

Intermolecular contacts between N(4) and O(2) indicate hydrogen bonding in both structures (Hamilton & Ibers, 1968). For (I) N(4)···O(2) is 2.69 and N(4)···H(O2) is 1.79 Å. For (II) N(4)···O(2) is 2.72 and N(4)···H(O2) is 1.74 Å. The combined van der Waals radii are N···O 2.9 and N···H(O) 2.7 Å.

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Diastereoisomeric Epoxides of Heliotridine and Retronecine, C₈H₁₃NO₃

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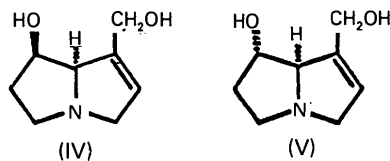
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Abstract. $M_r = 171.1$, $\lambda(\text{Mo } K\bar{\alpha}) = 0.71069 \text{ \AA}$, $T = 298 \text{ K}$. β -Epoxyheliotridine (I): orthorhombic, $P2_12_12_1$, $a = 6.2951 (3)$, $b = 7.7019 (5)$, $c = 17.722 (1) \text{ \AA}$, $U = 859.25 (9) \text{ \AA}^3$, $Z = 4$, $D_m = 1.321$, $D_x = 1.322 \text{ g cm}^{-3}$, $\mu(\text{Mo } K\bar{\alpha}) = 0.63 \text{ cm}^{-1}$, $F(000) = 368$. α -Epoxyheliotridine (II): monoclinic, $P2_1$, $a = 6.251 (2)$, $b = 8.578 (1)$, $c = 8.069 (2) \text{ \AA}$, $\beta = 110.68 (2)^\circ$, $U = 404.8 (2) \text{ \AA}^3$, $Z = 2$, $D_m = 1.403$, $D_x = 1.405 \text{ g cm}^{-3}$, $\mu(\text{Mo } K\bar{\alpha}) = 0.66 \text{ cm}^{-1}$, $F(000) = 184$. α -Epoxyretronecine (III): monoclinic, $P2_1$, $a = 6.509 (1)$, $b = 8.340 (1)$, $c = 7.799 (1) \text{ \AA}$, $\beta = 105.85 (1)^\circ$, $U = 407.29 (1) \text{ \AA}^3$, $Z = 2$, $D_m = 1.395$,

$D_x = 1.396 \text{ g cm}^{-3}$, $\mu(\text{Mo } K\bar{\alpha}) = 0.66 \text{ cm}^{-1}$, $F(000) = 184$. Full-matrix least-squares refinement converged at R values of 0.037, 0.038 and 0.032 for 1389, 1299 and 1395 reflections for (I), (II) and (III), respectively. There are no unusual bond distances or angles. There is intermolecular H bonding between N and the O atom of the CH₂OH group (N···O 2.70, 2.73 and 2.77 Å, respectively). The absolute configurations of the three diastereomers are defined by reference to the absolute configurations of the parent alkaloids heliotridine and retronecine [Warren & Von Klemperer (1958). *J. Chem. Soc.* pp. 4574–4575; Warren (1970). *The Alkaloids*, Vol. XII, edited by Manske, ch. 4, pp. 246–262. London, New York: Academic Press; and references therein].

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Introduction. It has been estimated that 3% of the world's flowering plants contain pyrrolizidine alkaloids and such plants can be expected to be present in most environments (Smith & Culvenor, 1981). Approximately 300 pyrrolizidine alkaloids have been characterized and many have been shown to be responsible for the long-known hepatotoxicity in humans and animals (Roitman, 1983; McLean, 1970). We have been interested, for several years, in the antitumor activity of certain of these alkaloids (Zalkow *et al.*, 1979; Zalkow, Glinski, Gelbaum, Fleischmann, McGowan & Gordon, 1985) and semisynthetic analogs (Gelbaum, Gordon, Miles & Zalkow, 1982). Almost all of the biologically active (toxic and antitumor) pyrrolizidine alkaloids and *N*-oxides contain, as their necine bases, either retronecine (IV) or heliotridine (V) and the biological activity is reportedly dependent upon the allylic oxygen function in ring *A* (Culvenor, 1968). In a previous communication, we reported the X-ray structures of retronecine (IV) and heliotridine (V) (Gelbaum, Glinski, Van Derveer & Zalkow, 1985).



