

**(3*a*S,8*a*S,9*R*,9*a*S)-9-Hydroxy-2,3,3*a*,7,8,8*a*,9,9*a*-octahydrofuro[3,2-*f*]indolizin-6(4*H*)-one****Viktor Vrábek,<sup>a\*</sup> Jozef Kožíšek,<sup>b</sup>  
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Marchalín<sup>d</sup> and Fridrich Szemes<sup>d</sup>**<sup>a</sup>Department of Analytical Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinského 9, SK-812 37 Bratislava, Slovak Republic, <sup>b</sup>Department of Physical Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinského 9, SK-812 37 Bratislava, Slovak Republic, <sup>c</sup>Department of Environmental Inorganic Chemistry, Chalmers University of Technology, SE-41296 Göteborg, Sweden, and <sup>d</sup>Department of Organic Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinského 9, SK-812 37 Bratislava, Slovak Republic

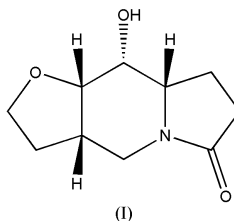
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**Key indicators**Single-crystal X-ray study  
*T* = 183 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$   
*R* factor = 0.067  
*wR* factor = 0.166  
Data-to-parameter ratio = 13.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound,  $\text{C}_{10}\text{H}_{15}\text{NO}_3$ , the central six-membered ring has a chair conformation. The conformations of the fused furan and oxopyrrolidine rings are close to envelopes. Molecules form chains parallel to the *b* axis, via  $\text{O}-\text{H}\cdots\text{O}$  intermolecular hydrogen bonds.

**Comment**

Indolizine derivatives are an important class of heterocyclic bioactive compounds which have a wide range of applications, such as pharmaceutical drugs, potential central nervous system depressants, calcium entry blockers, cardiovascular agents, spectral sensitizers and novel dyes (Gubin *et al.*, 1992; Poty *et al.*, 1994; Hema *et al.*, 2003). Several polyhydroxylated indolizines are interesting as inhibitors of glycosides (Hempel *et al.*, 1993; Brandi *et al.*, 1995). Indolizines have also been tested as antimycobacterial agents against mycobacterial tuberculosis (Gundersen *et al.*, 2003). The structural investigation of the title compound, (I), has been undertaken as part of our study of the conformational changes caused by different substituents at positions on the furo-indolizine ring system.



The structure of (I) is shown in Fig. 1. The bond lengths and angles in the molecule (Table 1) are comparable with those in a related structure (Vrábek *et al.*, 2004).

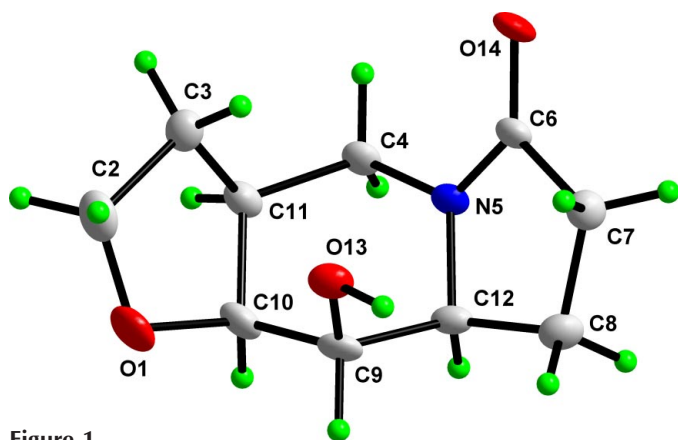
The central six-membered ring adopts a chair conformation (Cremer & Pople, 1975). A calculation of least-squares planes shows that this ring is puckered in such a manner that the four atoms C4, C9, C11 and C12 are coplanar to within 0.009 (3) Å, while atoms N5 and C10 are displaced from this plane on opposite sides, with out-of-plane displacements of 0.597 (3) and 0.588 (3) Å, respectively. The furan and pyrrolidine rings are each distorted towards an envelope conformation, with atoms C11 and C12 as the flaps. The displacements of atoms C11 and C12 from the mean planes of the remaining four atoms are 0.582 (3) and 0.309 (3) Å, respectively.

Intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds link the molecules of (I) into extended chains, which run parallel to the *b* axis (Fig. 2 and Table 2).

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

## Experimental

The title compound was prepared by the stereoselective hydrogenation of both the furan ring and the carbonyl group under the usual conditions. Activated Raney nickel (50% powder, 2.0 g) was added to a solution of (8*aS*)-8,8*a*-dihydrofuro[3,2-*f*]indolizine-6,9(4*H*,7*H*)-dione (1.0 g, 5.2 mmol) in anhydrous methanol (50 ml) and stirred at room temperature under a hydrogen atmosphere for 48 h. The solution was then filtered through a Celite pad to remove the catalyst. After concentration *in vacuo*, the solid residue was purified by column chromatography (dichloromethane–acetone, 9:1). Colourless single crystals of (I) were obtained by recrystallization from an ethanol solution.

