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(1*S*,2*R*)-*N*-[(*E*)-4-Methoxycinnamoyl]-10,2-camphorsultam

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The title compound, $C_{20}H_{25}NO_4S$, is the precursor molecule for the synthesis of (2*R*,3*R*)-2-(4-methoxyphenyl)-3-{1-[(3a*S*)-(3aα,6α,7aα)-hexahydro-8,8-dimethyl-3*H*-3a,6-methano-2,2-dioxo-2,1-benzoisothiazolyl]carbonyl}-4-isopropoxycarbonyl-5-methyl-2,2-dihydrofuran, which is selectively formed by an oxidative addition of a β-keto ester.

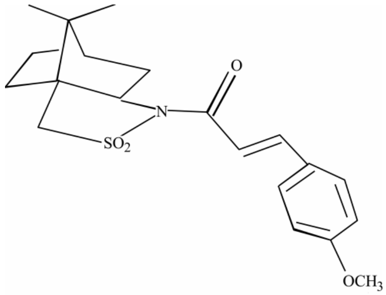
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.103
Data-to-parameter ratio = 16.9

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

Comment

The title compound, (I), was prepared by reaction between (*E*)-4-methoxy-cinnamoyl chloride and the sodium salt of (−)-10,2-camphorsultam (Oppolzer *et al.*, 1992). The starting camphorsultam is a commercial product (the pure 1*S*,2*R* enantiomer) with a specific rotation of −33° in ethyl alcohol for a concentration c = 1. The $Mn(OAc)_3$-induced radical addition of a β-keto ester to (1) gives, preferentially, a major diastereoisomer of a chiral dihydrofuran possessing two newly formed adjacent stereogenic centres (Garzino *et al.*, 2000). The specific rotation of the synthesized compound is −85.4° in chloroform for a concentration c = 1. The crystal structure of compound (I) was determined by X-ray analysis in order to understand the origin of the facial selectivity of this oxidative addition. It is known that in such radical reactions the transition state resembles the substrate (Curran *et al.*, 1996).



(I)

Experimental

Colourless crystals, shaped as needles, were obtained by evaporation of an ethanol solution.

Crystal data

$C_{20}H_{25}NO_4S$
M_r = 375.47
Monoclinic, $C2$
a = 10.147 (2) Å
b = 10.938 (2) Å
c = 17.681 (3) Å
β = 92.65 (3)°
V = 1960.3 (6) Å^3
Z = 4
D_x = 1.272 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8349 reflections
θ = 2.9–28.9°
μ = 0.19 mm^{-1}
T = 293 (2) K
Needle, colourless
0.39 × 0.22 × 0.19 mm

Data collection

Nonius KappaCCD diffractometer
φ scans
Absorption correction: none
8349 measured reflections
4046 independent reflections
3702 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.041$
$\theta_{\rm max} = 28.9^\circ$
$h = -12 \rightarrow 13$
$k = -14 \rightarrow 13$
$l = -23 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.103$
$S = 1.05$
4046 reflections
239 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.4741P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.17$ e Å^{-3}
Absolute structure: Flack (1983); 4107 Friedel pairs
Flack parameter = 0.06 (7)

Data collection: *DENZO* (Otwinowski & Minor, 1997); cell refinement: *DENZO*; data reduction: *DENZO*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *PLATON* (Spek, 1990).

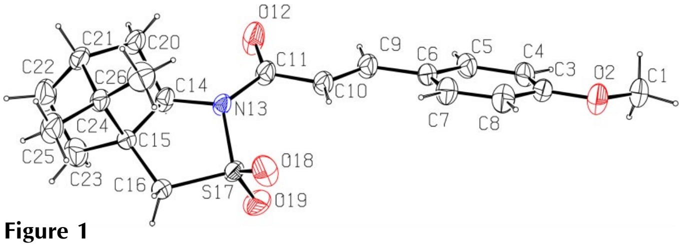


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (1) with displacement ellipsoids drawn at the 50% level. 1*S* and 2*R* correspond to atoms C15 and C14, respectively.

References

Curran, D. P., Porter, N. A. & Giese, B. (1996). *Stereochemistry of Radical Reactions*. Weinheim: VCH.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Garzino, F., Méou, A. & Brun, P. (2000). *Tetrahedron Lett.* **41**, 9803–9807.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-3974. Oak Ridge National Laboratory, Tennessee, USA.
Oppolzer, W., Tamura, O. & Deerberg, J. (1992). *Helv. Chim. Acta*, **75**, 1965–1978.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter and R. M. Sweet, pp. 307–326. London: Academic Press.
Sheldrick, G. M. (1997). *SHELX*97 and *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

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