triazole rings joined by an aliphatic $-(CH_2)_6-$ chain. The crystal structure is stabilized by intermolecular N—H···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); Eliott *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*

$C_{12}H_{20}N_6O_2$
$M_r$ = 280.34
Triclinic, $P\overline{1}$
$a$ = 6.3641 (2) Å
$b$ = 7.3034 (2) Å
$c$ = 7.7774 (2) Å
$\alpha$ = 93.299 (2)°
$\beta$ = 109.578 (2)°
$\gamma$ = 94.707 (2)°
$V$ = 338.05 (2) Å$^3$
$Z$ = 1
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 101 K
0.40 × 0.16 × 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 $I > 2\sigma(I)$
$R_{int}$ = 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
$\Delta\rho_{max}$ = 0.32 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H3···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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# supplementary materials

*Acta Cryst.* (2010). E**66**, o2615 [ **doi:10.1107/S1600536810037311** ]

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

**R. Ustabas, U. Çoruh, D. Ünlüer, T. Hökelek and E. Ermis**

## 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 (Eliott *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 $C_{14}H_{16}N_6O_2S$ (Ustabaş *et al.*, 2007); 1.288 (2)Å in $C_{16}H_{28}N_6O_2$ (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 $C_{16}H_{20}N_6O_2S$ (Ustabaş *et al.*, 2006); 1.220 (2)Å in $C_{24}H_{20}N_4O_2S$ (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 (*Cg*1···*Cg*1= 3.277 (8) Å; symmetry code: –*X*, 2-Y, –*Z*).

## 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.

## 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].

## Figures

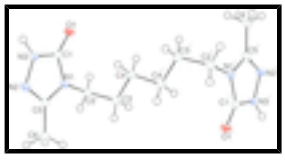

Fig. 1. An ellipsoid plot of the title compound, with the atom numbering scheme. Atoms with primed labels are related via an inversion center (1-x, 1-y, 1-z). Displacement ellipsoids are drawn at the 50% probability level.

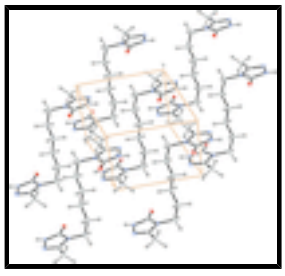

Fig. 2. A packing diagram, viewed along b.

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

*Crystal data*

| | |
|---|---|
| $C_{12}H_{20}N_6O_2$ | $Z = 1$ |
| $M_r = 280.34$ | $F(000) = 150$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.377$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 6.3641$ (2) Å | Cell parameters from 1309 reflections |
| $b = 7.3034$ (2) Å | $\theta = 2.8$–$28.3°$ |
| $c = 7.7774$ (2) Å | $\mu = 0.10$ mm$^{-1}$ |
| $\alpha = 93.299$ (2)° | $T = 101$ K |
| $\beta = 109.578$ (2)° | Rod-shaped, colorless |
| $\gamma = 94.707$ (2)° | 0.40 × 0.16 × 0.12 mm |
| $V = 338.05$ (2) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker Kappa APEXII CCD area-detector diffractometer | 1673 independent reflections |
| Radiation source: fine-focus sealed tube | 1309 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.033$ |
| φ and ω scans | $\theta_{max} = 28.3°$, $\theta_{min} = 2.8°$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2007) | $h = -8 \rightarrow 8$ |
| $T_{min} = 0.962$, $T_{max} = 0.988$ | $k = -9 \rightarrow 9$ |
| 6074 measured reflections | $l = -10 \rightarrow 10$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |

| | |
|---|---|
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.041$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.112$ | All H-atom parameters refined |
| $S = 1.03$ | $w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.0594P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 1673 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 131 parameters | $\Delta\rho_{max} = 0.32$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.28$ e Å$^{-3}$ |

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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 codes: (i) −*x*+1, −*y*+1, −*z*+1.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H3···O1$^{ii}$ | 0.90 (2) | 1.89 (2) | 2.7707 (15) | 167 (2) |

Symmetry codes: (ii) −*x*+1, −*y*, −*z*+2.

**Fig. 1**

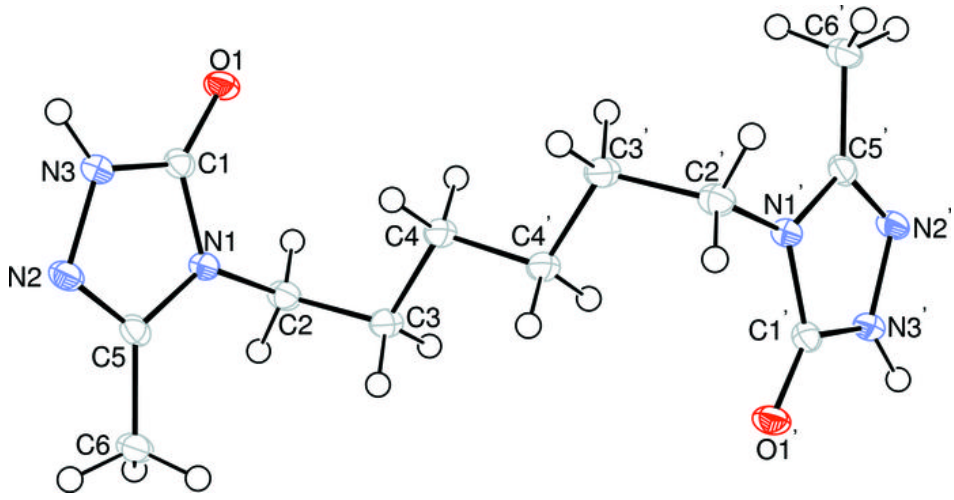

**Fig. 2**