C2—C3	1.490 (12)	C14—C15	1.363 (15)
C3—C4	1.398 (13)	C15—C16	1.375 (15)
C3—C8	1.406 (12)	C16—C17	1.396 (13)
C4—C5	1.374 (15)	C18—C19	1.388 (11)
C5—C6	1.378 (17)	C18—C23	1.390 (11)
C6—C7	1.376 (18)	C19—C20	1.382 (12)
C7—C8	1.399 (12)	C20—C21	1.374 (12)
C8—C9	1.476 (13)	C21—C22	1.377 (14)
C9—C10	1.473 (12)	C22—C23	1.389 (13)

Br1—C1—C2	112.7 (5)	C8—C9—C10	118.3 (8)
Br1—C1—C10	117.7 (6)	C1—C10—C9	119.1 (7)
Br1—C1—C11	119.0 (5)	C1—C10—C11	60.7 (5)
C2—C1—C10	118.8 (7)	C9—C10—C11	122.0 (7)
C2—C1—C11	119.4 (7)	C1—C11—C10	59.6 (5)
C10—C1—C11	59.6 (5)	C1—C11—C12	118.9 (7)
O1—C2—C1	122.0 (7)	C1—C11—C18	118.4 (6)
O1—C2—C3	120.8 (8)	C10—C11—C12	116.6 (7)
C1—C2—C3	117.2 (7)	C10—C11—C18	121.1 (7)
C2—C3—C4	118.0 (8)	C12—C11—C18	112.6 (7)
C2—C3—C8	121.8 (8)	C11—C12—C13	120.9 (8)
C3—C8—C9	121.5 (8)	C11—C12—C17	119.5 (7)
C7—C8—C9	120.4 (9)	C11—C18—C19	121.1 (7)
O2—C9—C8	122.5 (8)	C11—C18—C23	119.7 (7)
O2—C9—C10	119.3 (8)		

Br1—C1—C2—O1	23.3 (7)	C3—C8—C9—O2	165.3 (13)
Br1—C1—C2—C3	−155.5 (9)	C3—C8—C9—C10	−14.3 (8)
Br1—C1—C10—C9	138.4 (9)	C7—C8—C9—O2	−13.6 (9)
Br1—C1—C10—C11	−109.2 (7)	C7—C8—C9—C10	166.8 (12)
Br1—C1—C11—C10	106.9 (7)	O2—C9—C10—C1	−163.2 (12)
Br1—C1—C11—C12	1.2 (6)	O2—C9—C10—C11	125.0 (12)
Br1—C1—C11—C18	−141.8 (9)	C8—C9—C10—C1	16.4 (7)
C10—C1—C2—O1	166.8 (11)	C8—C9—C10—C11	−55.4 (9)
C10—C1—C2—C3	−12.0 (7)	C1—C10—C11—C1	0.0 (5)
C2—C1—C10—C9	−3.4 (7)	C1—C10—C11—C12	109.5 (8)
C2—C1—C10—C11	109.1 (8)	C1—C10—C11—C18	−106.9 (8)
C11—C1—C2—O1	−123.8 (11)	C9—C10—C11—C1	107.9 (9)
C11—C1—C2—C3	57.4 (8)	C9—C10—C11—C12	−142.6 (11)
C2—C1—C11—C10	−108.1 (8)	C9—C10—C11—C18	1.0 (7)
C2—C1—C11—C12	146.3 (10)	C1—C11—C12—C13	109.6 (10)
C2—C1—C11—C18	3.2 (7)	C1—C11—C12—C17	−74.9 (9)
C11—C1—C10—C9	−112.5 (8)	C1—C11—C18—C19	54.5 (8)
C10—C1—C11—C10	0.0 (5)	C1—C11—C18—C23	−130.5 (10)
C11—C1—C10—C11	0.0 (5)	C10—C11—C12—C13	41.3 (8)
C10—C1—C11—C12	−105.6 (8)	C10—C11—C12—C17	−143.3 (11)
C10—C1—C11—C18	111.3 (8)	C10—C11—C18—C19	124.3 (10)
O1—C2—C3—C4	13.5 (8)	C10—C11—C18—C23	−60.7 (8)
O1—C2—C3—C8	−163.9 (12)	C18—C11—C12—C13	−105.4 (10)
C1—C2—C3—C4	−167.6 (11)	C18—C11—C12—C17	70.1 (9)
C1—C2—C3—C8	14.9 (8)	C12—C11—C18—C19	−90.8 (9)
C2—C3—C4—C5	−178.2 (13)	C12—C11—C18—C23	84.3 (9)
C2—C3—C8—C7	177.2 (13)	C11—C12—C13—C14	177.5 (13)
C2—C3—C8—C9	−1.8 (8)	C11—C12—C17—C16	−177.2 (12)
C4—C3—C8—C9	−179.1 (13)	C11—C18—C19—C20	176.0 (12)
C6—C7—C8—C9	179.3 (14)	C11—C18—C23—C22	−176.5 (12)

The highest residual electron-density peak is located near Br1.

