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2,4,4a,5,6,7,9,9a-Octahydrofuro[2,3-*f*]-indolizin-7-one

Viktor Vrábel,[a]* Jozef Kožíšek,[b] Vratislav Langer[c] and Štefan Marchalín[d]

[a]Department of Analytical Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinského 9, SK-81237 Bratislava, Slovak Republic, [b]Department of Physical Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinského 9, SK-81237 Bratislava, Slovak Republic, [c]Department of Environmental Inorganic Chemistry, Chalmers University of Technology, SE-41296 Göteborg, Sweden, and [d]Department of Organic Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinského 9, SK-81237 Bratislava, Slovak Republic

Correspondence e-mail: vrabel@cvt.stuba.sk

Key indicators

Single-crystal X-ray study
T = 183 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.115
Data-to-parameter ratio = 15.7

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

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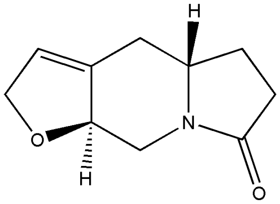
In the title compound, $C_{10}H_{13}NO_2$, the conformation of the oxopyrrolidine ring is close to that of an envelope. The central six-membered ring has a chair conformation. The nearly planar furan ring attached to the indolizine ring system is slightly buckled, with a mean deviation of 0.037 Å.

Comment

Heterocyclic compounds, such as indolizines, are important bioactive compounds that have a wide range of applications in biology, pharmacology and agrochemistry (Gubin *et al.*, 1992). Several polyhydroxylated indolizines show interesting activity as inhibitors of glycosides (Hempel *et al.*, 1993; Brandi *et al.*, 1995). An X-ray crystallographic analysis was undertaken to determine the structure and stereochemistry of the title compound, (I).



(I)

The structure of (I) (Fig. 1) is stabilized by van der Waals forces; the shortest intermolecular contact is 3.413 (2) Å for C5···O1($1 - x, y - \frac{1}{2}, \frac{1}{2} - z$). The central six-membered ring is not planar and resembles a chair (Cremer & Pople, 1975). A calculation of the least-squares plane has shown that this ring is puckered in such a manner that the four atoms C4, C6, N10 and C12 are coplanar to within 0.022 (2) Å, while atoms C5 and C11 are unequally displaced from this plane on opposite sides, with out-of-plane displacements of 0.626 (2) and 0.615 (2) Å, respectively. The oxopyrrolidine ring is distorted towards an envelope conformation, with C7 as the flap. The deviation of atom C7 from the mean plane of the remaining four atoms is 0.243 (2) Å. The fused furan ring is almost planar (mean deviation = 0.037 Å). Bond lengths and angles in the indolizine ring system are in agreement with values from the literature (Camus *et al.*, 2000, 2003; Lokaj *et al.*, 1999). Atom N10 is sp^2 hybridized, as evidenced by the sum of the valence angles around it (359.3°). These data are consistent with conjugation of the lone-pair electrons on N10 with the adjacent carbonyl, similar to what is observed for amides.

Experimental

The title compound was prepared by diastereoselective reduction of the furan ring of starting (4*R*,4a*S*)-4-hydroxy-4,4a,5,6,7,9-hexahydro[2,3-*f*]indolizin-7-one (Szemes *et al.*, 1998) to the 2,5-dihydro-

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furo homolog by triethylsilane in trifluoroacetic acid. Colorless block-shaped single crystals were prepared by recrystallization from an ethanol solution.

Crystal data

$C_{10}H_{13}NO_2$
$M_r = 179.21$
Orthorhombic, $P2_12_12_1$
$a = 6.6561$ (2) Å
$b = 11.1986$ (3) Å
$c = 11.8739$ (3) Å
$V = 885.07$ (4) Å^3
$Z = 4$
$D_x = 1.345$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 8192 reflections
$\theta = 3.5$–31.3°
$\mu = 0.09$ mm^{-1}
$T = 183$ (2) K
Block, colorless
0.26 × 0.25 × 0.10 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{\min} = 0.976$, $T_{\max} = 0.991$
15 853 measured reflections

1854 independent reflections
1611 reflections with $F^2 > 2\sigma(F^2)$
$R_{\rm int} = 0.036$
$\theta_{\max} = 32.9°$
$h = -10 \rightarrow 10$
$k = -17 \rightarrow 17$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.115$
$S = 1.03$
1854 reflections
118 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0663P)^2 + 0.14P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.010$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C3—C4	1.333 (2)	C6—C7	1.536 (3)
C5—C6	1.539 (2)	C9—O13	1.230 (2)
C6—N10	1.468 (2)	C9—N10	1.353 (2)
C3—C4—C5	132.86 (14)	O13—C9—N10	125.26 (15)
C5—C4—C12	117.89 (13)	C9—N10—C11	125.09 (13)
C7—C6—C5	115.22 (17)	C11—N10—C6	119.36 (13)
O1—C2—C3—C4	6.16 (19)	C5—C6—N10—C11	−57.90 (19)
C7—C8—C9—N10	8.7 (2)	C2—O1—C12—C11	128.43 (14)
C7—C6—N10—C9	−10.3 (2)	N10—C11—C12—C4	−47.48 (16)

H atoms were positioned geometrically and treated as riding atoms (C—H = 0.95–0.99 Å), with $U_{\rm iso}$ set at $1.2U_{\rm eq}$ of the parent atom. The absolute configuration could not be reliably determined for this compound using Mo radiation, and has been assigned arbitrarily. Friedel pairs were merged.

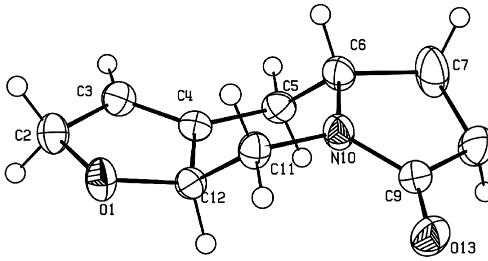

Figure 1
The molecular structure of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT* and *SADABS* (Sheldrick, 2002); program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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