Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(1,2,3,4-tetrahydroquinolin-6-yl)-methane

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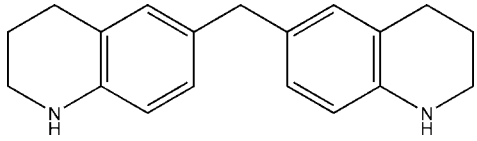
Received 2 August 2008; accepted 7 August 2008

Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.004 Å; R factor = 0.048; wR factor = 0.138; data-to-parameter ratio = 8.1.

The asymmetric unit of the title compound, $C_{19}H_{22}N_2$, contains one half-molecule. The 1,2,3,4-tetrahydroquinoline units are linked by a methylene bridge, which lies on a twofold rotation axis. The non-aromatic ring adopts a flattened-boat conformation. The dihedral angle between the two symmetry-related benzene rings is 64.03 (7)°.

Related literature

For general background, see: Xiao *et al.* (2008*a*,*b*,*c*); Xiao *et al.* (2007*a*,*b*); Xue *et al.* (2007). For ring conformation puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{19}H_{22}N_2$
M_r = 278.39
Orthorhombic, *Fdd*2
a = 17.515 (3) Å
b = 29.660 (4) Å
c = 5.7678 (8) Å
V = 2996.2 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 292 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.979, T_{max} = 0.986
4545 measured reflections
814 independent reflections
773 reflections with $I > 2\sigma(I)$
R_{int} = 0.073

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.138
S = 1.07
814 reflections
100 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

The work was financed by the Natural Science Foundation of Jiangsu Province (grant No. BK2007081).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2506).

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supporting information

Acta Cryst. (2008). E**64**, o1747 [doi:10.1107/S1600536808025464]

Bis(1,2,3,4-tetrahydroquinolin-6-yl)methane

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S1. Comment

Nitrogen heterocyclic compounds show diverse biological activities such as antiproliferative (Xiao *et al.*, 2008*a*,b), antibacterial (Xiao *et al.*, 2007*a*; Xue *et al.*, 2007; Xiao *et al.*, 2008*c*), and urease inhibitory (Xiao *et al.*, 2007*b*) activities. The title compound is a heterocyclic compound, which may be used for screening the biological activities. We report herein its crystal structure.

The asymmetric unit of the title compound (Fig. 1) contains one-half molecule. 1,2,3,4-Tetrahydroquinoline moieties are joined by a methylene bridge. Ring A (N1/C2-C4/C9/C10) adopts flattened-boat [φ = 126.28 (2)°, θ = 26.77 (3)°] conformation, having total puckering amplitude, Q_T, of 0.448 (3) Å (Cremer & Pople, 1975). The dihedral angle between the two symmetry related phenyl rings is 64.03 (7)°.

S2. Experimental

Heating of 4,4′-methylenedibenzenamine (2 g), bis(4-nitrophenyl)methane (0.5 g), H_3AsO_4 (1.5 g), concentrated H_2SO_4 (3 ml), and glycerol (8.6 ml) at 413 K for 5 h, addition of water, removal of resinous matter, making alkalization with NaOH, taking up in ether, dehydration with K_2CO_3, and recrystallization of the residue from alcohol gives diquinolin-6-ylmethane. The resulting product (1 g) was subsequently heated with Sn (5.5 g) and HCl (22 ml, 32%) in a water bath for 8 h, addition of water, precipitation of the Sn as $Sn(OH)_2$ by NaOH, taking up in ether, and drying with K_2CO_3, gives the title compound (yield; 0.9 g), which was recrystallized from petroleum ether-ethyl acetate to give colorless prisms.

S3. Refinement

H1 atom (for bridging CH_2) was located in difference syntheses and refined isotropically [C-H = 0.97 (2) Å and U_{iso}(H) = 0.067 (10) Å^2]. The remaining H atoms were positioned geometrically, with N-H = 0.86 Å (for NH) and C-H = 0.93 and 0.97 Å for aromatic and methylene H, respectively, and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C).

