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4-(2-Thienyl)-3,4-dihydrobenzo[*h*]-quinolin-2(1*H*)-one

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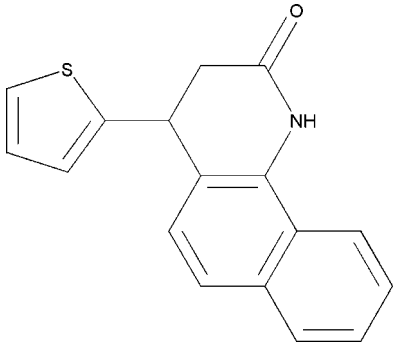
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.073; wR factor = 0.248; data-to-parameter ratio = 12.9.

The title compound, $C_{17}H_{13}NOS$, has been synthesized by the reaction of thiophene-2-carbaldehyde and 2-naphthylamine with Meldrum's acid in ethylene glycol under microwave irradiation. The pyridine ring adopts a twist conformation.

Related literature

For related literature, see: Abd & Hisham (1997); Hewawasam & Starrett (2001); Sit & Meanwell (1999); Sun *et al.* (1997). For bond-length data, see: Allen *et al.* (1987). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{17}H_{13}NOS$
M_r = 279.34
Monoclinic, $P2_1/c$
a = 10.275 (3) Å
b = 14.494 (4) Å
c = 9.829 (3) Å
β = 112.612 (4)°
V = 1351.2 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 298 (2) K
0.45 × 0.40 × 0.29 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.902, T_{max} = 0.936
6785 measured reflections
2339 independent reflections
1332 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.248
S = 1.05
2339 reflections
181 parameters
63 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); 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: HK2388).

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supplementary materials

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4-(2-Thienyl)-3,4-dihydrobenzo[*h*]quinolin-2(1*H*)-one

X. Zhu, S. Tu and D. Zhou

Comment

Various quinolone derivatives are known (Abd & Hisham, 1997; Sun *et al.*, 1997) to display interesting biological properties ranging from microbial activity to cytotoxicity. As a member of the quinolone family, 4-aryl-quinoline-2(1*H*) -ones are modulators of the large-conductance, calcium-activated potassium (Maxi-K and BK) channels and are potentially useful in the treatment of diseases which arise from dysfunction of cellular membrane polarization and conductance (Sit & Meanwell, 1999; Hewawasam & Starrett, 2001). We report herein the crystal structure of the title compound, (I).

In the molecule of (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

Ring A (N1/C1—C4/C13) is not planar, having total puckering amplitude, Q_T, of 0.439 (2) Å [φ = −33.08 (3)°, θ = 115.28 (4)°] (Cremer & Pople, 1975), and adopts twisted conformation. Rings B (C4—C7/C12/C13), C (C7—C12) and D (S1/C14—C17) are, of course, planar and the dihedral angles between them are B/C = 3.20 (3)°, B/D = 84.31 (2)° and C/D = 84.23 (3)°.

Experimental

The title compound, (I), was prepared by the reaction of thiophene-2 -carbaldehyde (112 mg, 1 mmol), 2-naphthylamine (143 mg, 1 mmol) with Meldrum's acid (144 mg, 1 mmol) in the solvent of ethylene glycol (2.0 ml) at 383 K under microwave irradiation (maximum power 250 W, initial power 200 W) for 8 min (yield; 231 mg, 83%, m.p. 511–512 K). Single crystals suitable for X-ray analysis were obtained from an ethanol solution (95%) by slow evaporation.

Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH) and C—H = 0.93, 0.98 and 0.97 Å, for aromatic, methine and methylene H atoms and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C,*N*).

Figures

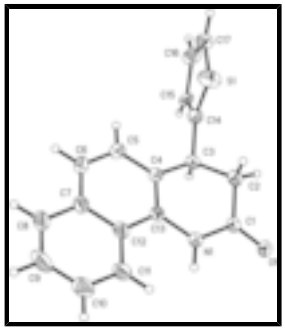

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

4-(2-Thienyl)-3,4-dihydrobenzo[*h*]quinolin-2(1*H*)-one

Crystal data

$C_{17}H_{13}NOS$	F_{000} = 584
M_r = 279.34	D_x = 1.373 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 511-512 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.275 (3) Å	Cell parameters from 1358 reflections
b = 14.494 (4) Å	θ = 2.6–24.3º
c = 9.829 (3) Å	μ = 0.23 mm^{-1}
β = 112.612 (4)º	T = 298 (2) K
V = 1351.2 (7) Å^3	Block, pale yellow
Z = 4	0.45 × 0.40 × 0.29 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2339 independent reflections
Radiation source: fine-focus sealed tube	1332 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.042
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −7→12
T_{min} = 0.902, T_{max} = 0.936	k = −16→17
6785 measured reflections	l = −11→11

