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1-[(2-Naphthylsulfonyl)oxy]pyrrolidine-2,5-dione

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The title compound, $C_{14}H_{11}NO_5S$, is an ester of β-naphthylsulfonic acid and *N*-hydroxysuccinimide. The N atom retains a flattened pyramidal geometry. The C atoms of the succinic ring are coplanar and the N atom is slightly displaced from their plane, leading to a pseudo-envelope conformation. The crystal structure is stabilized by a three-dimensional network of weak C—H···O and offset π-stacking interactions.

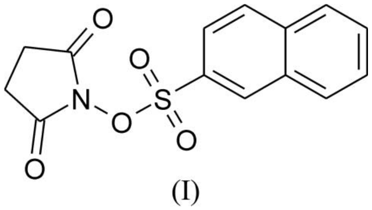
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.133
Data-to-parameter ratio = 30.4

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

Comment

The molecular structure and atom-numbering scheme of the title compound, (I), are presented in Fig. 1. The O1—N bond length [1.384 (1) Å] is comparable with the distances reported for non-tosylated *N*-hydroxyimides [mean value 1.383 (5) Å; Karolak-Wojciechowska *et al.*, 1993; Miao *et al.*, 1995], but slightly smaller than those reported for benzenesulfonic derivatives [1.395 (1)–1.401 (1) Å; Grigorieva & Chetkina, 1977]. The C11—N [1.392 (1) Å] and N—C14 [1.401 (1) Å] bond lengths are slightly longer than typical values for C—N succinimide bonds [*ca* 1.375 (5) Å; Karolak-Wojciechowska *et al.*, 1993]. The N atom is displaced from the C11/C14/O1 plane by 0.150 (1) Å and thus has a flattened pyramidal geometry, as may also be deduced from the sum of the O1—N—C14, O1—N—C11 and C14—N—C11 bond angles [356.6°; Table 2]. Such a situation was also observed for *N*-benzenesulfonyloxynaphthalimide (Grigorieva & Chetkina, 1977), but was not observed for almost-planar *N*-hydroxyimides (Karolak-Wojciechowska *et al.*, 1993; Miao *et al.*, 1995; Abell & Oldham, 1999) or slightly puckered *N*-halogensuccinimides (Brown, 1961).



(I)

The β-naphthyl system of (I) is nearly planar, the largest deviation from planarity being 0.019 (1) Å for atom C6. This slight deviation from planarity is reflected by a dihedral angle between the two fused rings of 1.30 (6)°.

The S atom has a distorted tetrahedral environment, as already observed for arylsulfonyl-related compounds (White *et al.*, 1970; Karapetyan *et al.*, 1998; Jones *et al.*, 1986). The expanded O2—S—O3 angle [121.0 (1)°] and the resulting O1—S—C2 angle [101.6 (1)°], smaller than the ideal tetrahedral value, are attributed to the Thorpe–Ingold effect (Bassindale, 1984).

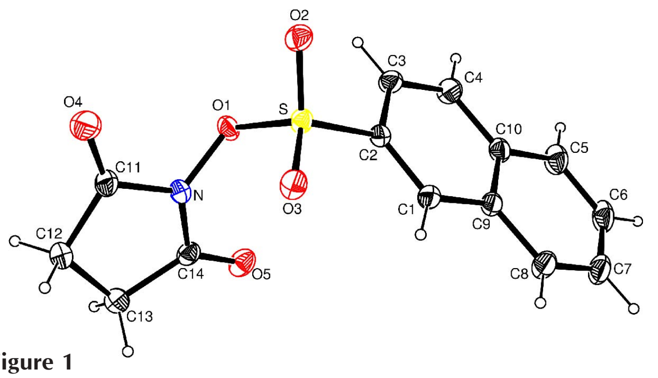


Figure 1
A view of the molecule of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

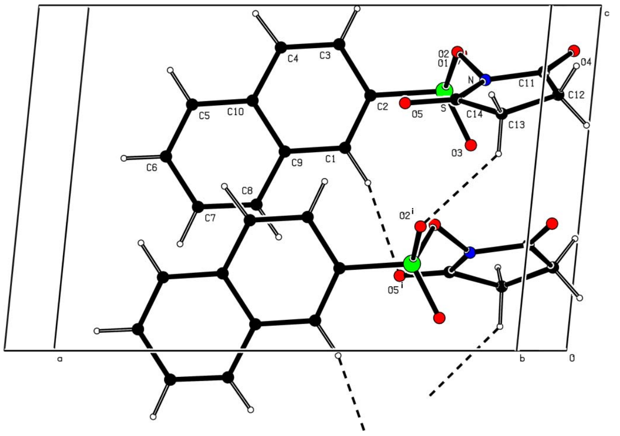


Figure 2
A view showing the packing of the molecules of (I) in the unit cell. Dashed lines indicate the short C—H···O contacts. [Symmetry code: (i) $x, \frac{3}{2} - y, z - \frac{1}{2}$.]

