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(1*R*,4*bS*,8*aR*)-(1,2,3,4*b*,5,6,7,8,8*a*,9*a*-Decahydro-4,9*a*-diazafluoren-1-yl)methanol

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Decahydro-4,9*a*-diazafluoren-1-yl)-
methanol**

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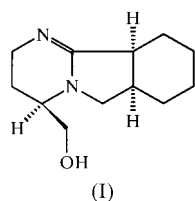
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The crystal structure of the title compound, C₁₂H₂₀N₂O, was determined in order to establish the configuration of C4*b* (*S*) and C8*a* (*R*) with respect to the known configuration of C1 (*R*). The compound forms infinite chains of hydrogen-bonded molecules parallel to the *c* axis.

Comment

The title compound, (I), was prepared during a study on the synthesis of enantiopure hydroxyamidines (Ostendorf *et al.*, 2000). The crystal structure determination was undertaken in order to establish the configuration of atoms C4*b* and C8*a* with respect to the known (*R*) configuration of C4. C4*b* turned out to have the *S* configuration, while C8*a* has the *R* configuration.



Due to the presence of the C3=N4 double bond and the *sp*² hybridization of N9*a*, the atoms of the nitrogen-containing rings more or less lie within one plane [maximum deviation from the least-squares plane is 0.118 (3) Å for C1], with the exception of atoms C2 [deviation 0.695 (3) Å] and C8*a* [deviation 0.515 (3) Å] which protrude on opposite sides. The five-membered ring and the six-membered heteroatomic ring are therefore in a somewhat distorted envelope conformation. The six-membered carbon ring has adopted a slightly deformed chair conformation.

The hydroxyl group donates an intermolecular hydrogen bond to N4 (geometric details in Table 2), thus forming an infinite one-dimensional chain parallel to the *c* axis [graph set *C*(7); Bernstein *et al.*, 1995]. The two screw-related anti-parallel chains present in the unit cell are joined by C—H...O

interactions involving C4*a* and O11 (geometric details in Table 2), creating a two-dimensional network perpendicular to the *a* axis. The C—H...O interactions by themselves organise the molecules in infinite one-dimensional chains [graph set *C*(7)] parallel to the *b* axis.

Experimental

Details of the preparation of (I) are given by Ostendorf *et al.* (2000). Crystals were obtained after recrystallization from benzene.

Crystal data

C ₁₂ H ₂₀ N ₂ O	Mo <i>K</i> α radiation
<i>M_r</i> = 208.30	Cell parameters from 24 reflections
Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁	θ = 10.05–15.57°
<i>a</i> = 8.4691 (9) Å	μ = 0.081 mm ⁻¹
<i>b</i> = 10.3985 (8) Å	<i>T</i> = 150 K
<i>c</i> = 12.4538 (15) Å	Block, colourless
<i>V</i> = 1096.8 (2) Å ³	0.3 × 0.2 × 0.1 mm
<i>Z</i> = 4	
<i>D_x</i> = 1.262 Mg m ⁻³	

Data collection

Enraf–Nonius CAD-4T diffractometer	θ_{\max} = 27.49°
ω scans	<i>h</i> = -11 → 0
2161 measured reflections	<i>k</i> = -10 → 13
1461 independent reflections	<i>l</i> = 0 → 16
1007 reflections with <i>I</i> > 2σ(<i>I</i>)	3 standard reflections
<i>R</i> _{int} = 0.035	frequency: 60 min
	intensity decay: 6%

Refinement

Refinement on <i>F</i> ²	H atoms treated by a mixture of independent and constrained refinement
<i>R</i> (<i>F</i>) = 0.050	$wR(F^2) = 0.104$
<i>S</i> = 0.993	$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2]$
1461 reflections	where $P = (F_o^2 + 2F_c^2)/3$
139 parameters	(Δ/σ) _{max} < 0.001
	$\Delta\rho_{\max} = 0.23 \text{ e } \text{Å}^{-3}$
	$\Delta\rho_{\min} = -0.24 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O11—C10	1.412 (3)	N9 <i>a</i> —C4 <i>a</i>	1.361 (3)
N4—C3	1.475 (4)	N9 <i>a</i> —C9	1.466 (3)
N4—C4 <i>a</i>	1.277 (4)	N9 <i>a</i> —C1	1.457 (3)
C3—N4—C4 <i>a</i>	114.8 (2)	C4 <i>a</i> —N9 <i>a</i> —C9	112.24 (19)
C1—N9 <i>a</i> —C4 <i>a</i>	123.1 (2)	C1—N9 <i>a</i> —C9	122.7 (2)

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O11—H11...N4 ⁱ	1.03 (3)	1.74 (3)	2.750 (3)	167 (2)
C4 <i>b</i> —H4 <i>B</i> ...O11 ⁱⁱ	1.00	2.42	3.351 (3)	154

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

Due to the absence of significant anomalous dispersion, the absolute structure cannot be determined reliably. The configuration of C1 was set equal to *R*, consistent with the starting materials used (see Ostendorf *et al.*, 2000). The 2197 measured reflections reduced to

1924 unique reflections. After merging of the Friedel pairs, 1461 reflections remained. The hydroxyl H atom was located on a difference Fourier map and its coordinates were refined. The other H atoms were also located on a difference Fourier map, but in order to limit the number of refined parameters, they were included in the refinement on calculated positions riding on their carrier atoms. All H-atom displacement parameters were set equal to the equivalent isotropic displacement parameter of their carrier atom, multiplied by a constant factor of 1.5 (hydroxyl H) or 1.2 (other H atoms).

Data collection: locally modified *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *SET4* (de Boer & Duisenberg, 1984); data reduction: *HELENA* (Spek, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2000); software used to prepare material for publication: *PLATON*.

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