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(7*S*)-*N*-(4-Phenyltricyclo[4.2.1.$0^{2,5}$]non-3-ene-3-carbonyl)-2,10-camphorsultam

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The regio- and absolute stereochemistry of the title compound, $C_{26}H_{31}NO_3S$, has been established by X-ray analysis. The molecular dimensions are in the normal range. In the crystal structure, weak C—H···O interactions link molecules, related by 2_1 screw axes, into extended tapes in the *b*-axis direction.

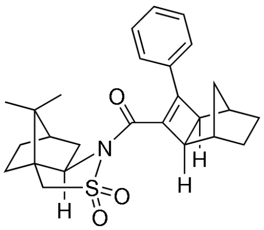
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.104
Data-to-parameter ratio = 17.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Recently, we reported the first examples of asymmetric ruthenium-catalyzed [2 + 2]-cycloadditions between bicyclic alkenes and a chiral acetylenic acyl sultam (Villeneuve & Tam, 2004). Two different diastereoisomers could be formed in the cycloadditions. When norbornene was used as the bicyclic alkene component and phenyl–acetylene–acyl camphorsultam was used as the alkyne component, two diastereoisomers were obtained in a 131:1 ratio. These diastereoisomers were separated by fractional recystallization. The absolute stereochemistry of the major isomer, (I), was established by the single-crystal X-ray diffraction analysis described here.



(I)

A view of the title compound is shown in Fig. 1. The structure determination establishes the absolute configuration of the following atoms in Fig. 1: C2-*S*, C5-*R*, C8-*R*, C14-*S*, C15-*R*, C18-*S* and C19-*R*. The molecular dimensions, a selection of which are listed in Table 1, are in the normal range. Weak C—H···O interactions, the geometric details of which are given in Table 2, link molecules, related by 2_1 screw axes, into extended tapes in the **b** direction (Fig. 2). The hydrogen-bonding motif consists of two main graph-set descriptors (Bernstein *et al.*, 1995); C(6) chains are formed *via* the C1—H1*B*···O3^{i} [symmetry code: (i) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$] interactions and $S_2^1(6)$ rings are fromed through the C3—H3*B*···O1^{ii} and C8—H8*A*···O1^{ii} [symmetry code: (ii) $1 - x$, $y - \frac{1}{2}$, $\frac{3}{2} - z$] interactions. Both of these interactions propagate along the same 2_1 screw axes, but in opposite directions with respect to the polarity of the C1—H1*B*···O3^{i} hydrogen bond.

Experimental

Addition of norbornene and (*S*)-phenyl–acetylene–acyl camphorsultam to Cp*RuCl(cyclooctadiene) in tetrahydrofuran at 298 K provided two diastereoisomers in a 131:1 ratio. Fractional recystallization in hexanes provided the major diastereoisomer, (I). Suitable crystals were grown from hexanes.

Crystal data

$C_{26}H_{31}NO_3S$
$M_r = 437.58$
Orthorhombic, $P2_12_12_1$
$a = 7.2089$ (1) Å
$b = 11.6317$ (2) Å
$c = 26.3219$ (5) Å
$V = 2207.14$ (6) Å^3
$Z = 4$
$D_x = 1.317$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 31 147 reflections
$\theta = 2.6$–27.5°
$\mu = 0.18$ mm^{-1}
$T = 150$ (1) K
Cut needle, colourless
0.26 × 0.20 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{\min} = 0.921$, $T_{\max} = 0.963$
18 496 measured reflections
4876 independent reflections
3962 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.071$
$\theta_{\max} = 27.5°$
$h = -9 \rightarrow 9$
$k = -15 \rightarrow 15$
$l = -34 \rightarrow 34$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.104$
$S = 1.04$
4876 reflections
281 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.0897P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0106 (16)
Absolute structure: Flack (1983); 1990 Friedel pairs
Flack parameter = −0.02 (7)

Table 1
Selected geometric parameters (Å, °).

