

scan rate a function of count rate ($4.0^\circ \text{ min}^{-1}$ minimum, $30.0^\circ \text{ min}^{-1}$ maximum), 1793 reflections measured, 1514 unique, $R_{\text{int}} = 0.013$, 1400 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz and polarization effects. Empirical ellipsoidal absorption correction, max. and min. transmission = 0.57, 0.22, respectively. Structure solved by direct methods. The least-squares refinement used program *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.00030$. Secondary-extinction parameter $p = 0.023$ (2) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin^2\theta]^{0.25}$. 149 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for H atoms, methyl H atoms included using riding model, C—H = 0.96 Å, H—C—H = 109.5° , $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. $(\Delta/\sigma)_{\text{max}} = 0.003$, $R = 0.047$, $wR = 0.060$, $S = 2.468$. Final difference Fourier excursions 0.43 and -0.41 e \AA^{-3} . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).† Table 1 gives atom coordinates, and Table 2 bond distances and angles. Atom numbering follows that shown in Fig. 1.

† 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 43001 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a 1,2-Diazepine Analogue of a Diaza[2₄](1,2,4,5)cyclophane*

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Abstract. Ethyl 1,2,5,6,9,10,13,14-octahydro-4,11-metheno-3,12-nitrilo-7*H*-dicycloocta[*c,f*][1,2]diazepine-7-carboxylate, $\text{C}_{21}\text{H}_{23}\text{N}_3\text{O}_2$, $M_r = 349.4$, monoclinic, $P2_1$, $a = 10.727$ (1), $b = 9.581$ (1), $c = 8.498$ (1) Å, $\beta = 106.40$ (1)°, $V = 837.9$ Å³, $Z = 2$, $D_m = 1.37$, $D_x = 1.384$ g cm⁻³, Cu $K\alpha_1$, $\lambda = 1.54056$ Å, $\mu = 7.3$ cm⁻¹, $F(000) = 372$, $T = 296$ K, final $R = 0.051$ for 1267 unique observed reflections. The analysis confirms the proposed formulation. Inter-ring repulsion is inferred from bridging bond lengths as long as 1.578 (7) Å. The diazepine ring is a distorted boat; the four bridgehead atoms are roughly

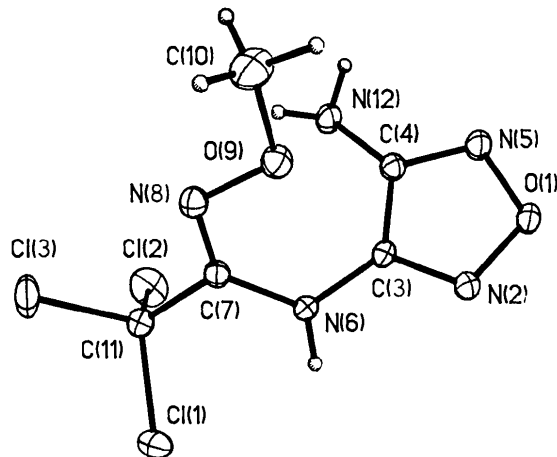


Fig. 1. Thermal-ellipsoid plot of title compound with ellipsoids drawn at 20% probability level. Bonds between C(7)—N(8), N(2)—C(3) and C(4)—N(5) are formally double bonds.

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coplanar, and the three remaining atoms are bent away from the opposing pyridine ring.

Experimental. Glassy, orange crystal of irregular shape, $0.2 \times 0.3 \times 0.4$ mm, D_m by flotation in aqueous potassium iodide solution. Picker four-circle diffractometer with Ni-filtered Cu $K\alpha$ radiation, θ - 2θ scan, $2\theta \leq 130^\circ$. Cell constants from 30 reflections, $50 \leq \theta \leq 65^\circ$. Max. $(\sin\theta)/\lambda = 0.588$ Å⁻¹, $h = -12$ to 12, $k = 0$ to 11, $l = 0$ to 9. No absorption corrections. One standard reflection (040) measured every 60 reflections, mean intensity 100 466 (671) counts; no indication of specimen decay. 1523 unique reflections scanned, of which 256 unobserved [$I_{\text{net}} \leq 3\sigma(I_{\text{net}})$]; σ