We felt it was essential to prepare epoxides of retronecine and heliotridine in order to gain insight into the role of the double bond and the oxirane ring in these biologically important molecules. We report here, for the first time, the preparation and X-ray structural information of β -epoxyheliotridine (I), α -epoxyheliotridine (II) and α -epoxyretronecine (III).

Experimental. β -Epoxyheliotridine (I) and α -epoxyheliotridine (II) were prepared by the reaction of heliotridine (V) with *N*-bromoacetamide followed, *in situ*, by treatment with base, and these products were isolated after chromatography on alumina. Similar treatment of retronecine (IV) gave α -epoxyretronecine (III) and several other products. None of the isomeric β -epoxyretronecine was detected in the reaction product.

Crystals of (I), (II) and (III) were obtained from methanol. Specific rotations $[\alpha]_D^{25}$ (ethanol, 1.0 g dm⁻³) and melting points (uncorrected) for (I), (II) and (III) were +2.5°, 424–425.5 K; –17.3°, 426.5–428 K; and –24.0°, 447–449 K, respectively.

Experimental details for the X-ray determinations are in Table 1. All densities determined by flotation in hexane–carbon tetrachloride. *Lp* corrections but no extinction or absorption corrections. Structures all solved in same manner. *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) used

Table 1. X-ray data collection and solution

	(I) β -Epoxyheliotridine	(II) α -Epoxyheliotridine	(III) α -Epoxyretronecine
Crystal size (mm)	0.35 × 0.28 × 0.20	0.36 × 0.25 × 0.10	0.43 × 0.34 × 0.17
No. of reflections for lattice parameters	62	15	15
Diffractometer	Syntex P2 ₁		
Radiation	Mo K α , $\lambda = 0.71069$ Å; graphite monochromator		
2 θ range (°)	20.35–39.01	7.15–24.55	9.76–25.46
Max. 2 θ for data collection (°)	50	50	50
Scan type	θ -2 θ		
Scan speed (° min ⁻¹)	2.02–29.30		
<i>h, k, l</i> range	$\pm h, +k, +l$	$\pm h, \pm k, +l$	$\pm h, \pm k, +l$
No. of reflections measured	1710	1540	1571
No. of reflections with $F > 3\sigma(F)$	1389	1299	1395
<i>R</i>	0.037	0.038	0.032
<i>wR</i>	0.045	0.047	0.037
$g w = 1.0 / (\sigma F)^2 + g F^2$	0.005	0.008	0.002
Max. Δ/σ	0.016	0.007	0.075
Max., min. in $\Delta\rho$ (e Å ⁻³)	0.29–0.27	0.38–0.33	0.30–0.32

to generate a series of *E* maps, one of which correctly located most non-H atoms; after three cycles of full-matrix least-squares refinement (on *F*), remaining non-H atoms located from difference Fourier map; non-H atoms refined anisotropically and H atoms located from subsequent difference Fourier map. Parameters varied: overall scale factor, coordinates of non-H atoms, anisotropic temperature factors of non-H atoms, isotropic temperature factors for H atoms. Scattering factors as in *SHELX76* (Sheldrick, 1976).

Discussion. The *ORTEPII* (Johnson, 1976) views of β -epoxyheliotridine (I), α -epoxyheliotridine (II) and α -epoxyretronecine (III) are shown in Figs. 1, 2 and 3, respectively, using 50% probability ellipsoids. The thermal parameters of the H atoms have been artificially reduced to clarify the pictures.