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.073	H-atom parameters constrained
$wR(F^2)$ = 0.248	$w = 1/[\sigma^2(F_o^2) + (0.1463P)^2 + 0.208P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
2339 reflections	$\Delta\rho_{max}$ = 0.62 e Å^{-3}
181 parameters	$\Delta\rho_{min}$ = −0.44 e Å^{-3}
63 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	1.07503 (15)	0.14122 (10)	0.40707 (18)	0.0745 (6)
N1	1.4634 (3)	0.3769 (2)	0.5495 (4)	0.0389 (8)
H1	1.5242	0.4200	0.5869	0.047*
O1	1.3269 (3)	0.4762 (2)	0.3784 (3)	0.0516 (9)
C1	1.3463 (4)	0.3981 (3)	0.4327 (5)	0.0385 (10)
C2	1.2399 (5)	0.3229 (3)	0.3745 (5)	0.0474 (11)
H2A	1.1811	0.3359	0.2724	0.057*
H2B	1.1796	0.3218	0.4300	0.057*
C3	1.3090 (4)	0.2279 (3)	0.3846 (5)	0.0372 (10)
H3	1.3574	0.2280	0.3160	0.045*
C4	1.4197 (4)	0.2133 (3)	0.5378 (5)	0.0393 (10)
C5	1.4558 (5)	0.1251 (3)	0.6019 (5)	0.0491 (12)
H5	1.4070	0.0734	0.5517	0.059*
C6	1.5616 (5)	0.1150 (3)	0.7367 (6)	0.0577 (13)
H6	1.5856	0.0560	0.7755	0.069*
C7	1.6355 (4)	0.1907 (3)	0.8188 (5)	0.0464 (11)
C8	1.7424 (5)	0.1807 (4)	0.9609 (6)	0.0642 (15)
H8	1.7692	0.1220	0.9996	0.077*
C9	1.8064 (5)	0.2555 (4)	1.0416 (6)	0.0726 (17)
H9	1.8744	0.2479	1.1362	0.087*
C10	1.7706 (6)	0.3434 (4)	0.9831 (6)	0.0639 (14)
H10	1.8161	0.3943	1.0385	0.077*
C11	1.6698 (5)	0.3562 (3)	0.8457 (5)	0.0501 (12)
H11	1.6467	0.4157	0.8092	0.060*
C12	1.5998 (4)	0.2804 (3)	0.7581 (5)	0.0415 (10)
C13	1.4937 (4)	0.2886 (3)	0.6154 (4)	0.0350 (9)
C14	1.1979 (4)	0.1533 (3)	0.3348 (5)	0.0413 (10)
C15	1.1854 (4)	0.0836 (2)	0.2173 (4)	0.0377 (10)
H15	1.2410	0.0777	0.1621	0.045*
C16	1.0632 (6)	0.0264 (3)	0.2104 (6)	0.0692 (16)
H16	1.0320	−0.0237	0.1469	0.083*
C17	1.0004 (5)	0.0519 (4)	0.3023 (6)	0.0639 (14)
H17	0.9219	0.0218	0.3062	0.077*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0578 (10)	0.0801 (11)	0.0865 (12)	−0.0166 (7)	0.0289 (8)	−0.0064 (8)
N1	0.0329 (19)	0.0351 (18)	0.041 (2)	−0.0008 (14)	0.0059 (16)	0.0032 (15)
O1	0.0408 (18)	0.0375 (17)	0.060 (2)	0.0030 (13)	0.0019 (15)	0.0081 (15)
C1	0.033 (2)	0.035 (2)	0.043 (2)	0.0012 (17)	0.0090 (19)	0.0008 (19)
C2	0.036 (2)	0.046 (2)	0.047 (3)	0.0019 (19)	0.002 (2)	0.004 (2)
C3	0.031 (2)	0.038 (2)	0.039 (2)	−0.0015 (17)	0.0094 (19)	−0.0004 (18)
C4	0.031 (2)	0.036 (2)	0.049 (3)	−0.0003 (17)	0.014 (2)	0.0054 (19)
C5	0.044 (3)	0.037 (2)	0.063 (3)	0.0006 (19)	0.017 (2)	0.009 (2)
C6	0.043 (3)	0.050 (3)	0.071 (3)	0.007 (2)	0.012 (3)	0.027 (3)
C7	0.034 (2)	0.057 (3)	0.047 (3)	0.002 (2)	0.015 (2)	0.020 (2)
C8	0.039 (3)	0.081 (4)	0.064 (3)	0.001 (3)	0.010 (3)	0.038 (3)
C9	0.038 (3)	0.121 (5)	0.046 (3)	−0.014 (3)	0.002 (2)	0.020 (3)
C10	0.055 (3)	0.081 (4)	0.044 (3)	−0.015 (3)	0.005 (2)	0.007 (3)
C11	0.040 (3)	0.059 (3)	0.044 (3)	−0.007 (2)	0.010 (2)	0.002 (2)
C12	0.031 (2)	0.053 (3)	0.043 (2)	0.0003 (18)	0.017 (2)	0.010 (2)
C13	0.027 (2)	0.037 (2)	0.040 (2)	0.0035 (16)	0.0122 (18)	0.0065 (18)
C14	0.041 (2)	0.039 (2)	0.037 (2)	−0.0025 (17)	0.0084 (19)	0.0008 (18)
C15	0.045 (2)	0.0226 (19)	0.045 (2)	−0.0068 (16)	0.017 (2)	0.0002 (17)
C16	0.082 (4)	0.047 (3)	0.054 (3)	−0.018 (3)	−0.001 (3)	0.001 (2)
C17	0.044 (3)	0.061 (3)	0.070 (3)	−0.013 (2)	0.003 (3)	0.013 (3)