* NRCC No. 25907.

from counting statistics]. Net intensities derived by analysis of individual reflection profiles (Grant & Gabe, 1978). Structure solved by direct methods (*MULTAN78*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Refined by block-diagonal least squares, minimizing $\sum w\Delta F^2$, $w^{-1} = \sigma^2(F_o) + 0.001 F_o^2$. H atoms assigned reasonable positions (C-H = 1.08 Å) and isotropic temperature factors. Parameters refined were scale factor, secondary-extinction coefficient (1330; Larson, 1970), atomic coordinates and temperature factors for C, N and O. Final $R = 0.051$ (0.063 including unobserved reflections), $wR = 0.073$, $S = 1.83$. Ratio of the max. shift to e.s.d. in final cycle 0.03. Density in the final difference Fourier synthesis did not exceed $\pm 0.2 \text{ e } \text{Å}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computer program system described by Larson & Gabe (1978). The structure is defined in Fig. 1 and in Tables 1 and 2.*

Related literature. The synthesis of the material is described by Kang & Boekelheide (1984). The account includes a brief description of the molecular geometry, attributed to Hanson (private communication).

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43086 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

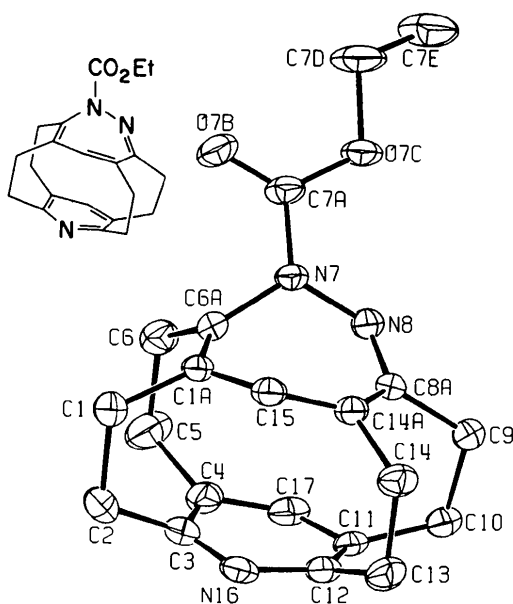


Fig. 1. A view of the molecule, showing thermal ellipsoids of 50% probability (Johnson, 1971), H atoms omitted.

Table 1. Atomic coordinates (e.s.d.'s) and equivalent isotropic temperature factors for the non-H atoms

B_{eq} is the arithmetic mean of the principal axes of the thermal ellipsoid.

	x	y	z	$B_{eq}(\text{Å}^2)$
C(1)	0.8547 (5)	-0.0595 (5)	0.6541 (7)	4.9
C(1A)	0.9201 (4)	0.0786 (4)	0.6417 (6)	3.6
C(2)	0.7179 (6)	-0.0458 (6)	0.6846 (10)	7.4
C(3)	0.6876 (4)	0.1032 (6)	0.6969 (7)	5.0
C(4)	0.7510 (4)	0.1839 (6)	0.8375 (7)	5.0
C(5)	0.8565 (6)	0.1225 (8)	0.9786 (8)	7.1
C(6)	0.9900 (5)	0.1121 (6)	0.9486 (6)	5.1
C(6A)	0.9947 (4)	0.1417 (5)	0.7754 (6)	3.8
N(7)	1.0686 (3)	0.2589 (4)	0.7469 (4)	3.5
C(7A)	1.1998 (4)	0.2526 (5)	0.8131 (6)	4.0
O(7B)	1.2566 (3)	0.1484 (4)	0.8730 (5)	5.6
O(7C)	1.2588 (3)	0.3719 (4)	0.7969 (5)	5.3
C(7D)	1.4006 (5)	0.3692 (8)	0.8597 (11)	8.5
C(7E)	1.4524 (5)	0.5000 (8)	0.9049 (10)	8.0
N(8)	1.0061 (3)	0.3905*	0.7225 (4)	3.6
C(8A)	0.9046 (3)	0.3993 (4)	0.5944 (5)	3.3
C(9)	0.8393 (4)	0.5397 (5)	0.5831 (6)	4.1
C(10)	0.7167 (4)	0.5369 (5)	0.6490 (7)	4.5
C(11)	0.6805 (3)	0.3884 (5)	0.6719 (5)	3.7
C(12)	0.6285 (4)	0.3002 (5)	0.5407 (6)	3.9
C(13)	0.6109 (4)	0.3492 (6)	0.3659 (6)	4.9
C(14)	0.7430 (4)	0.3352 (6)	0.3199 (6)	4.4
C(14A)	0.8555 (4)	0.2925 (5)	0.4691 (5)	3.5
C(15)	0.8775 (4)	0.1530 (5)	0.4887 (5)	3.7
N(16)	0.6232 (3)	0.1609 (4)	0.5574 (5)	4.7
C(17)	0.7363 (4)	0.3268 (6)	0.8233 (6)	4.3