Final atom coordinates, angles and selected torsional angles are given in Tables 2–6.* The dihedral angle between the planes of N(4), C(8), C(7) and N(4), C(8), C(1) is 124.5 (I), 122.2 (II) and 124.9° (III). The conformational features of the ring *A*, consisting of N(4), C(8), C(1), C(2) and C(3), exhibit very close similarities in all three molecules, mainly because of its fusion with a 1,2-oxirane ring, making the plane of C(8), C(1), C(2), C(3) coplanar within 0.012 (I), 0.003 (II), and 0.016 Å (III). The N forms the top of an envelope which is 0.231 (I), 0.373 (II) and 0.0320 Å (III) from the plane of C(8), C(1), C(2) and C(3). The atoms C(9) and O(3) are 0.595 and 1.211 (I), 0.569 and 1.204 (II) and 0.588 and 1.213 Å (III) from the same plane. The dihedral angle between the plane of C(8), C(1), C(2), C(3) and the oxirane ring is 105.2 (I), 106.1 (II) and 105.0° (III).

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42147 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The less rigid ring *B*, consisting of N(4), C(8), C(7), C(6) and C(5), is coplanar within 0.241 (I), 0.252 (II) and 0.235 Å (III). Its conformation depends mainly on the configuration of the oxirane ring and of the C(7) hydroxyl. Thus, in (I), N(4), C(8), C(7) and C(6) form a plane which is coplanar within 0.073 Å. For (II), similarly, the most coplanar atoms are C(8), N(4), C(5) and C(6) with a greatest deviation from the plane of 0.004 Å. In (III) the plane of C(8), N(4), C(5) and C(6) is coplanar within 0.027 Å.

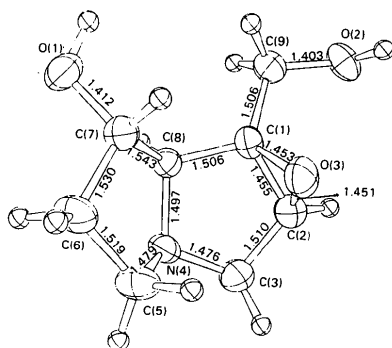


Fig. 1. β -Epoxyheliotridine (the bond-length errors are within 0.003 Å).

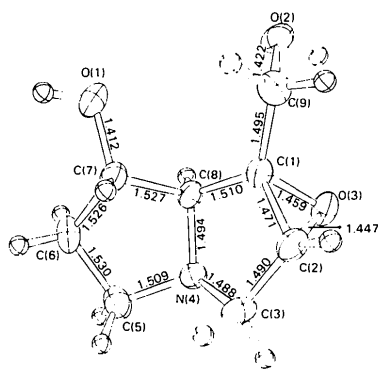


Fig. 2. α -Epoxyheliotridine (the bond-length errors are within 0.004 Å).

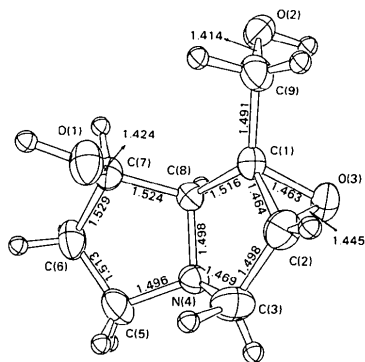


Fig. 3. α -Epoxyretreconcine (the bond-length errors are within 0.004 Å).

Table 2. Final atomic coordinates and U_{eq} values (\AA^2) for β -epoxyheliotridine (I)

Here and in Tables 3 and 4 $U_{eq} = \frac{1}{3} \sum U_{ii}$.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C(1)	-0.2037 (3)	0.0558 (3)	-0.03448 (1)	0.032
C(2)	-0.2375 (3)	0.2205 (3)	-0.3831 (1)	0.041
C(3)	-0.4688 (4)	0.2345 (3)	-0.4047 (1)	0.044
N(4)	-0.5596 (3)	0.0603 (2)	-0.3919 (1)	0.034
C(5)	-0.6008 (4)	-0.0427 (3)	-0.4608 (1)	0.043
C(6)	-0.5950 (4)	-0.2282 (3)	-0.4318 (1)	0.043
C(7)	-0.4077 (3)	-0.2279 (3)	-0.3768 (1)	0.033
C(8)	0.4105 (3)	-0.0426 (3)	-0.3436 (1)	0.029
C(9)	-0.0362 (3)	0.0212 (3)	-0.2860 (1)	0.040
O(1)	-0.4365 (3)	-0.3599 (2)	-0.3224 (1)	0.051
O(2)	0.1350 (2)	0.1374 (2)	-0.29050 (9)	0.043
O(3)	-0.1285 (2)	0.0801 (2)	-0.42154 (9)	0.044