Data collection: *CRYSTAN* (Edwards *et al.*, 1995). Cell refinement: *CRYSTAN*. Data reduction: *CRYSTAN*. Program(s) used to solve structure: *CRYSTAN*. Program(s) used to refine structure: *CRYSTAN*. Molecular graphics: *CRYSTAN*. Software used to prepare material for publication: *CRYSTAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1073). Services for accessing these data are described at the back of the journal.

References

Allen, F. H. (1980). *Acta Cryst.* B**36**, 81–96.

Brown, H. C. & Okamoto, Y. (1958). *J. Am. Chem. Soc.* **80**, 4979–4987.

CAChe Scientific (1995). *CAChe Work-System*. Version 3.8. CAChe Scientific, Inc., Beaverton, Oregon, USA.

Crabb, T. A. & Patel, A. V. (1992). *Rodd's Chemistry of Carbon Compounds*, 2nd Suppl. to the 2nd ed., edited by M. Sainsbury, Vol. II*A*/*B*, ch. 1, pp. 1–80. Amsterdam: Elsevier.

Edwards, C., Gilmore, C. J., Mackay, S. & Stewart, N. (1995). *CRYSTAN. A Computer Program for the Solution and Refinement of Crystal Structures*. MacScience, Yokohama, Japan.

Meijere, A. (1979). *Angew. Chem. Int. Ed. Engl.* **18**, 809–826.

Oshima, T., Tamada, K. & Nagai, T. (1994). *J. Chem. Soc. Perkin Trans.* 1, pp. 3325–3333.

Tidwell, T. T. (1985). *The Chemistry of the Cyclopropyl Group*, edited by Z. Rappoport, pp. 565–632. New York: John Wiley & Sons.

Wong, H. N. C., Hon, M.-Y., Tse, C.-W. & Yip, Y.-C. (1989). *Chem. Rev.* **89**, 165–198.

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Absolute structure of an *N*-pentenoyl benzisothiazole

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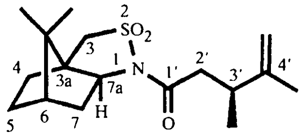
Abstract

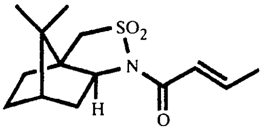
The title compound, (+)-(3a*R*,6*S*,7a*S*)-1-(8,8-dimethyl-2,2-dioxo-1,3,3a,4,5,6,7,7a-octahydro-3a,6-methano-2,1-benzisothiazol-1-yl)-3,4-dimethyl-4-penten-1-one, $C_{17}H_{27}NO_3S$, was isolated as the major product from a copper-mediated conjugate addition reaction. The absolute stereochemistry of the *N*-pentenoyl side chain was established. The latter is extended and the cyclohexane ring adopts a boat conformation, whilst the benzisothiazole ring is between a half-chair and envelope.

Comment

As part of a project on the synthesis of a marine natural product, a diastereoselective conjugate addition was performed which resulted in the isolation of the title compound, (1), as a mixture of diastereoisomers (yield 53%). The conjugate addition was between the cuprate, generated *in situ* from 2-propenyl magnesium bromide and cuprous iodide, and the crotonyl sultam, (2). The major diastereoisomer of the title compound, $[\alpha]_D^{24}$ + 64° (c = 0.06 in $CHCl_3$), crystallised from a hexane solution of the product mixture. The minor diastereoisomer was easily removed as it remained dissolved in the mother liquors. The diastereoisomeric excess was approximately 50% (based on ^1H NMR integral ratios). An X-ray analysis of crystals of (1) was undertaken to establish the absolute stereochemistry of the new *N*-pentenoyl side-chain.



