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1,3-Bis[5-(2-pyridyl)-1*H*-tetrazol-1-yl]-propane

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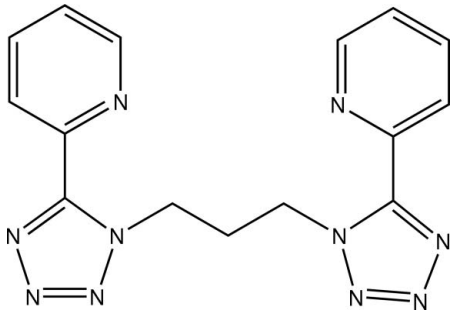
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.112; data-to-parameter ratio = 16.0.

The title compound, $C_{15}H_{14}N_{10}$, is a multidentate ligand obtained by the reaction of 5-(2-pyridyl)tetrazole with 1,3-dibromopropane. The molecule consists of two 5-(2-pyridyl)-1*H*-tetrazol-1-yl units connected by a propylene bridge in a U-like conformation. A twofold rotation axis passes through the central C atom.

Related literature

For related literature, see: Bronisz (2002); Gallardo *et al.* (2004); Meyer *et al.* (1998).



Experimental

Crystal data

$C_{15}H_{14}N_{10}$
M_r = 334.36
Monoclinic, $C2/c$
a = 14.486 (2) Å
b = 9.1322 (13) Å
c = 12.8032 (19) Å
β = 111.596 (2)°
V = 1574.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.2 × 0.2 × 0.2 mm

Data collection

Rigaku Scxmini 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.981, T_{max} = 0.981
4930 measured reflections
1845 independent reflections
1166 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.112
S = 1.02
1845 reflections
115 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2397).

References

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supporting information

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S1. Comment

The tetrazolate anion is an ambident system in which alkylation can occur at the N-1 or N-2 position, the relative proportions of which depend upon the reaction conditions, the nature of the alkylating agent and the influence of the 5-substituent (Meyer *et al.*, 1998; Bronisz, 2002). The crystal structure of one of the three regioisomers has already been published (Gallardo *et al.*, 2004). In our case, no regioselectivity was observed and the three possible regioisomers were isolated in equal amounts.

The structure of the title compound (I) is similar to that observed in the paper (Gallardo *et al.*, 2004) with bond lengths and angles in good agreement with expected values. The molecules of (I) are disposed about a crystallographic two-fold axis of symmetry with symmetrical 5-(2-pyridyl)- 2*H*-tetrazolyl units connected by a propylene bridge. The molecule is folded at the center of the bridge [C7-C8-C7^{i} 116.1 (2)°; symmetry code (i) = (-x+1,y,-z+1/2)] giving a U-like conformation to the free ligand. The inter-ring dihedral angle Py/Tz is 12.00 (7)°.

S2. Experimental

5-(2-Pyridyl)tetrazole, (3.0 g, 20.0 mmol) was dissolved in 25 ml of 2-butanone with stirring and to the solution 1,3-di-bromopropane (2.0 g, 10.0 mmol) and K_2CO_3 (5.5 g, 40.0 mmol) were added. The reaction mixture was heated under reflux for 24 h. After cooling the inorganic materials were filtered off and the solvent was removed under reduced pressure to afford the mixture of isomers. These isomers were separated by column chromatography on silica gel (1:4–2:1 EtOAc/Petroleum ether(60-90°C). The pure compound (I) (334 mg, 1.0 mmol) was dissolved in the solvent (1:1 EtOAc/Petroleum ether), and recrystallized from EtOAc/Petroleum ether affording colorless crystals. .

S3. Refinement

Positional parameters of all H atoms were calculated geometrically and were allowed to ride on the C atoms to which they are bonded, with C-H distances in the range 0.93-0.97Å and U_{iso}(H) = 1.2U_{eq}(C).