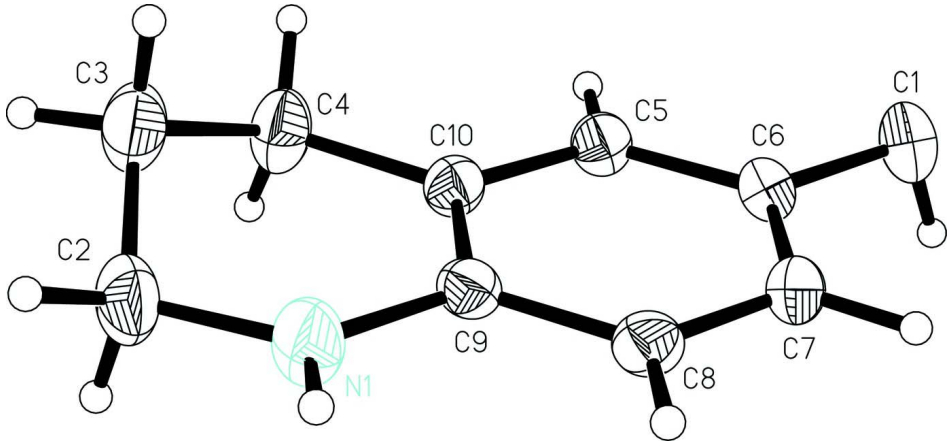

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme [symmetry code: (a) -x, 1 - y, z].

Bis(1,2,3,4-tetrahydroquinolin-6-yl)methane

Crystal data

$C_{19}H_{22}N_2$
$M_r = 278.39$
Orthorhombic, *Fdd*2
Hall symbol: F 2 -2d
$a = 17.515$ (3) Å
$b = 29.660$ (4) Å
$c = 5.7678$ (8) Å
$V = 2996.2$ (8) Å^3
$Z = 8$
$F(000) = 1200$
$D_x = 1.234$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1297 reflections
$\theta = 2.9$–$25.2°$
$\mu = 0.07$ mm^{-1}
$T = 292$ K
Prism, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.979$, $T_{max} = 0.986$
4545 measured reflections
814 independent reflections
773 reflections with $I > 2\sigma(I)$
$R_{int} = 0.073$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.7°$
$h = -21 \rightarrow 21$
$k = -36 \rightarrow 32$
$l = -7 \rightarrow 6$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.138$
$S = 1.08$
814 reflections
100 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0996P)^2 + 0.9957P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	0.26094 (12)	0.44383 (8)	1.0848 (5)	0.0605 (7)
H11A	0.2700	0.4566	1.2159	0.073*
C1	0.0000	0.5000	0.5624 (6)	0.0519 (9)
H1	0.0128 (16)	0.5254 (7)	0.464 (5)	0.067 (10)*
C2	0.31123 (16)	0.40914 (9)	1.0048 (7)	0.0637 (9)
H2A	0.3360	0.3951	1.1368	0.076*
H2B	0.3505	0.4224	0.9080	0.076*
C3	0.27005 (17)	0.37452 (10)	0.8715 (8)	0.0662 (9)
H3A	0.2357	0.3584	0.9740	0.079*
H3B	0.3064	0.3530	0.8094	0.079*
C4	0.22470 (16)	0.39493 (9)	0.6743 (6)	0.0579 (8)
H4A	0.2595	0.4045	0.5530	0.070*
H4B	0.1911	0.3722	0.6097	0.070*
C5	0.11375 (13)	0.44942 (7)	0.6314 (5)	0.0415 (6)
H5	0.1004	0.4345	0.4954	0.050*
C6	0.06877 (12)	0.48523 (8)	0.7038 (5)	0.0407 (6)
C7	0.09023 (12)	0.50718 (8)	0.9058 (5)	0.0434 (6)
H7	0.0615	0.5315	0.9581	0.052*
C8	0.15365 (13)	0.49360 (8)	1.0313 (5)	0.0435 (6)
H8	0.1667	0.5088	1.1667	0.052*
C9	0.19817 (12)	0.45735 (7)	0.9568 (5)	0.0384 (5)
C10	0.17797 (12)	0.43464 (7)	0.7524 (5)	0.0390 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0578 (13)	0.0610 (13)	0.0628 (17)	0.0115 (10)	−0.0273 (12)	−0.0153 (13)
C1	0.0451 (19)	0.068 (2)	0.042 (2)	0.0156 (17)	0.000	0.000
C2	0.0537 (15)	0.0635 (16)	0.074 (2)	0.0170 (11)	−0.0201 (16)	0.0025 (16)
C3	0.0623 (16)	0.0582 (15)	0.078 (2)	0.0198 (12)	−0.0097 (17)	−0.0042 (16)
C4	0.0622 (16)	0.0558 (14)	0.0558 (18)	0.0202 (12)	−0.0111 (15)	−0.0126 (14)
C5	0.0420 (12)	0.0442 (11)	0.0384 (13)	0.0016 (9)	−0.0014 (11)	−0.0069 (10)
C6	0.0324 (10)	0.0460 (10)	0.0437 (13)	0.0024 (8)	0.0016 (10)	0.0000 (11)
C7	0.0383 (11)	0.0416 (11)	0.0504 (15)	0.0045 (9)	0.0061 (10)	−0.0052 (11)
C8	0.0440 (12)	0.0432 (11)	0.0433 (14)	−0.0040 (9)	−0.0018 (11)	−0.0113 (11)
C9	0.0345 (11)	0.0392 (10)	0.0416 (13)	−0.0032 (8)	−0.0034 (10)	0.0013 (10)