* Coordinate held constant during refinement.

Table 2. Bond lengths (Å) and angles (°) and their e.s.d.'s

C(1)—C(1A)	1.515 (7)	C(7A)—O(7C)	1.332 (6)
C(1)—C(2)	1.566 (8)	O(7C)—C(7D)	1.463 (5)
C(1A)—C(6A)	1.336 (6)	C(7D)—C(7E)	1.381 (10)
C(1A)—C(15)	1.439 (7)	N(8)—C(8A)	1.307 (5)
C(2)—C(3)	1.474 (8)	C(8A)—C(9)	1.507 (6)
C(3)—C(4)	1.424 (8)	C(8A)—C(14A)	1.463 (6)
C(3)—N(16)	1.314 (7)	C(9)—C(10)	1.568 (6)
C(4)—C(5)	1.516 (7)	C(10)—C(11)	1.502 (7)
C(4)—C(17)	1.380 (8)	C(11)—C(12)	1.385 (6)
C(5)—C(6)	1.527 (7)	C(11)—C(17)	1.388 (7)
C(6)—C(6A)	1.514 (7)	C(12)—C(13)	1.518 (7)
C(6A)—N(7)	1.434 (6)	C(12)—N(16)	1.345 (6)
N(7)—C(7A)	1.362 (5)	C(13)—C(14)	1.578 (7)
N(7)—N(8)	1.416 (5)	C(14)—C(14A)	1.539 (6)
C(7A)—O(7B)	1.205 (6)	C(14A)—C(15)	1.359 (6)
C(1A)—C(1)—C(2)	114.3 (4)	C(7A)—O(7C)—C(7D)	115.0 (4)
C(1)—C(1A)—C(6A)	120.9 (4)	O(7C)—C(7D)—C(7E)	112.4 (5)
C(1)—C(1A)—C(15)	117.4 (4)	N(7)—N(8)—C(8A)	115.4 (3)
C(6A)—C(1A)—C(15)	120.4 (4)	N(8)—C(8A)—C(9)	111.9 (4)
C(1)—C(2)—C(3)	109.2 (4)	N(8)—C(8A)—C(14A)	127.2 (4)
C(2)—C(3)—C(4)	121.5 (5)	C(9)—C(8A)—C(14A)	120.8 (4)
C(2)—C(3)—N(16)	114.4 (4)	C(8A)—C(9)—C(10)	112.6 (4)
C(4)—C(3)—N(16)	122.2 (5)	C(9)—C(10)—C(11)	109.6 (4)
C(3)—C(4)—C(5)	121.7 (5)	C(10)—C(11)—C(12)	122.3 (4)
C(3)—C(4)—C(17)	116.7 (4)	C(10)—C(11)—C(17)	117.6 (4)
C(5)—C(4)—C(17)	119.7 (5)	C(12)—C(11)—C(17)	117.2 (5)
C(4)—C(5)—C(6)	114.8 (4)	C(11)—C(12)—C(13)	120.6 (4)
C(5)—C(6)—C(6A)	116.1 (4)	C(11)—C(12)—N(16)	122.7 (4)
C(1A)—C(6A)—C(6)	124.5 (4)	C(13)—C(12)—N(16)	114.4 (4)
C(1A)—C(6A)—N(7)	116.0 (4)	C(12)—C(13)—C(14)	110.1 (3)
C(6)—C(6A)—N(7)	118.9 (4)	C(13)—C(14)—C(14A)	111.6 (4)
C(6A)—N(7)—C(7A)	116.8 (4)	C(8A)—C(14A)—C(14)	116.8 (4)
C(6A)—N(7)—N(8)	117.4 (3)	C(8A)—C(14A)—C(15)	125.7 (4)
C(7A)—N(7)—N(8)	119.4 (4)	C(14)—C(14A)—C(15)	115.5 (4)
N(7)—C(7A)—O(7B)	123.5 (4)	C(1A)—C(15)—C(14A)	126.7 (4)
N(7)—C(7A)—O(7C)	112.6 (4)	C(3)—N(16)—C(12)	119.0 (4)
O(7B)—C(7A)—O(7C)	123.8 (4)	C(4)—C(17)—C(11)	120.8 (5)