Geometric parameters (Å, °)

N1—C1	1.340 (5)	C6—H6	0.9300
N1—C13	1.413 (5)	C7—C8	1.414 (7)
N1—H1	0.8600	C7—C12	1.419 (6)
O1—C1	1.235 (5)	C8—C9	1.354 (8)
S1—C17	1.649 (6)	C8—H8	0.9300
S1—C14	1.679 (4)	C9—C10	1.388 (7)
C1—C2	1.493 (6)	C9—H9	0.9300
C2—C3	1.535 (6)	C10—C11	1.362 (7)
C2—H2A	0.9700	C10—H10	0.9300
C2—H2B	0.9700	C11—C12	1.410 (6)
C3—C14	1.510 (5)	C11—H11	0.9300
C3—C4	1.513 (6)	C12—C13	1.412 (6)
C3—H3	0.9800	C14—C15	1.503 (5)
C4—C13	1.381 (6)	C15—C16	1.485 (6)
C4—C5	1.410 (6)	C15—H15	0.9300
C5—C6	1.360 (7)	C16—C17	1.348 (7)
C5—H5	0.9300	C16—H16	0.9300
C6—C7	1.400 (7)	C17—H17	0.9300
C1—N1—C13	124.6 (3)	C9—C8—C7	120.9 (5)
C1—N1—H1	117.7	C9—C8—H8	119.5
C13—N1—H1	117.7	C7—C8—H8	119.5

C17—S1—C14	93.6 (2)	C8—C9—C10	120.0 (5)
O1—C1—N1	121.6 (4)	C8—C9—H9	120.0
O1—C1—C2	122.0 (4)	C10—C9—H9	120.0
N1—C1—C2	116.4 (4)	C11—C10—C9	121.0 (5)
C1—C2—C3	112.2 (3)	C11—C10—H10	119.5
C1—C2—H2A	109.2	C9—C10—H10	119.5
C3—C2—H2A	109.2	C10—C11—C12	121.0 (4)
C1—C2—H2B	109.2	C10—C11—H11	119.5
C3—C2—H2B	109.2	C12—C11—H11	119.5
H2A—C2—H2B	107.9	C11—C12—C13	124.0 (4)
C14—C3—C4	114.1 (3)	C11—C12—C7	117.9 (4)
C14—C3—C2	110.4 (3)	C13—C12—C7	118.1 (4)
C4—C3—C2	110.0 (3)	C4—C13—C12	122.2 (4)
C14—C3—H3	107.4	C4—C13—N1	119.0 (4)
C4—C3—H3	107.4	C12—C13—N1	118.8 (4)
C2—C3—H3	107.4	C15—C14—C3	124.1 (4)
C13—C4—C5	118.4 (4)	C15—C14—S1	113.5 (3)
C13—C4—C3	118.9 (4)	C3—C14—S1	122.3 (3)
C5—C4—C3	122.7 (4)	C16—C15—C14	103.4 (4)
C6—C5—C4	120.4 (4)	C16—C15—H15	128.3
C6—C5—H5	119.8	C14—C15—H15	128.3
C4—C5—H5	119.8	C17—C16—C15	115.6 (4)
C5—C6—C7	122.1 (4)	C17—C16—H16	122.2
C5—C6—H6	119.0	C15—C16—H16	122.2
C7—C6—H6	119.0	C16—C17—S1	113.9 (4)
C6—C7—C8	122.2 (4)	C16—C17—H17	123.0
C6—C7—C12	118.7 (4)	S1—C17—H17	123.0
C8—C7—C12	119.1 (5)		

Fig. 1