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5,6,7,8-Tetrahydro-4a*H*-thieno[3,2-*b*]-quinolizin-4(10*H*)-one

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.114
Data-to-parameter ratio = 17.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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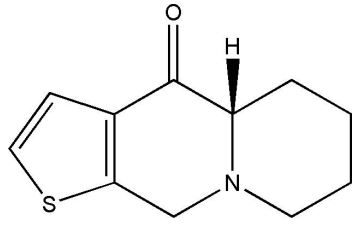
In the title compound, $C_{11}H_{13}NOS$, the thiophene ring is planar while the central six-membered ring has a half-chair conformation and the second ring of the quinolizine moiety is in a chair conformation. The angles at the N atom sum to 331.22°, indicating pyramidalization of this atom.

Comment

Hydroquinolizine derivatives continue to atract the attention of organic and medicinal chemists because of their potential application as pharmaceutical drugs for the treatment of diabetes (Kubo *et al.*, 2000). Benzoquinolizine derivatives are interesting as selective non-steroidal inhibitors of steroid 5α-reductase-1 (Guarna *et al.*, 2001). Selective inhibition of 5αR-1 is currently investigated as a potential therapeutic tool for the treatment of dihydrotestosterone-related skin disorders, such as acne, alopecia, male baldness and hirsutism (Harris & Kozarich, 1997). X-ray crystallographic analysis was undertaken to determine the structure and stereochemistry of the title compound, (I).



(I)

The X-ray analysis shows that the molecule consists of a thiophene ring fused to a quinolizine moiety (Fig. 1). The central ring (atoms N12, C9, C13, C10, C4 and C11) is in a half-chair conformation (Cremer & Pople, 1975). The sp^3 hybridization of atom N12 is confirmed [the sum of the bond angles

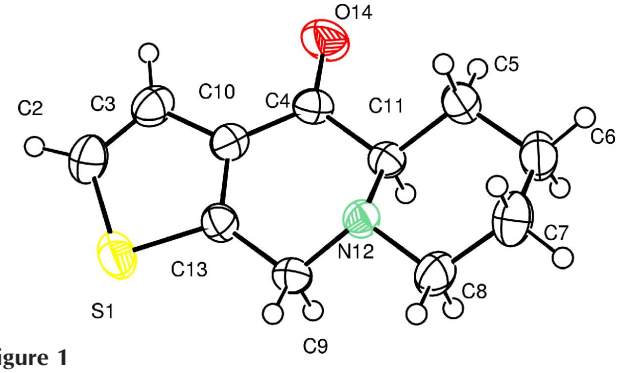


Figure 1
The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

around N12 is 331.22°]. The second ring of the quinolizine moiety (atoms N12, C11, C5, C6, C7 and C8) assumes a chair conformation. The ring is puckered in such a manner that atoms C5, C6, C8 and N12 are coplanar to within 0.009 (2) Å, while atoms C7 and C11 are displaced from this plane on opposite sides, with out-of-plane displacements of 0.667 (2) and 0.661 (1) Å, respectively. The metrical parameters of the quinolizine moiety are comparable with those in similar structurally related compounds (Hussain *et al.*, 1996; Beckwith *et al.*, 1995).

Experimental

The title compound, (I), was prepared by intramolecular acylation of *N*-[(2-thienyl)methyl]-2-piperidinecarboxylic acid hydrochloride in polyphosphoric acid (Marchalín & Decroix, 1994). Colorless prismatic single crystals were prepared by recrystallization from cyclohexane.

Crystal data

$C_{11}H_{13}NOS$
M_r = 207.28
Monoclinic, $P2_1/c$
a = 6.808 (2) Å
b = 18.770 (9) Å
c = 8.256 (3) Å
β = 105.84 (3)°
V = 1014.9 (7) Å^3
Z = 4
D_x = 1.357 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2238 reflections
θ = 2.3–34.1°
μ = 0.28 mm^{-1}
T = 298 (2) K
Prism, colorless
0.60 × 0.50 × 0.30 mm

Data collection

Kuma KM-4 CCD diffractometer
ω scans
Absorption correction: none
8775 measured reflections
2238 independent reflections
1765 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.034
θ_{max} = 27.1°
$h = -8 \rightarrow 8$
$k = -24 \rightarrow 0$
$l = -10 \rightarrow 5$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.114
S = 1.01
2238 reflections
128 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 0.1855P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0060 (19)

Table 1
Selected geometric parameters (Å, °).

C2—S1	1.7214 (19)	C9—C13	1.489 (2)
C4—C10	1.454 (2)	C10—C13	1.362 (2)
C8—N12	1.4687 (19)	C11—N12	1.4660 (18)
C9—N12	1.4617 (18)	C13—S1	1.7062 (16)
C6—C5—C11	111.40 (13)	C13—S1—C2	91.57 (8)
C7—C6—C5	109.77 (13)	C9—N12—C11	111.62 (11)
C13—C10—C4	119.51 (13)	C9—N12—C8	107.99 (12)
C10—C13—C9	123.30 (13)	C11—N12—C8	111.51 (12)
C5—C6—C7—C8	54.5 (2)	N12—C9—C13—S1	−156.27 (11)
O14—C4—C10—C3	−2.1 (2)	C9—C13—S1—C2	179.76 (14)
C10—C4—C11—N12	−27.47 (16)	C5—C11—N12—C8	−57.20 (16)

H atoms were positioned geometrically and treated as riding atoms (C—H = 0.95–0.99 Å) with U_{iso} set to 1.2U_{eq} of the parent atom.

Data collection: *KM-4 Software* (Kuma, 1992); cell refinement: *KM-4 Software*; data reduction: *KM-4 Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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