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Absolute Crystal Structure of an *N*-Acylsultam Derivative*

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Abstract. The title compound (I) was obtained by EtAlCl₂-promoted asymmetric intramolecular Diels–Alder reaction of the camphorsultam derivative (*E,E*)-10,10-dimethyl-4-(2,8,10-undecatrienyl)-3-thia-4-azatricyclo[5.2.1.0^{1,5}]decane 3,3-dioxide (II); reduction of (I) gave the enantiomerically pure bicyclic alcohol 1,2,4a,5,6,7,8,8a-octahydro-1-naphthylmethanol (III) with regeneration of the unsubstituted chiral sultam precursor (IV) [Oppolzer & Dupuis (1985). *Tetrahedron Lett.* **26**, 44, 5437–5440]. (I): C₂₁H₂₃N₃O₂, *M_r* = 377.6, orthorhombic, *P*2₁2₁2₁, *a* = 8.0498 (12), *b* = 11.973 (2), *c* = 20.714 (3) Å, *V* = 1996.4 (5) Å³, *Z* = 4, *D_x* = 1.256 Mg m⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 1.735 cm⁻¹, *F*(000) = 816, m.p. 477–479 K, [α]_D²⁰ = -121.0° (*c* = 0.51% in CHCl₃), room temperature, *R* = 0.052 for 963 observed reflections [*|F_o*| ≥ 4σ(*F_o*) and *|F_o*| ≥ 8.0]. The chirality of enantiomerically pure (I) was confirmed from the known absolute configuration of the camphorsultam moiety as well as by least-squares refinement of the absolute-structure parameter *x* = 0.05 (31) [Bernardinelli & Flack (1985). *Acta Cryst.* **A41**, 500–511].

* (1*S*,5*R*,5'*R*)-10,10-Dimethyl-4-(1,2,3,4,4aβ,5,6,8aα-octahydro-1β-naphthyl)-3-thia-4-azatricyclo[5.2.1.0^{1,5}]decane 3,3-dioxide.

Experimental. Single crystals of (I) grown from methanol solution at room temperature. Colourless crystal of average dimensions 0.11 × 0.30 × 0.50 mm; Philips PW 1100 diffractometer, graphite-monochromated Mo *K*α; room temperature; cell dimensions from 24 reflections [2θ = 22–34°]; data collection: sinθ/λ ≤ 0.49 Å⁻¹, *h* 0–7, *k* 0–11, *l* 0–19; ω/2θ scans, ω-scan angle 1.2°; 2 standard reflections varied by max. of 1.6%; 1121 measured reflections; 963 observed with *|F|* ≥ 4σ(*F*) and *|F|* ≥ 8, Lorentz–

