Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

5,5′-(Butane-1,4-diyl)bis(1*H*-tetrazole) dihydrate

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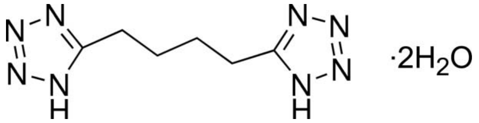
Received 22 November 2010; accepted 23 November 2010

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.147; data-to-parameter ratio = 13.6.

The title compound, $C_6H_{10}N_8\cdot 2H_2O$, was prepared by the reaction of hexanedinitrile and sodium azide. The di-1*H*-tetrazole molecule lies on a crystallographic centre of inversion and is linked to the water molecules by N—H···O and O—H···N hydrogen bonds, forming a two-dimensional supramolecular structure in the crystal.

Related literature

For tetrazole derivatives, see: Demko & Sharpless (2001); Diop *et al.* (2002); Kitagawa *et al.* (2004); Li *et al.* (2007); Tamura *et al.* (1998); Tong *et al.* (2009); Zhao *et al.* (2008).



Experimental

Crystal data

$C_6H_{10}N_8\cdot 2H_2O$
M_r = 230.25
Monoclinic, $C2/c$
a = 6.994 (3) Å
b = 11.590 (5) Å
c = 14.097 (6) Å
β = 100.716 (7)°
V = 1122.8 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 K
0.20 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.979, T_{max} = 0.983
2756 measured reflections
992 independent reflections
722 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.147
S = 1.04
992 reflections
73 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1WB···N3^{i}	0.85	2.02	2.851 (3)	165
O1W—H1WA···N4^{ii}	0.85	1.99	2.822 (3)	167
N1—H1···O1W	0.86	1.80	2.662 (3)	175

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL97*.

This work was was supported by the Postgraduate Foundation of East China of Technology (No·Y09–11–02)

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5418).

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supplementary materials

Acta Cryst. (2010). E**66**, o3328 [doi:10.1107/S1600536810048993]

5,5'-(Butane-1,4-diyl)bis(1*H*-tetrazole) dihydrate

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Comment

The tetrazole derivatives are very important molecules in pharmacological and biochemical properties (Tamura *et al.*, 1998). Since Sharpless *et al.* have introduced a simple and effective method to synthesize the tetrazole derivatives (Demko *et al.*, 2001), they have been used extensively in areas as diverse as medicinal chemistry, coordination chemistry and material chemistry (Zhao *et al.*, 2008; Kitagawa *et al.*, 2004; Li *et al.*, 2007). Among these, The flexible 5-substituted tetrazolate ligands have been less investigated (Diop *et al.*, 2002), although we have studied the coordination of the bis(tetrazole) ligands separated by alkyl $(CH_2)_n$ spacers (Tong *et al.*, 2009). Here, as the additional of our work, we report the crystal structure of the title compound (Fig. 1).

1,2-Bis(tetrazol-5-yl)butpane lies on a crystallographic centre of inversion and is linked to the water molecules by N—H···O and O—H···N hydrogen bonds into a 2-D supramolecular structure (Fig. 2).

Experimental

1,2-Bis(tetrazol-5-yl)butane was prepared using a reported procedure (Tong *et al.*, 2009) (Scheme I). 1,2-Bis(tetrazol-5-yl)butane and water (12 ml) was sealed in a 25 ml Teflon-lined stainless steel vessel and heated at 393 k for 72 hr., then cooled to room temperature. Colorless prism-shaped crystals of the title compound were isolated and washed with water and ethanol and dried in air.

Refinement

All H atoms were placed in idealized positions (O—H = 0.85 Å, N—H = 0.86 Å and C—H = 0.95 Å) and refined as riding atoms with $U_{iso}(H) = 1.2U_{eq}(C, N)$ and $U_{iso}(H) = 1.5U_{eq}(O)$.

Figures

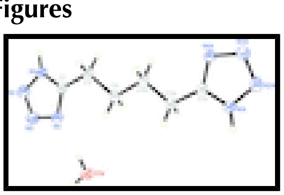

Fig. 1. The asymmetric unit of the title compound, (I), with displacement ellipsoids drawn at the 30% probability level.