Table 3. Final atomic coordinates and U_{eq} values (\AA^2) for α -epoxyheliotridine (II)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C(1)	0.7680 (4)	1.08176	-0.6899 (3)	0.024
C(2)	0.5617 (4)	1.0671 (4)	-0.6433 (4)	0.032
C(3)	0.5116 (4)	0.8985 (4)	-0.6305 (4)	0.036
C(5)	0.6854 (5)	0.6625 (4)	0.7172 (4)	0.037
C(6)	0.7885 (5)	0.6807 (4)	0.8622 (4)	0.039
C(7)	0.7879 (4)	0.8561 (4)	-0.8931 (3)	0.031
C(8)	0.8505 (4)	0.9186 (4)	-0.7047 (3)	0.024
C(9)	0.8175 (4)	1.2170 (4)	-0.7877 (3)	0.031
N(4)	0.7254 (3)	0.8161 (4)	-0.6200 (3)	0.026
O(1)	0.9463 (4)	0.9101 (4)	-0.9687 (3)	0.053
O(2)	1.0551 (3)	1.2515 (4)	-0.7311 (2)	0.034
O(3)	0.7754 (3)	1.1164 (3)	-0.5110 (2)	0.035

Table 4. Final atomic coordinates and U_{eq} values (\AA^2) for α -epoxyretreconcine (III)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C(1)	0.1926 (2)	0.56820	-0.3065 (2)	0.028
C(2)	0.4067 (3)	0.5618 (3)	-0.3327 (2)	0.040
C(3)	0.4716 (3)	0.3900 (3)	-0.3384 (3)	0.044
N(4)	0.2738 (2)	0.2968 (3)	-0.3655 (2)	0.032
C(5)	0.2980 (4)	0.1435 (3)	-0.2623 (3)	0.051
C(6)	0.1385 (3)	0.1494 (3)	-0.1530 (2)	0.043
C(7)	0.1074 (3)	0.3283 (3)	-0.1256 (2)	0.032
C(8)	0.1155 (2)	0.3968 (2)	-0.3048 (2)	0.026
C(9)	0.1095 (3)	0.7013 (3)	-0.2167 (2)	0.035
O(1)	0.2782 (2)	0.3921 (2)	0.0125 (2)	0.041
O(2)	-0.1144 (2)	0.7212 (2)	-0.2769 (2)	0.039
O(3)	0.2271 (2)	0.6059 (2)	-0.4797 (2)	0.040

Table 5. Angles ($^\circ$) with *e.s.d.*'s in parentheses for (I), (II) and (III)

	(I)	(II)	(III)
C(8)-C(1)-C(2)	108.6 (2)	107.2 (2)	107.3 (1)
C(9)-C(1)-C(8)	120.5 (2)	122.5 (2)	122.3 (1)
O(3)-C(1)-C(8)	111.0 (2)	112.0 (2)	110.2 (1)
C(3)-C(2)-C(1)	108.8 (2)	109.0 (2)	109.0 (1)
O(3)-C(2)-C(3)	113.0 (2)	113.2 (2)	113.2 (2)
C(5)-N(4)-C(3)	115.4 (2)	113.9 (2)	114.5 (1)
C(8)-N(4)-C(5)	107.3 (2)	107.1 (2)	106.6 (1)
C(7)-C(6)-C(5)	103.5 (2)	104.4 (2)	104.3 (1)
O(1)-C(7)-C(6)	109.6 (2)	115.6 (2)	111.4 (1)
N(4)-C(8)-C(1)	105.5 (2)	105.1 (2)	105.3 (1)
C(7)-C(8)-N(4)	106.2 (1)	104.9 (2)	106.3 (1)
C(2)-O(3)-C(1)	60.1 (1)	60.8 (1)	60.5 (1)
C(9)-C(1)-C(2)	125.4 (2)	124.4 (2)	125.0 (1)
O(3)-C(1)-C(2)	59.9 (1)	59.2 (2)	59.2 (1)
O(3)-C(1)-C(9)	116.2 (2)	115.1 (2)	116.1 (1)
O(3)-C(2)-C(1)	60.0 (1)	60.0 (2)	60.4 (1)
N(4)-C(3)-C(2)	105.6 (2)	105.0 (2)	105.5 (1)
C(8)-N(4)-C(3)	109.0 (2)	107.4 (2)	108.1 (1)
C(6)-C(5)-N(4)	102.8 (2)	105.7 (2)	106.7 (1)
C(8)-C(7)-C(6)	103.6 (2)	101.5 (2)	101.4 (1)
O(1)-C(7)-C(8)	113.9 (2)	110.1 (2)	109.8 (1)
C(7)-C(8)-C(1)	116.8 (2)	115.4 (2)	117.1 (1)
O(2)-C(9)-C(1)	112.7 (2)	112.3 (2)	113.9 (1)