C10	0.0398 (11)	0.0375 (10)	0.0398 (12)	0.0008 (8)	−0.0007 (10)	−0.0036 (11)

Geometric parameters (Å, º)

N1—C2	1.431 (3)	C4—H4B	0.9700
N1—H11A	0.8600	C5—C6	1.387 (3)
C1—C6	1.519 (3)	C5—C10	1.394 (3)
C1—C6^{i}	1.519 (3)	C5—H5	0.9300
C1—H1	0.97 (2)	C7—C6	1.386 (4)
C2—C3	1.472 (4)	C7—H7	0.9300
C2—H2A	0.9700	C8—C7	1.386 (3)
C2—H2B	0.9700	C8—H8	0.9300
C3—H3A	0.9700	C9—N1	1.384 (3)
C3—H3B	0.9700	C9—C8	1.396 (3)
C4—C3	1.514 (4)	C9—C10	1.403 (4)
C4—H4A	0.9700	C10—C4	1.503 (3)
C9—N1—C2	121.7 (3)	C10—C4—H4B	109.2
C9—N1—H11A	119.2	C3—C4—H4B	109.2
C2—N1—H11A	119.2	H4A—C4—H4B	107.9
C6—C1—C6^{i}	115.1 (3)	C6—C5—C10	123.3 (2)
C6—C1—H1	110.8 (18)	C6—C5—H5	118.4
C6^{i}—C1—H1	105.9 (19)	C10—C5—H5	118.4
N1—C2—C3	111.6 (2)	C7—C6—C5	117.3 (2)
N1—C2—H2A	109.3	C7—C6—C1	122.0 (2)
C3—C2—H2A	109.3	C5—C6—C1	120.6 (2)
N1—C2—H2B	109.3	C8—C7—C6	121.3 (2)
C3—C2—H2B	109.3	C8—C7—H7	119.3
H2A—C2—H2B	108.0	C6—C7—H7	119.3
C2—C3—C4	111.8 (2)	C7—C8—C9	120.7 (2)
C2—C3—H3A	109.3	C7—C8—H8	119.6
C4—C3—H3A	109.3	C9—C8—H8	119.6
C2—C3—H3B	109.3	N1—C9—C8	120.2 (2)
C4—C3—H3B	109.3	N1—C9—C10	120.6 (2)
H3A—C3—H3B	107.9	C8—C9—C10	119.2 (2)
C10—C4—C3	112.0 (2)	C5—C10—C9	118.2 (2)
C10—C4—H4A	109.2	C5—C10—C4	122.4 (2)
C3—C4—H4A	109.2	C9—C10—C4	119.4 (2)
C9—N1—C2—C3	−33.5 (4)	C9—C8—C7—C6	0.3 (4)
C6^{i}—C1—C6—C7	−39.05 (19)	C8—C9—N1—C2	−175.1 (2)
C6^{i}—C1—C6—C5	142.3 (3)	C10—C9—N1—C2	5.6 (4)
N1—C2—C3—C4	54.2 (4)	N1—C9—C8—C7	−179.4 (2)
C10—C4—C3—C2	−48.1 (4)	C10—C9—C8—C7	0.0 (4)
C10—C5—C6—C7	0.6 (4)	N1—C9—C10—C5	179.4 (2)
C10—C5—C6—C1	179.3 (2)	C8—C9—C10—C5	0.0 (3)
C6—C5—C10—C9	−0.3 (4)	N1—C9—C10—C4	0.7 (4)
C6—C5—C10—C4	178.3 (2)	C8—C9—C10—C4	−178.6 (2)

C8—C7—C6—C5	−0.6 (4)	C5—C10—C4—C3	−157.7 (2)
C8—C7—C6—C1	−179.2 (2)	C9—C10—C4—C3	20.9 (4)

Symmetry code: (i) −*x*, −*y*+1, *z*.