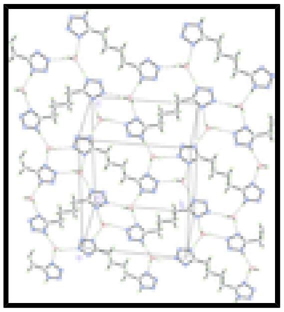

Fig. 2. The packing diagram of the title compound. Hydrogen bonds are shown as dashed line.

5,5'-(Butane-1,4-diyl)bis(1*H*-tetrazole) dihydrate

Crystal data

$C_6H_{10}N_8·2H_2O$	$F(000) = 488$
$M_r = 230.25$	$D_x = 1.362$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 1178 reflections
$a = 6.994$ (3) Å	$\theta = 2.9–25.0°$
$b = 11.590$ (5) Å	$\mu = 0.11$ mm^{-1}
$c = 14.097$ (6) Å	$T = 294$ K
$\beta = 100.716$ (7)°	Block, colorless
$V = 1122.8$ (8) Å^3	0.20 × 0.18 × 0.16 mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	992 independent reflections
Radiation source: fine-focus sealed tube	722 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.025$
φ and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.9°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$h = -7\rightarrow8$
$T_{min} = 0.979$, $T_{max} = 0.983$	$k = -13\rightarrow10$
2756 measured reflections	$l = -16\rightarrow15$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.052$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.147$	H-atom parameters constrained
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0759P)^2 + 0.9248P]$ where $P = (F_o^2 + 2F_c^2)/3$
992 reflections	$(\Delta/\sigma)_{max} < 0.001$

73 parameters	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.21 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}
O1W	0.2175 (3)	0.10913 (15)	0.88483 (13)	0.0697 (7)
H1WA	0.2503	0.0847	0.9424	0.105*
H1WB	0.2153	0.1823	0.8886	0.105*
N1	0.2521 (3)	−0.00531 (16)	0.72515 (14)	0.0487 (6)
H1	0.2430	0.0355	0.7751	0.058*
N2	0.2517 (4)	−0.11987 (17)	0.72219 (16)	0.0607 (7)
N3	0.2679 (4)	−0.14652 (18)	0.63566 (16)	0.0624 (7)
N4	0.2787 (4)	−0.05060 (17)	0.58246 (14)	0.0536 (7)
C1	0.2683 (4)	0.0365 (2)	0.63995 (16)	0.0422 (6)
C3	0.2750 (4)	0.1611 (2)	0.61690 (17)	0.0505 (7)
H3A	0.3991	0.1922	0.6488	0.061*
H3B	0.1737	0.2006	0.6428	0.061*
C4	0.2488 (4)	0.1863 (2)	0.50977 (17)	0.0463 (7)
H4A	0.3525	0.1493	0.4838	0.056*
H4B	0.1261	0.1541	0.4771	0.056*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1W	0.131 (2)	0.0449 (11)	0.0368 (11)	0.0010 (11)	0.0256 (11)	−0.0017 (8)
N1	0.0817 (16)	0.0361 (12)	0.0306 (11)	−0.0005 (10)	0.0162 (10)	0.0006 (9)
N2	0.101 (2)	0.0406 (13)	0.0419 (13)	−0.0030 (12)	0.0169 (12)	0.0072 (10)
N3	0.108 (2)	0.0365 (12)	0.0451 (14)	−0.0027 (12)	0.0196 (13)	0.0011 (10)
N4	0.0960 (18)	0.0327 (11)	0.0344 (11)	−0.0022 (11)	0.0181 (11)	0.0010 (9)
C1	0.0603 (16)	0.0364 (12)	0.0306 (12)	−0.0024 (11)	0.0103 (10)	0.0003 (10)
C3	0.082 (2)	0.0344 (13)	0.0373 (14)	−0.0022 (12)	0.0157 (12)	−0.0011 (11)
C4	0.0661 (16)	0.0366 (13)	0.0373 (13)	−0.0007 (12)	0.0120 (11)	0.0020 (10)