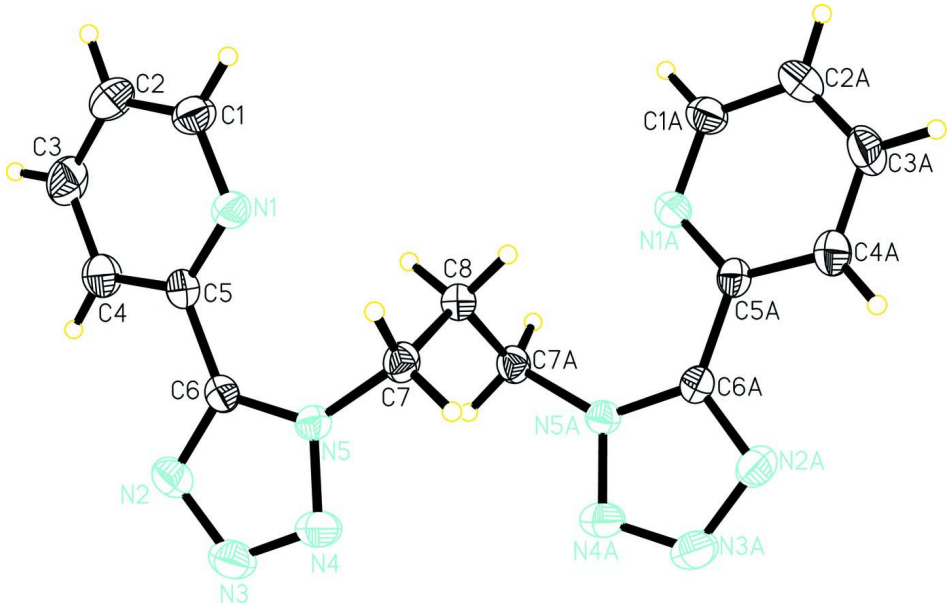

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level (Symmetry code(A): (-x+1,y,-z+1/2)

1,3-Bis[5-(2-pyridyl)-1*H*-tetrazol-1-yl]propane

Crystal data

$C_{15}H_{14}N_{10}$
$M_r = 334.36$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 14.486\ (2)$ Å
$b = 9.1322\ (13)$ Å
$c = 12.8032\ (19)$ Å
$\beta = 111.596\ (2)°$
$V = 1574.8\ (4)$ Å^3
$Z = 4$
$F(000) = 696$
$D_x = 1.410$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 2.7–28.3°$
$\mu = 0.10$ mm^{-1}
$T = 298$ K
Block, colourless
0.2 × 0.2 × 0.2 mm

Data collection

Rigaku Scxmini 1K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.192 pixels mm^{-1}
thin–slice ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.981$, $T_{max} = 0.981$
4930 measured reflections
1845 independent reflections
1166 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.7°$
$h = -15\rightarrow18$
$k = -7\rightarrow12$
$l = -16\rightarrow16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.112$
$S = 1.02$
1845 reflections
115 parameters
0 restraints

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2 + 0.113P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0018 (5)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C2	0.37119 (12)	0.2927 (2)	−0.15496 (13)	0.0542 (5)	
H2A	0.3811	0.2170	−0.1982	0.065*	
N2	0.39676 (9)	0.07129 (14)	0.09971 (10)	0.0372 (3)	
N3	0.40148 (11)	−0.07629 (15)	0.10268 (13)	0.0520 (4)	
N4	0.38893 (11)	−0.11910 (16)	0.00162 (14)	0.0592 (4)	
N5	0.37593 (11)	−0.00222 (17)	−0.06788 (12)	0.0527 (4)	
C1	0.36961 (10)	0.26606 (18)	−0.04949 (12)	0.0388 (4)	
N1	0.35696 (10)	0.37150 (15)	0.01654 (10)	0.0447 (4)	
C3	0.34557 (13)	0.50772 (19)	−0.02391 (15)	0.0517 (5)	
H3B	0.3380	0.5826	0.0215	0.062*	
C4	0.34441 (14)	0.5440 (2)	−0.12819 (15)	0.0579 (5)	
H4B	0.3349	0.6404	−0.1533	0.069*	
C5	0.35767 (15)	0.4344 (2)	−0.19443 (15)	0.0642 (6)	
H5B	0.3575	0.4556	−0.2655	0.077*	
C6	0.38094 (11)	0.11518 (17)	−0.00589 (12)	0.0382 (4)	
C7	0.40529 (11)	0.15178 (18)	0.20192 (12)	0.0411 (4)	
H7A	0.4022	0.0827	0.2581	0.049*	
H7B	0.3491	0.2177	0.1851	0.049*	
C8	0.5000	0.2392 (2)	0.2500	0.0418 (5)	
H8B	0.4950	0.3021	0.3087	0.063*	0.50
H8A	0.5050	0.3021	0.1913	0.063*	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0685 (12)	0.0581 (13)	0.0403 (9)	0.0009 (9)	0.0250 (8)	−0.0008 (9)
N2	0.0384 (7)	0.0343 (8)	0.0384 (7)	−0.0012 (6)	0.0136 (5)	0.0013 (6)
N3	0.0556 (9)	0.0364 (9)	0.0627 (10)	−0.0013 (7)	0.0203 (7)	0.0003 (7)