### Crystal data

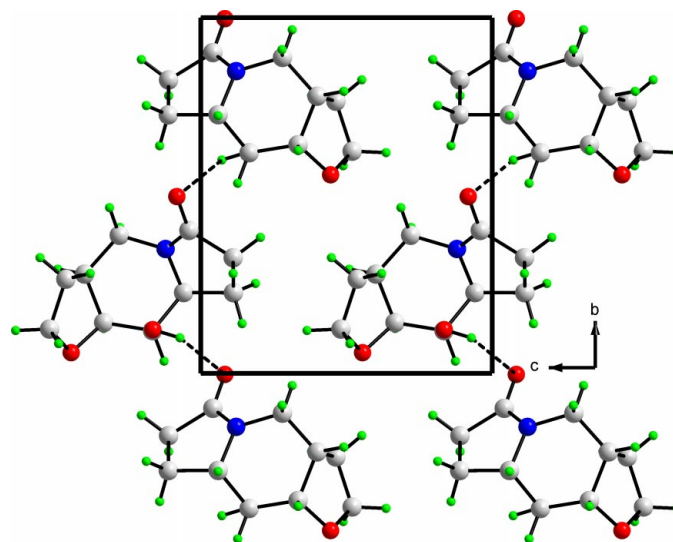
$C_{10}H_{15}NO_3$	$D_x = 1.410 \text{ Mg m}^{-3}$
$M_r = 197.23$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 3686 reflections
$a = 5.3003(3) \text{ \AA}$	$\theta = 2.4\text{--}33.0^\circ$
$b = 10.3540(5) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 8.5137(4) \text{ \AA}$	$T = 183(2) \text{ K}$
$\beta = 96.127(1)^\circ$	Thin plate, colourless
$V = 464.56(4) \text{ \AA}^3$	$0.35 \times 0.12 \times 0.02 \text{ mm}$
$Z = 2$	

### Data collection

Siemens SMART CCD area-detector diffractometer	1748 independent reflections
$\omega$ scans	1377 reflections with $F^2 > 2\sigma(F^2)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2002)	$R_{\text{int}} = 0.085$
$T_{\text{min}} = 0.965$ , $T_{\text{max}} = 0.998$	$\theta_{\text{max}} = 33.0^\circ$
7003 measured reflections	$h = -8 \rightarrow 7$
	$k = -15 \rightarrow 15$
	$l = -12 \rightarrow 12$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.088P)^2 + 0.2047P]$
$R[F^2 > 2\sigma(F^2)] = 0.067$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.166$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.47 \text{ e \AA}^{-3}$
1748 reflections	$\Delta\rho_{\text{min}} = -0.39 \text{ e \AA}^{-3}$
131 parameters	
H atoms treated by a mixture of independent and constrained refinement	



**Figure 2**  
A packing diagram for (I), viewed down the  $a$  axis. Intermolecular O...H...O hydrogen bonds are shown as dashed lines.

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

O1—C10	1.427 (4)	C6—O14	1.239 (4)
C3—C11	1.538 (5)	C8—C12	1.545 (5)
N5—C6	1.338 (4)	C9—O13	1.418 (4)
N5—C12	1.451 (4)	C9—C12	1.533 (4)
C10—O1—C2	109.1 (2)	O13—C9—C12	110.5 (2)
C6—N5—C4	125.3 (3)	C11—C10—C9	114.2 (3)
C12—N5—C4	118.3 (3)	N5—C12—C9	107.5 (2)
O14—C6—N5	125.8 (3)	C9—C12—C8	114.9 (3)
O1—C2—C3—C11	17.3 (3)	O13—C9—C10—O1	−51.0 (3)
N5—C6—C7—C8	4.5 (3)	N5—C4—C11—C10	−37.3 (4)
C2—O1—C10—C9	95.2 (3)	O13—C9—C12—C8	53.9 (3)

**Table 2**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
O13—H13...O14 <sup>i</sup>	0.85 (5)	1.92 (5)	2.708 (3)	155 (4)
C3—H3A...O13	0.99	2.48	2.937 (4)	108
C4—H4B...O14	0.99	2.55	2.920 (4)	102

Symmetry code: (i)  $1 - x, y - \frac{1}{2}, -z$ .

Atom H13 was refined isotropically. All other H atoms were positioned geometrically and treated as riding atoms ( $C\text{--}H = 0.95\text{--}0.99 \text{ \AA}$ ), with  $U_{\text{iso}}(H) = 1.2U_{\text{eq}}$  of the parent atom. The absolute configuration could not be reliably determined for this compound using Mo radiation, and has been assigned according to the synthesis. Friedel pairs have been merged.

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: SHELXL97 (Sheldrick, 1997); molecular graphics: DIAMOND (Brandenburg, 2002); software used to prepare material for publication: SHELXL97.

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