Table 6. Selected torsional angles ($^{\circ}$) for (I), (II) and (III)

E.s.d.'s are $\sim 0.3^{\circ}$.

	(I)	(II)	(III)	(I)	(II)	(III)	
N(4)—C(8)—C(1)—C(9)	162.5	168.4	165.8	C(2)—O(3)—C(1)—C(2)	99.9	97.5	98.6
C(8)—C(1)—C(9)—O(2)	173.3	115.2	122.6	N(4)—C(8)—C(1)—C(2)	7.5	14.5	10.6
C(1)—C(2)—C(3)—N(4)	11.1	15.5	15.4	N(4)—C(8)—C(7)—C(6)	2.2	39.0	34.9
C(8)—C(1)—C(2)—C(3)	2.2	0.6	2.8	N(4)—C(8)—C(7)—O(1)	161.6	5.8	83.0
N(4)—C(8)—C(1)—O(3)	56.6	8.6	52.2	C(8)—C(7)—C(6)—C(5)	32.7	38.2	37.4
N(4)—C(3)—C(2)—O(3)	53.5	49.1	49.7	C(7)—C(6)—C(5)—N(4)	41.2	23.7	26.7
C(1)—O(3)—C(2)—C(3)	99.1	99.3	99.5	C(7)—C(8)—N(4)—C(5)	13.5	25.0	19.2

Table 7. Intermolecular distances (\AA)

E.s.d.'s for N...O distances are $\sim 0.004 \text{\AA}$.

	(I)	(II)	(III)	van der Waals distance
N(4)...O(2)	2.70	2.73	2.77	2.90
N(4)...H(O2)	1.76	1.84	1.86	2.70

All three structures show intermolecular contacts between N(4) and O(2) (Table 7) which are somewhat shorter than the combined van der Waals radii. The N(4)...H(O2) distances confirm hydrogen bonding (Hamilton & Ibers, 1968). The orientation of the C(9) hydroxyl [O(2)] seems to be influenced by the position of the epoxide oxygen [O(3)], although the real distance O(2)...O(3) 2.887 (I), 2.802 (II) and 3.699 \AA (III) is greater than the O...O van der Waals distance. Finally, the ORTEP views of (I), (II) and (III) (Figs. 1, 2 and 3) indicate that the C(9)—OH and C(7)—OH bonds are *anti* to each other in each case.

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Structure of 1,8-Bis(trimethylsilyl)naphthalene, C₁₆H₂₄Si₂

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Abstract. $M_r = 272.5$, orthorhombic, $Pna2_1$, $a = 30.849$ (1), $b = 8.248$ (2), $c = 6.381$ (2) \AA , $V = 1623.4 \text{\AA}^3$, $Z = 4$, $D_x = 1.332 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71073 \text{\AA}$, $\mu = 2.689 \text{ cm}^{-1}$, $F(000) = 592$, $T = 183$ (1) K, $R = 0.0499$ for 810 observed reflections. Exhibiting non-crystallographic twofold rotation symmetry, the molecular structure displays effects of intramolecular strain associated with bulky substituent

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