N4	0.0665 (10)	0.0428 (9)	0.0669 (11)	−0.0018 (7)	0.0228 (8)	−0.0092 (8)
N5	0.0594 (9)	0.0484 (9)	0.0490 (9)	−0.0014 (7)	0.0188 (7)	−0.0109 (7)
C1	0.0354 (8)	0.0455 (10)	0.0342 (8)	−0.0019 (7)	0.0112 (6)	−0.0013 (7)
N1	0.0554 (8)	0.0395 (8)	0.0404 (8)	0.0038 (6)	0.0192 (6)	0.0028 (6)
C3	0.0619 (11)	0.0418 (10)	0.0533 (10)	0.0044 (8)	0.0234 (9)	0.0059 (9)
C4	0.0646 (12)	0.0524 (12)	0.0590 (11)	0.0033 (9)	0.0256 (9)	0.0179 (10)
C5	0.0799 (14)	0.0731 (15)	0.0431 (10)	−0.0029 (11)	0.0269 (9)	0.0142 (10)
C6	0.0369 (8)	0.0412 (9)	0.0353 (8)	−0.0003 (7)	0.0119 (6)	−0.0043 (7)
C7	0.0465 (9)	0.0453 (10)	0.0342 (8)	0.0013 (7)	0.0180 (7)	0.0021 (7)
C8	0.0472 (13)	0.0396 (13)	0.0357 (11)	0.000	0.0120 (9)	0.000

Geometric parameters (Å, º)

C2—C5	1.377 (2)	C3—C4	1.370 (2)
C2—C1	1.381 (2)	C3—H3B	0.9300
C2—H2A	0.9300	C4—C5	1.370 (3)
N2—N3	1.3493 (18)	C4—H4B	0.9300
N2—C6	1.3464 (18)	C5—H5B	0.9300
N2—C7	1.4661 (19)	C7—C8	1.5091 (18)
N3—N4	1.2988 (19)	C7—H7A	0.9700
N4—N5	1.358 (2)	C7—H7B	0.9700
N5—C6	1.3199 (19)	C8—C7^{i}	1.5091 (18)
C1—N1	1.3378 (19)	C8—H8B	0.9700
C1—C6	1.473 (2)	C8—H8A	0.9700
N1—C3	1.334 (2)		
C5—C2—C1	118.27 (16)	C4—C5—C2	119.51 (16)
C5—C2—H2A	120.9	C4—C5—H5B	120.2
C1—C2—H2A	120.9	C2—C5—H5B	120.2
N3—N2—C6	108.29 (13)	N5—C6—N2	108.26 (14)
N3—N2—C7	119.27 (12)	N5—C6—C1	123.92 (14)
C6—N2—C7	132.41 (13)	N2—C6—C1	127.81 (14)
N4—N3—N2	106.59 (13)	N2—C7—C8	113.25 (11)
N3—N4—N5	110.55 (14)	N2—C7—H7A	108.9
C6—N5—N4	106.31 (13)	C8—C7—H7A	108.9
N1—C1—C2	123.13 (15)	N2—C7—H7B	108.9
N1—C1—C6	117.12 (13)	C8—C7—H7B	108.9
C2—C1—C6	119.75 (15)	H7A—C7—H7B	107.7
C3—N1—C1	116.87 (14)	C7—C8—C7^{i}	116.09 (19)
N1—C3—C4	124.01 (17)	C7—C8—H8B	108.3
N1—C3—H3B	118.0	C7^{i}—C8—H8B	108.3
C4—C3—H3B	118.0	C7—C8—H8A	108.3
C5—C4—C3	118.19 (17)	C7^{i}—C8—H8A	108.3
C5—C4—H4B	120.9	H8B—C8—H8A	107.4
C3—C4—H4B	120.9		
C6—N2—N3—N4	0.07 (16)	N4—N5—C6—C1	−179.43 (14)
C7—N2—N3—N4	178.05 (12)	N3—N2—C6—N5	−0.06 (16)

N2—N3—N4—N5	−0.06 (17)	C7—N2—C6—N5	−177.67 (14)
N3—N4—N5—C6	0.02 (18)	N3—N2—C6—C1	179.36 (14)
C5—C2—C1—N1	−0.9 (2)	C7—N2—C6—C1	1.8 (2)
C5—C2—C1—C6	178.24 (15)	N1—C1—C6—N5	167.21 (14)
C2—C1—N1—C3	0.0 (2)	C2—C1—C6—N5	−12.0 (2)
C6—C1—N1—C3	−179.23 (13)	N1—C1—C6—N2	−12.1 (2)
C1—N1—C3—C4	1.2 (2)	C2—C1—C6—N2	168.64 (14)
N1—C3—C4—C5	−1.3 (3)	N3—N2—C7—C8	111.61 (15)
C3—C4—C5—C2	0.3 (3)	C6—N2—C7—C8	−71.0 (2)
C1—C2—C5—C4	0.8 (3)	N2—C7—C8—C7^{i}	−67.96 (10)
N4—N5—C6—N2	0.02 (17)		

Symmetry code: (i) −*x*+1, *y*, −*z*+1/2.