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(2*S*,3*S*)-2-Benzyl-3-(nosylamino)butano-4-lactone

Ilia A. Guzei,[a]* Joseph M. Langenhan[b] and Yong Jun Chung[b]

[a]2124 Chemistry Department, University of Wisconsin-Madison, 1101 University Ave, Madison, WI 53706, USA, and [b]Chemistry Department, University of Wisconsin-Madison, 1101 University Ave, Madison, WI 53706, USA

Correspondence e-mail: iguzei@chem.wisc.edu

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.029
wR factor = 0.073
Data-to-parameter ratio = 11.1

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

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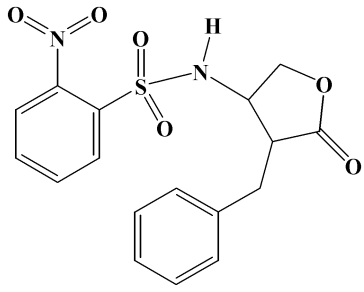
In the crystalline state, the title molecule, $C_{17}H_{16}N_2O_6S$, with its five-membered ring in an envelope conformation, produces a three-dimensional network of weak intermolecular hydrogen-bonding interactions.

Comment

During our studies aimed at α,β-disubstituted β-amino acids possessing sheet-forming propensity, we isolated and structurally characterized (2*S*,3*S*)-2-benzyl-3-(nosylamino)butano-4-lactone, (I), where nosyl is 2-nitrophenylsulfonyl.



(I)

Compound (I) crystallizes in the chiral space group $P2_12_12_1$ as discrete molecules with the *S* configuration about the two chiral centers C7 and C10 (Fig. 1). The five-membered heterocycle is in an envelope conformation, with C7 deviating from the C8/C9/C10/O5 plane by 0.409 (2) Å. The puckering amplitude q_2 is 0.257 (2) Å, while the phase angle φ_2 is 0.7 (4)° (Cremer & Pople, 1975); as expected, this corresponds to the ^1E conformation. The nitro group at C1 is not coplanar with the C1—C6 ring as the planar arrangement would bring atoms O1 and H2, as well as atoms O1 and O3, into close proximity, thereby inducing steric repulsion. The torsion angle O1—N1—C1—C2 measures 50.8 (2)°.

There are weak intermolecular hydrogen-bonding interactions in the crystal of (I), resulting in a three-dimensional network. These include N—H···O, C—H···O and C—H···π interactions (Table 2; Cg*A* is the centroid of the C1—C6 ring and Cg*B* is the centroid of the C12—C17 ring). In addition, the centroids of rings C1—C6 and C12—C17 are separated by 3.807 (1) Å, resulting in intramolecular π-stacking. The rings are almost parallel, with a dihedral angle between the ring planes of 13.03 (9)°.

The sum of the bond angles around N2 is 352.58 (13)°; thus atom N2 is essentially in an sp^2 configuration. A search of the Cambridge Structural Database (Allen & Kennard, 1993) revealed 24 molecules with similar S—N single bonds. The

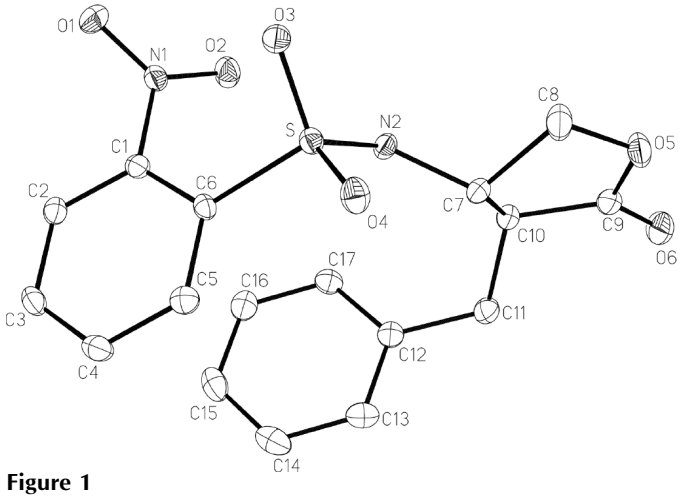


Figure 1
The molecular structure of (I) with displacement ellipsoids shown at the 30% probability level.

average bond length of 1.62 (2) Å somewhat exceeds the length of the S—N2 bond in (I) [1.5987 (16) Å] which is likely due to the increased sp^2 character of atom N2.

Experimental

(2*S*,3*S*)-2–Benzyl-3-(nosylamino)butano-4-lactone was synthesized from (3*S*)-3-(nosylamino)butano-4-lactone according to the procedure developed by Jefford & Wang (1993) and Jefford & McNulty (1994). To a solution of hexamethyldisilazane (HMDS, 2.3 equivalents) and *n*-BuLi (2.3 equivalents) at 195 K, (3*S*)-3-(nosylamino)-butano-4-lactone was added. Benzyl bromide (2.1 equivalents) was added to the resultant dianion to afford a mixture of products which, after chromatography, provided (I) in a 42% yield. ^{1}H NMR ($CDCl_3$ + DMSO, 296 K, p.p.m.): δ 8.30 (*s*, 1 H), 7.95–7.92 (*d*, 1 H), 7.80–7.69 (*m*, 2 H), 7.14–7.02 (*m*, 5 H), 4.19–3.99 (*AB* of *ABX*, 2H), 3.94–3.80 (*X* of *ABX*, 1 H), 3.12 (*m*, 1 H), 3.00–2.97 (*m*, 2 H); ^{13}C NMR ($CDCl_3$ + DMSO, 296 K, p.p.m.): δ 175.3, 136.3, 133.7, 133.4, 132.2, 130.3, 129.1, 128.2, 126.5, 124.6, 70.9, 52.8, 45.7, 32.7.

Crystal data

$C_{17}H_{16}N_2O_6S$
M_r = 376.38
Orthorhombic, $P2_12_12_1$
a = 7.3711 (5) Å
b = 11.0756 (7) Å
c = 20.5014 (12) Å
V = 1673.72 (18) Å^3
Z = 4
D_x = 1.494 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5380 reflections
θ = 2.0–26.0°
μ = 0.23 mm^{-1}
T = 173 (2) K
Block, colorless
0.50 × 0.40 × 0.30 mm

Data collection

Bruker CCD-1000 diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Blessing, 1995)
T_{min} = 0.893, T_{max} = 0.934
7922 measured reflections
3312 independent reflections
3081 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 26.4°
$h = -9 \rightarrow 8$
$k = -13 \rightarrow 13$
$l = -25 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.073
S = 1.03
3312 reflections
299 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 0.1922P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.04 (6)

Table 1
Selected bond lengths (Å).

S—O4	1.4277 (14)	S—C6	1.7891 (17)
S—O3	1.4391 (13)	O1—N1	1.2218 (19)
S—N2	1.5987 (16)	O2—N1	1.2203 (19)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2N···O3^{i}	0.78 (2)	2.19 (2)	2.917 (2)	154 (2)
C7—H7···O6^{ii}	0.867 (19)	2.532 (19)	3.269 (2)	143.5 (16)
C2—H2···CgB^{iiii}	0.93 (2)	2.66 (2)	3.4214 (18)	139.7 (17)
C14—H14···CgA^{iv}	0.97 (2)	3.17 (2)	3.936 (2)	136.8 (18)

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

H atoms were located from a difference map and both positional and isotropic displacement parameters were refined. For H atoms, the C—H range is 0.87 (2)–1.03 (2) Å and the N—H distance is 0.78 (2) Å.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1983, 1995).

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