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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): C₂₁H₃₁NO₃S, *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_c|* ≥ 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–

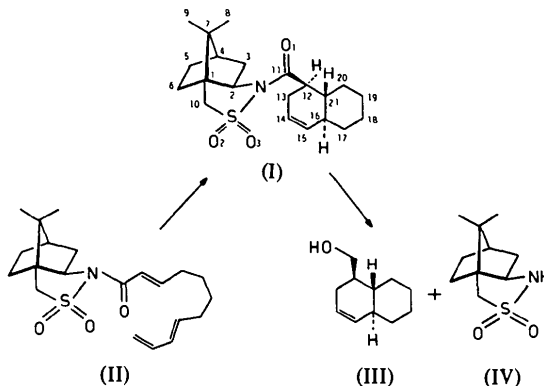


Table 1. Fractional coordinates and equivalent isotropic temperature factors, U_{eq} ($\text{\AA}^2 \times 10^3$), with e.s.d.'s in parentheses

U_{eq} is the average of the eigenvalues of U.				
	x	y	z	U_{eq}
S	0.00906 (23)	0.22815 (16)	0.71574 (8)	45.2 (7)
N	0.1762 (6)	0.2340 (5)	0.76599 (23)	37.8 (20)
O(1)	0.4506 (5)	0.2527 (5)	0.78171 (24)	64.6 (20)
O(2)	-0.0317 (6)	0.1148 (4)	0.7043 (3)	66.3 (21)
O(3)	-0.0363 (6)	0.2976 (4)	0.66073 (21)	62.1 (20)
C(1)	-0.0513 (8)	0.2889 (6)	0.8371 (3)	36.0 (24)
C(2)	0.1431 (8)	0.2904 (6)	0.8283 (3)	37 (3)
C(3)	0.2065 (8)	0.2339 (7)	0.8898 (3)	47 (3)
C(4)	0.0496 (9)	0.2253 (6)	0.9313 (3)	48 (3)
C(5)	-0.0094 (11)	0.3434 (6)	0.9482 (3)	58 (3)
C(6)	-0.0869 (10)	0.3851 (6)	0.8836 (3)	52 (3)
C(7)	-0.0799 (9)	0.1845 (6)	0.8812 (4)	46 (3)
C(8)	-0.0408 (11)	0.0684 (6)	0.8541 (4)	61 (3)
C(9)	-0.2590 (10)	0.1777 (8)	0.9078 (4)	73 (3)
C(10)	-0.1303 (8)	0.2930 (6)	0.7717 (3)	52 (3)
C(11)	0.3401 (8)	0.2231 (6)	0.7462 (3)	44.5 (23)
C(12)	0.3763 (8)	0.1687 (6)	0.6807 (3)	39.7 (24)
C(13)	0.4510 (9)	0.0522 (6)	0.6935 (3)	55 (3)
C(14)	0.5186 (11)	0.0029 (6)	0.6323 (4)	54 (3)
C(15)	0.5466 (10)	0.0615 (6)	0.5800 (4)	55 (3)
C(16)	0.5188 (9)	0.1858 (6)	0.5751 (3)	46 (3)
C(17)	0.6519 (11)	0.2449 (7)	0.5362 (4)	71 (3)
C(18)	0.6135 (12)	0.3705 (8)	0.5304 (5)	80 (4)
C(19)	0.5877 (11)	0.4221 (7)	0.5963 (5)	77 (4)
C(20)	0.4561 (9)	0.3616 (6)	0.6361 (4)	55 (3)
C(21)	0.5002 (9)	0.2374 (5)	0.6422 (3)	40.1 (23)

polarization; no absorption correction; systematic absences: $h00: h = 2n + 1$, $0k0: k = 2n + 1$, $00l: l = 2n + 1$; distribution of data $\langle E^2 - 1 \rangle = 0.794$ indicated non-centrosymmetric space group; structure solved by *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Two-block-matrix least squares using $|F|$ values. 237 parameters refined; no secondary-extinction correction; all coordinates of H atoms calculated. $R = 5.2\%$, $w(F) = 1$. Max. and av. ratio of $|\text{shift}|$ to e.s.d.: 0.66 and 0.07 respectively; max. and min. heights in final difference electron density map 0.40 and -0.33 e \AA^{-3} ; atomic scattering factors and anomalous-dispersion terms for S, N and O atoms from *International Tables for X-ray Crystallography* (1974); all calculations performed with a local version of *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) and *ORTEPII* (Johnson, 1976).^{*} Atomic coordinates are given in Table 1, bond lengths and torsional angles in Table 2 and a stereoview of the molecule in Fig. 1.