S1—N1	1.6921 (17)	C12—C13	1.364 (3)
S1—C1	1.7885 (19)	C12—C19	1.517 (3)
N1—C11	1.407 (2)	C13—C14	1.516 (3)
N1—C8	1.486 (2)	C14—C19	1.573 (3)
O2—S1—O1	117.76 (12)	C12—C13—C14	93.26 (15)
N1—S1—C1	95.61 (9)	C13—C14—C19	86.69 (13)
C11—N1—C8	116.39 (15)	C12—C19—C14	85.40 (13)
C13—C12—C19	94.63 (14)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C1—H1B⋯O3$^{\rm i}$	0.99	2.41	3.361 (3)	161
C3—H3B⋯O1$^{\rm ii}$	0.99	2.51	3.377 (3)	146
C8—H8A⋯O1$^{\rm ii}$	1.00	2.50	3.425 (2)	153

Symmetry codes: (i) $1-x, \frac{1}{2}+y, \frac{3}{2}-z$; (ii) $1-x, y-\frac{1}{2}, \frac{3}{2}-z$.

All H atoms were placed in calculated positions, with C—H distances ranging from 0.95 to 1.00 Å, and included in the refinement in a riding-model approximation, with $U_{\rm iso} = 1.2U_{\rm eq}$(C) ($1.5U_{\rm eq}$ for methyl).

Data collection: *COLLECT* (Nonius, 1997–2002); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

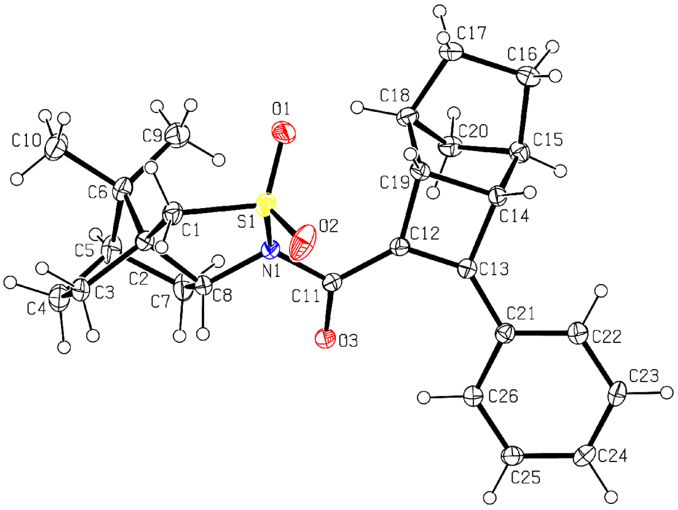


Figure 1
View of the molecule of (I), showing the crystallographic labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

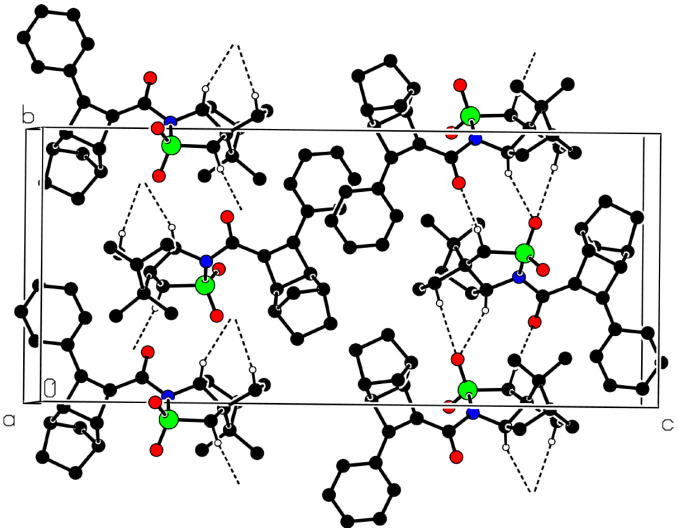


Figure 2
Packing diagram (Spek, 2004) showing weak intermolecular C—H⋯O interactions as dashed lines. S atoms are shown in green, O atoms in red, and N atoms in blue. All H atoms except H1B, H3B and H8A have been omitted for clarity

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