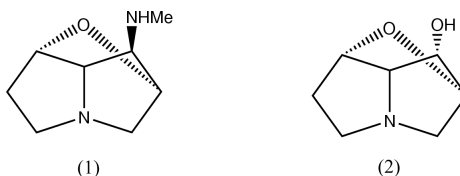


(1 α ,6 α ,7 α ,7 α β)-2,3,5,6,7,7 α -Hexahydro-1,6-epoxy-1*H*-pyrrolizin-7-olRichard S. Glass,^{a*} Donald R. Deardorff,^b Nhu Y. T. Stessman^c and Michael D. Carducci^a^aDepartment of Chemistry, The University of Arizona, Tucson, AZ 85721, USA, ^bDepartment of Chemistry, Occidental College, Los Angeles, CA 90041, USA, and ^cDepartment of Chemistry, California State University, Stanislaus, Turlock, CA 95382, USA

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Key indicatorsSingle-crystal X-ray study
T = 200 K
Mean σ (C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.081
Data-to-parameter ratio = 9.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, C₇H₁₁NO₂, is an intermediate in a synthetic approach to the pyrrolizidine alkaloid loline. It has a brendane skeleton with a 91.8(2)° bond angle for the bridging carbon bearing the OH group.**Comment**

Loline alkaloids (Powell & Petroski, 1992) are a group of pyrrolizidine alkaloids that feature a novel bridging ether as illustrated in loline, (1). These alkaloids occur in *Lolium cuneatum* (Dannhardt & Steindl, 1985), *Festuca arundinacea* (Yates & Tookey, 1965), *Adenocarpus decorticans* (Ribas-Barceló & Ribas-Marqués, 1968), and *Argyreia mollis* (Tofern *et al.*, 1999) and have interesting biological properties. A number of synthetic approaches to these alkaloids have been reported (Wilson & Sawicki, 1978, Glass *et al.*, 1978; Wilson *et al.*, 1981), as well as a successful synthesis of (±)-loline (Tufariello *et al.*, 1986) and (+)-loline (Blakemore *et al.*, 2001). In our synthetic approach, the title compound, (2), was synthesized and characterized structurally. This paper reports its complete X-ray structural elucidation. This X-ray study shows that the molecule is an analog of brendane and, in particular, the small C–C–C bond angle, 91.8(2)°, for the one-carbon bridge bearing the OH group renders such alcohol derivatives unreactive in nucleophilic displacement reactions. In addition, neighboring group participation by β -nitrogen is precluded by the geometry of the tricyclic ring system. Molecules of (2) form infinite linear chains through hydrogen bonding of the alcohol moiety of one molecule with the amine moiety of the next molecule (Table 1).

**Experimental**The synthesis of (2) was carried out as reported (Glass *et al.*, 1978). Crystals of (2) were grown by evaporation of a benzene solution.*Crystal data*C₇H₁₁NO₂
M_r = 141.17
Orthorhombic, *Pna*2₁
a = 9.9294 (16) Å
b = 7.2910 (12) Å
c = 9.1653 (15) Å
V = 663.52 (19) Å³
Z = 4
D_x = 1.413 Mg m⁻³Mo *K* α radiation
Cell parameters from 2854 reflections
 θ = 3.5–28.2°
 μ = 0.10 mm⁻¹
T = 200 (2) K
Irregular block, colorless
0.09 × 0.09 × 0.08 mm

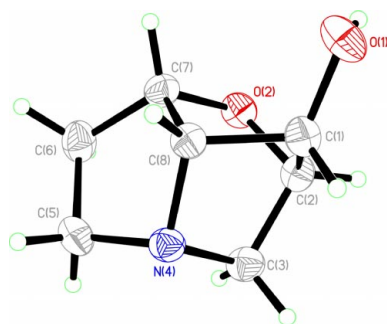


Figure 1
View of (2). Displacement ellipsoids are drawn at the 50% probability level.

Data collection

Bruker SMART CCD area-detector diffractometer	880 independent reflections
φ and ω scans	795 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2000)	$R_{\text{int}} = 0.029$
$T_{\text{min}} = 0.893$, $T_{\text{max}} = 0.992$	$\theta_{\text{max}} = 28.3^\circ$
6311 measured reflections	$h = -12 \rightarrow 13$
	$k = -9 \rightarrow 9$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0382P)^2 + 0.1884P]$
$R[F^2 > 2\sigma(F^2)] = 0.032$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.081$	$(\Delta/\sigma)_{\text{max}} = 0.006$
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.29 \text{ e } \text{\AA}^{-3}$
880 reflections	$\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{\AA}^{-3}$
94 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O1-H1A \cdots N4^i$	0.76 (3)	2.01 (3)	2.760 (2)	167 (3)

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

As a result of the very weak anomalous scattering in this experiment [Flack (1983) parameter $x = 0.0(18)$], all Friedel pairs were merged in the final cycles of refinement. The structure is racemic, and the absolute polarity of the crystal is unknown. All H atoms were positioned geometrically at idealized positions, constrained to ride on the atom to which they are attached and given U_{iso} values equal to 1.2 or 1.5 times U_{eq} of the bonded atom. In the final cycles of refinement, the hydroxyl H atom was freed from the riding constraint.

Data collection: SMART (Bruker, 1997); cell refinement: SMART; data reduction: SAINT (Bruker, 1997); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

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