(1)



(2)

The structure of (1) is given in Fig. 1. This illustrates that the *N*-pentenoyl side-chain is extended, the torsion angles N1—C9—C10—C11, C9—C10—C11—C12 and C10—C11—C12—C13 being 150.1 (4), 171.6 (4) and 111.3 (6)°, respectively. The cyclohexane ring adopts a fairly regular boat conformation with asymmetry parameter (Duax & Norton, 1975) ΔC_s(C3a) 10.6°. The distortions from a regular boat are due to the C8 bridge from C3a to C6 and the fusion to the isothiazole ring. The latter assumes a conformation between a half-chair and an envelope as indicated by the pseudo-rotation parameter (Altona *et al.*, 1968) Δ = 12.0°, with φ_m = 32.5°. Selected geometric parameters are given in Table 1. There being no hydrogen-bonding donors in the structure, the molecules are held together in the crystal by van der Waals interactions only.

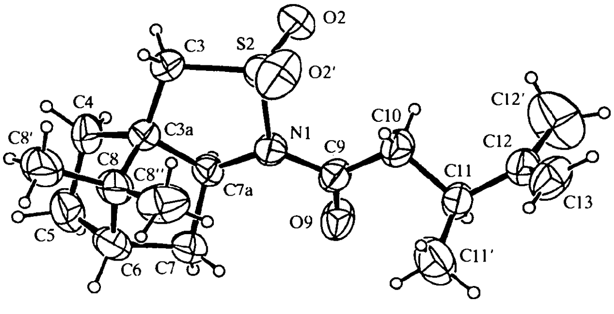


Fig. 1. Perspective view of the molecular structure of (1) with displacement ellipsoids scaled to 50% probability and atom numbering. The H atoms are denoted by spheres of an arbitrary radius.

Experimental

A suspension of CuI (0.93 g) in dry tetrahydrofuran (THF, 20 ml) was cooled to 213 K. A solution of 2-propenyl magnesium bromide (0.01 mol) in THF (5 ml) was slowly added to the suspension. The mixture was stirred at 213 K for 20 min, and then a solution of the crotonyl sultam, (2), (0.6 g, 2.1 mmol; Oppolzer & Barras, 1987) in dry THF (7 ml) was slowly added. The reaction mixture was stirred at 253 K for 3 h and the bright yellow solution was quenched by the addition of a saturated aqueous solution of ammonium chloride and then extracted with ether. The ethereal extracts were washed with brine, dried ($CaSO_4$), filtered and concentrated under reduced pressure to give the *N*-pentenoyl sultam as a colourless solid which was a mixture of diastereoisomers (0.38 g, yield 53%). Recrystallization of a sample of the mixture from hexane afforded the major sultam as a single diastereoisomer, (1).

Crystal data

$C_{17}H_{27}NO_3S$
M_r = 325.5
Orthorhombic
$P2_12_12_1$
a = 7.922 (2) Å
b = 8.198 (2) Å
c = 26.580 (4) Å
V = 1726.2 (7) Å^3
Z = 4
D_x = 1.252 Mg m^{-3}
D_m = 1.25 (1) Mg m^{-3}
D_m measured by flotation in aqueous $K_2[HgI_4]$

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 20.3–30.8°
μ = 1.76 mm^{-1}
T = 293 (2) K
Prismatic
0.48 × 0.32 × 0.32 mm
Colourless

Data collection

Rigaku/AFC diffractometer
$\omega/2\theta$ scans
Absorption correction: Gaussian (*SHELX*76; Sheldrick, 1976)
T_{min} = 0.539, T_{max} = 0.628
2031 measured reflections
1695 independent reflections
1562 reflections with $I > 2\sigma(I)$

R_{int} = 0.029
θ_{max} = 64.8°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 31$
3 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.152
S = 1.055
1695 reflections
206 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.1092P)^2 + 0.5883P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.014
$\Delta\rho_{max}$ = 0.235 e Å^{-3}
$\Delta\rho_{min}$ = −0.349 e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0058 (9)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.01 (4)