Geometric parameters (Å, °)

O1W—H1WA	0.8500	C1—C3	1.482 (3)
O1W—H1WB	0.8500	C3—C4	1.516 (3)
N1—C1	1.320 (3)	C3—H3A	0.9700
N1—N2	1.328 (3)	C3—H3B	0.9700
N1—H1	0.8600	C4—C4^{i}	1.503 (5)
N2—N3	1.284 (3)	C4—H4A	0.9700
N3—N4	1.351 (3)	C4—H4B	0.9700
N4—C1	1.305 (3)		
H1WA—O1W—H1WB	106.1	C1—C3—H3A	108.8
C1—N1—N2	109.8 (2)	C4—C3—H3A	108.8
C1—N1—H1	125.1	C1—C3—H3B	108.8
N2—N1—H1	125.1	C4—C3—H3B	108.8
N3—N2—N1	105.69 (19)	H3A—C3—H3B	107.7
N2—N3—N4	110.7 (2)	C4^{i}—C4—C3	111.7 (3)
C1—N4—N3	106.1 (2)	C4^{i}—C4—H4A	109.3
N4—C1—N1	107.7 (2)	C3—C4—H4A	109.3
N4—C1—C3	127.6 (2)	C4^{i}—C4—H4B	109.3
N1—C1—C3	124.7 (2)	C3—C4—H4B	109.3
C1—C3—C4	113.8 (2)	H4A—C4—H4B	108.0
C1—N1—N2—N3	0.1 (3)	N2—N1—C1—N4	−0.1 (3)
N1—N2—N3—N4	0.0 (3)	N2—N1—C1—C3	−179.6 (2)
N2—N3—N4—C1	0.0 (3)	N4—C1—C3—C4	13.6 (4)
N3—N4—C1—N1	0.1 (3)	N1—C1—C3—C4	−167.1 (3)
N3—N4—C1—C3	179.5 (2)	C1—C3—C4—C4^{i}	178.4 (3)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1WB···N3^{ii}	0.85	2.02	2.851 (3)	165
O1W—H1WA···N4^{iii}	0.85	1.99	2.822 (3)	167
N1—H1···O1W	0.86	1.80	2.662 (3)	175

Symmetry codes: (ii) $-x+1/2, y+1/2, -z+3/2$; (iii) $x, -y, z+1/2$.

Fig. 1

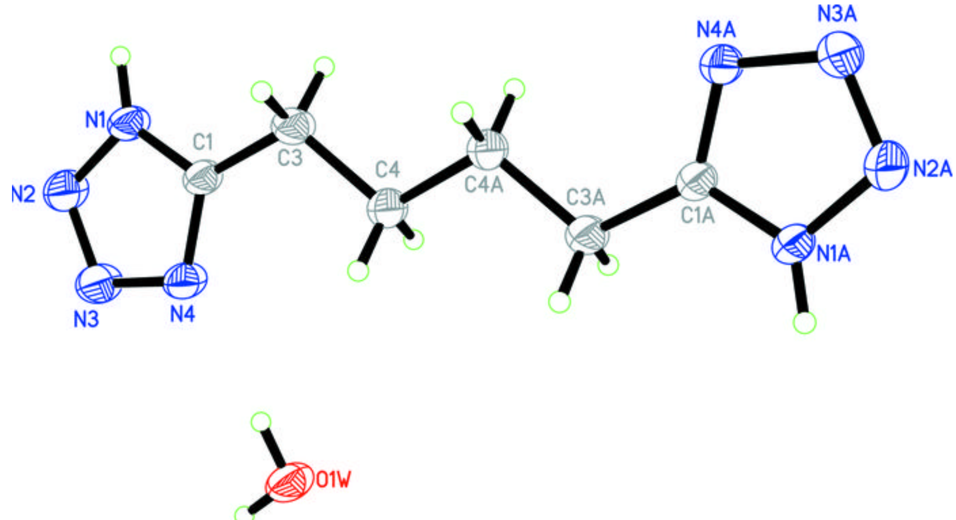

Fig. 2