^{*} Lists of structure factors, atomic positional and thermal parameters for all atoms and other information in the printed form of the Standard Crystallographic File Structure of Brown (1985) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43103 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Interatomic distances (\AA) and selected torsional angles ($^\circ$) with e.s.d.'s in parentheses

S-N	1.703 (5)	C(5)-C(6)	1.559 (10)
S-O(2)	1.416 (6)	C(7)-C(8)	1.533 (10)
S-O(3)	1.427 (5)	C(7)-C(9)	1.545 (11)
S-C(10)	1.790 (7)	C(11)-C(12)	1.532 (10)
N-C(2)	1.481 (8)	C(12)-C(13)	1.542 (10)
N-C(11)	1.387 (8)	C(12)-C(21)	1.519 (9)
O(1)-C(11)	1.208 (8)	C(13)-C(14)	1.499 (11)
C(1)-C(2)	1.576 (9)	C(14)-C(15)	1.311 (11)
C(1)-C(6)	1.528 (10)	C(15)-C(16)	1.509 (10)
C(1)-C(7)	1.565 (10)	C(16)-C(17)	1.516 (11)
C(1)-C(10)	1.498 (9)	C(16)-C(21)	1.529 (9)
C(2)-C(3)	1.531 (10)	C(17)-C(18)	1.540 (12)
C(3)-C(4)	1.531 (10)	C(18)-C(19)	1.513 (14)
C(4)-C(5)	1.533 (10)	C(19)-C(20)	1.525 (12)
C(4)-C(7)	1.549 (10)	C(20)-C(21)	1.535 (9)
C(10)-S-N-C(2)	-2.8 (5)	C(13)-C(14)-C(15)-C(16)	1.7 (13)
N-S-C(10)-C(1)	-16.0 (5)	C(14)-C(15)-C(16)-C(21)	15.2 (11)
S-N-C(2)-C(1)	19.6 (6)	C(21)-C(16)-C(17)-C(18)	-57.1 (8)
S-N-C(11)-C(12)	-19.2 (9)	C(15)-C(16)-C(21)-C(12)	-48.6 (8)
C(10)-C(1)-C(2)-N	-30.8 (7)	C(15)-C(16)-C(21)-C(20)	-174.8 (6)
C(2)-C(1)-C(10)-S	29.1 (7)	C(17)-C(16)-C(21)-C(12)	-175.2 (6)
N-C(11)-C(12)-C(21)	132.8 (6)	C(16)-C(17)-C(18)-C(19)	54.0 (9)
O(1)-C(11)-C(12)-C(21)	-50.0 (9)	C(17)-C(18)-C(19)-C(20)	-54.1 (10)
C(21)-C(12)-C(13)-C(14)	-48.8 (8)	C(18)-C(19)-C(20)-C(21)	55.8 (9)
C(13)-C(12)-C(21)-C(16)	66.8 (7)	C(19)-C(20)-C(21)-C(16)	-56.7 (8)
C(12)-C(13)-C(14)-C(15)	15.4 (11)		

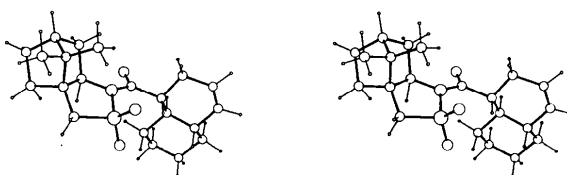


Fig. 1. Stereoview showing the configuration of the molecule.

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