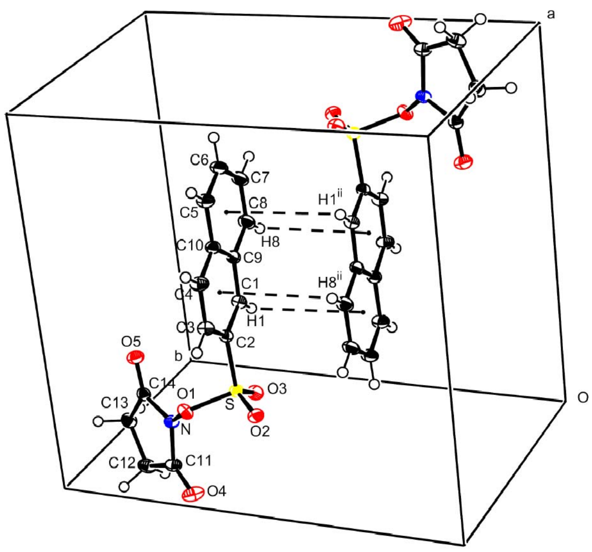


Figure 3
A view showing the offset π-stacking (dashed lines) between the naphthyl rings. [Symmetry code: (ii) $1 - x, 1 - y, 1 - z$.]

The crystal packing of (I) is characterized by two C—H···O-type hydrogen bonds (Table 2). Symmetry-related molecules are linked to each other in the *ac* plane, forming ribbons (Fig. 2). The β-naphthyl systems of adjacent ribbons are anti-parallel to each other and the distance between β-naphthyl planes is 3.536 (2) Å, resulting in an offset π-stacking interaction, with atoms H1 and H8 of the naphthyl ring lying over the ring centroids (Fig. 3).

Experimental

For the synthesis of compound (I), naphthalene-2-sulfonyl chloride (6.50 g, 28.7 mmol) and *N*-hydroxysuccinimide (3.30 g, 28.7 mmol) were dissolved in tetrahydrofuran (30 ml); triethylamine (1.7 ml) was then added dropwise over 20 min. After 40 min, the solvent was removed *in vacuo*, and distilled water (50 ml) with 3 drops of concentrated hydrochloric acid was added. The precipitate was filtered off, washed twice with water and recrystallized from ethyl acetate. The yield of compound (I) was 8.00 g (98%), m.p. 438 K.

Crystal data

$C_{14}H_{11}NO_5S$
$M_r = 305.31$
Monoclinic, $P2_1/c$
$a = 12.090$ (3) Å
$b = 13.403$ (3) Å
$c = 8.157$ (3) Å
$\beta = 95.79$ (3)°
$V = 1315.0$ (7) Å^3
$Z = 4$
$D_x = 1.542$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14 458 reflections
$\theta = 4.7$–38.0°
$\mu = 0.27$ mm^{-1}
$T = 100$ (2) K
Block, colourless
0.45 × 0.3 × 0.2 mm

Data collection

Oxford Xcalibur PX κ geometry diffractometer
ω and φ scans
Absorption correction: numerical (*CrysAlisRED*; Oxford Diffraction, 2003)
$T_{\min} = 0.896$, $T_{\max} = 0.952$
29 699 measured reflections
7118 independent reflections
5112 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$
$\theta_{\max} = 38.0°$
$h = -20 \rightarrow 20$
$k = -23 \rightarrow 21$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.133$
$S = 1.05$
7118 reflections
234 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0771P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.64$ e Å^{-3}
$\Delta\rho_{\min} = -0.52$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S—O3	1.4289 (10)	N—C11	1.3918 (13)
S—O2	1.4305 (8)	N—C14	1.4006 (13)
S—O1	1.6599 (8)	C1—C2	1.3701 (15)
O1—N	1.3842 (11)	C1—C9	1.4198 (14)
O1—N—C11	118.97 (8)	C11—N—C14	116.25 (8)
O1—N—C14	121.34 (8)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O5^i	0.98 (2)	2.31 (2)	3.275 (2)	166 (1)
C13—H13*A*···O2^i	0.96 (2)	2.54 (2)	3.316 (2)	138 (1)

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

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Data collection: *CrysAlisCCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlisRED* (Oxford Diffraction, 2003); data reduction: *CrysAlisRED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

References

Abell, A. D. & Oldham, M. D. (1999). *Bioorg. Med. Chem. Lett.* **9**, 497–500.

Bassindale, A. (1984). *The Third Dimension in Organic Chemistry*, ch.1, p. 11. New York: John Wiley & Sons.

Brown R. N. (1961). *Acta Cryst.* **14**, 711–715.

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Grigorieva, L. P. & Chetkina, L. A. (1977). *Zh. Strukt. Khim.* (*Russ. J. Struct. Chem.*), **18**, 908–916.

Jones, P. G., Edwards, M. R. & Kirby, A. J. (1986). *Acta Cryst.* C**42**, 1228–1230.

Karapetyan, A. A., Terzian, S. S. & Topuzian, V. O. (1998). *Zh. Strukt. Khim.* (*Russ. J. Struct. Chem.*), **39**, 973–977.

Karolak-Wojciechowska, J., Kwiatkowski, W. & Markowicz, W. (1993). *J. Cryst. Spectrosc. Res.* **23**, 423–429.

Miao, F. M., Wang, J. L. & Miao, X. S. (1995). *Acta Cryst.* C**51**, 712–713.

Oxford Diffraction (2003). *CrysAlisCCD* and *CrysAlisRED*. Versions 1.171. Oxford Diffraction Poland, Wrocław, Poland.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

White, E. H., Todd, M. J., Ribi, M., Ryan, T. J., Sieber, A. A. F., Dickerson, R. E. & Bordner, J. (1970). *Tetrahedron Lett.* **50**, 4467–4472.

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