Table 1. *Selected geometric parameters (Å, °)*

N1—C9	1.387 (5)	O2—S2	1.437 (4)
N1—C7a	1.464 (5)	O2′—S2	1.422 (4)
N1—S2	1.694 (3)	S2—C3	1.795 (4)
C9—N1—C7a	119.1 (3)	O2—S2—N1	108.9 (2)
C9—N1—S2	123.0 (3)	O2′—S2—C3	111.4 (3)
C7a—N1—S2	112.2 (3)	O2—S2—C3	111.0 (2)
O2′—S2—O2	118.0 (3)	N1—S2—C3	96.0 (2)
O2′—S2—N1	109.2 (2)		
N1—C9—C10—C11	150.1 (4)	S2—N1—C7a—C3a	−27.6 (4)
C9—C10—C11—C12	171.6 (4)	C7a—N1—S2—C3	12.6 (3)
C10—C11—C12—C13	111.3 (6)	C3a—C4—C5—C6	3.8 (5)
O9—C9—C10—C11	−29.2 (6)	C4—C5—C6—C7	−74.9 (5)
C11′—C11—C12—C13	−13.6 (8)	C5—C6—C7—C7a	65.3 (5)
N1—S2—C3—C3a	6.6 (3)	C6—C7—C7a—C3a	9.3 (5)
S2—C3—C3a—C7a	−23.0 (4)	C4—C3a—C7a—C7	−79.4 (4)
C3—C3a—C7a—N1	32.2 (5)	C7a—C3a—C4—C5	69.7 (5)

The non-methyl and methyl-H atoms were given refined isotropic displacement factors of 0.083 (5) and 0.110 (7) Å^2, respectively. 336 equivalent reflections within the range h −9 to −6, k 0 to 9, l −31 to −1 were measured.

Data collection: Rigaku/AFC software (Rigaku, 1974). Cell refinement: Rigaku/AFC software. Data reduction: Rigaku/AFC software. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OB1002). Services for accessing these data are described at the back of the journal.

References

Altona, C., Geise, H. J. & Romers, C. (1968). *Tetrahedron*, **24**, 13–32.
Duax, W. L. & Norton, D. A. (1975). *Atlas of Steroid Structure*, Vol. 1, pp. 18–199. New York: Plenum Press.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Oppolzer, W. & Barras, J.-P. (1987). *Helv. Chim. Acta*, **70**, 1666–1675.
Rigaku (1974). Rigaku/AFC software. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination*. University of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. University of Göttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

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(*R*)-*N*-(1-Hydroxybut-2-yl)pyrrolidine-2,5-dione

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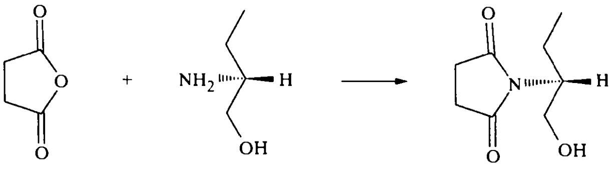
Abstract

The asymmetric unit of the title compound, $C_8H_{13}NO_3$, presents two independent molecules that give two intermolecular hydrogen bonds; the hydroxyl group of the first molecule is both acceptor and donor; that of the second one links to a carbonyl-O atom. This packing accounts for the small differences between the two independent molecules.

Comment

The title compound, (I), has been prepared in order to extend our previous study (Biju Kumar *et al.*, 1997) on the synthesis of novel ring systems and to obtain more information on the well known biological activity of succinimide derivatives (Khazhakyan & Khachaturyan, 1987).



(I)

The asymmetric unit of this compound contains two independent molecules. We gave both the same numbering, changing only the suffix (*A* and *B*). Fig. 1 shows molecule *A*. The molecular chirality was assigned on the grounds of the reactants' chirality (see Scheme). Selected bond lengths and angles are reported in Table 1. The differences between equivalent geometric parameters of the two independent molecules are mostly less than three s.u.'s. Relevant exceptions are observed for the parameters involving ethyl groups for which a remarkable apparent bond shortening is found [C3*B*—C4*B* 1.382 (10) *versus* C3*A*—C4*A* 1.499 (6) Å in molecules *B* and *A*, respectively], due to the very large anisotropic displacement parameters affecting C3*B* and C4*B*. This probably means a dynamical disorder of the ethyl group of molecule *B*, but no attempt to split the two atoms was carried out. Larger differences are found for torsion angles, the most relevant ones being reported in Table 1. These differences are most probably due to packing forces; in fact, the environments of

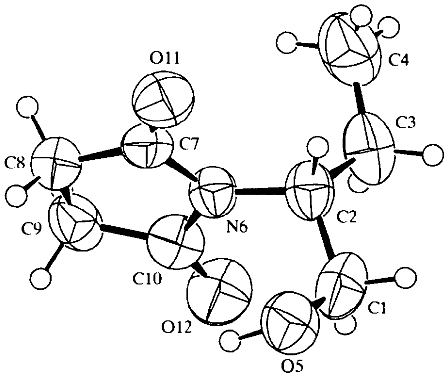


Fig. 1. *ORTEP*II (Johnson, 1976) plot of molecule *A* with 50% probability displacement ellipsoids. (The numbering scheme for molecule *B* is the same as for *A*.)