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N-(2-Aminoethyl)-5-(dimethylamino)-naphthalene-1-sulfonamide

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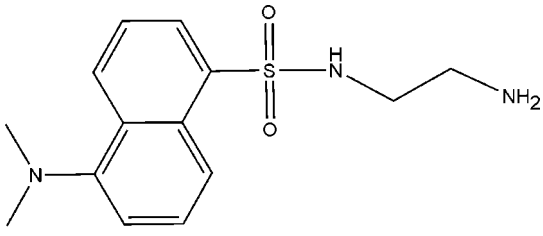
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.107; data-to-parameter ratio = 16.4.

In the title compound, $C_{14}H_{19}N_3O_2S$, the N atom of the dimethylamino group and the S atom are displaced by 0.078 (2) and 0.084 (2) Å, respectively, from the naphthalene ring plane. The 2-aminoethyl group has a coiled conformation with an N—C—C—NH_2 torsion angle of 53.6 (4)°. In the crystal structure, intermolecular N—H···N and weak C—H···O hydrogen bonds link molecules into chains along [001].

Related literature

For applications of ligands containing the 5-(dimethylamino)naphthalene-1-sulfonyl (dansyl) group, see: Corradini *et al.* (1996, 1997); Christoforou *et al.* (2006).



Experimental

Crystal data

$C_{14}H_{19}N_3O_2S$
M_r = 293.38
Orthorhombic, $Pna2_1$
a = 15.5221 (15) Å
b = 11.5423 (11) Å
c = 8.1360 (8) Å
V = 1457.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 298 K
0.20 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.956, T_{max} = 0.956
7478 measured reflections
3140 independent reflections
3012 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.107
S = 1.11
3140 reflections
192 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack (1983), 1332 Friedel pairs
Flack parameter: −0.03 (8)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O1	0.93	2.48	3.093 (3)	123
N3—H3A···N2	0.88 (5)	2.52 (6)	2.972 (4)	113 (4)
C11—H11···O1^{i}	0.93	2.49	3.146 (3)	128
N2—H2D···N3^{ii}	0.87 (3)	2.02 (4)	2.869 (4)	163 (3)

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2821).

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supporting information

Acta Cryst. (2009). E**65**, o1452 [doi:10.1107/S160053680901962X]

N-(2-Aminoethyl)-5-(dimethylamino)naphthalene-1-sulfonamide

Shi-lei Zhang, Bi-lin Zhao, Zhen-hong Su, Xian-you Xia and Yong Zhang

S1. Comment

The dansyl (5-(dimethylamino)naphthalene-1-sulfonyl) group has been widely used as a fluorophore in the design of fluorescent probes. Recently many fluorescent ligands bearing dansyl group have been reported (Corradini *et al.*, 1996,1997; Christoforou *et al.*, 2006). We are interested in preparing fluorescent ligands that are expected to bind to hydrophobic sites in proteins or membranes. With this mind, the title compound, (I), was prepared and we report the crystal stucture herein.

In the molecule (Fig. 1), atoms N1 and S1 are located approximately in the naphthalene ring plane with their deviations being 0.078 and 0.084 Å, respectively. The N2—C14—C15—N3 torsion angle of -53.6 (4)° indicates a coiled conformation for the aminoethyl group. In the crystal structure (Fig.2), intermolecular N—H···N and weak C-H···O hydrogen bonds link molecules into one-dimensional chains along [001].

S2. Experimental

Compound (I) was synthesized according to a literature procedure (Corradini *et al.*, 1996). Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a dichloromethane solution of (I) at room temperature.

S3. Refinement

All carbon bound H atoms were placed in their idealized positions [*C*—*H*(methyl)=0.96 Å and C—H(aromatic) =0.93 Å] and included in the refinement in the riding-model approximation, with U_{iso}(methyl H)= 1.5U_{eq}(C) and U_{iso}(aromatic H) = 1.2U_{eq}(C). Hydrogen atoms bonded to nitrogen atoms were found in the difference Fourier maps and refined with the constraints of N—H = 0.869(Å) and U_{iso}(H) = 1.2U_{eq}(N).

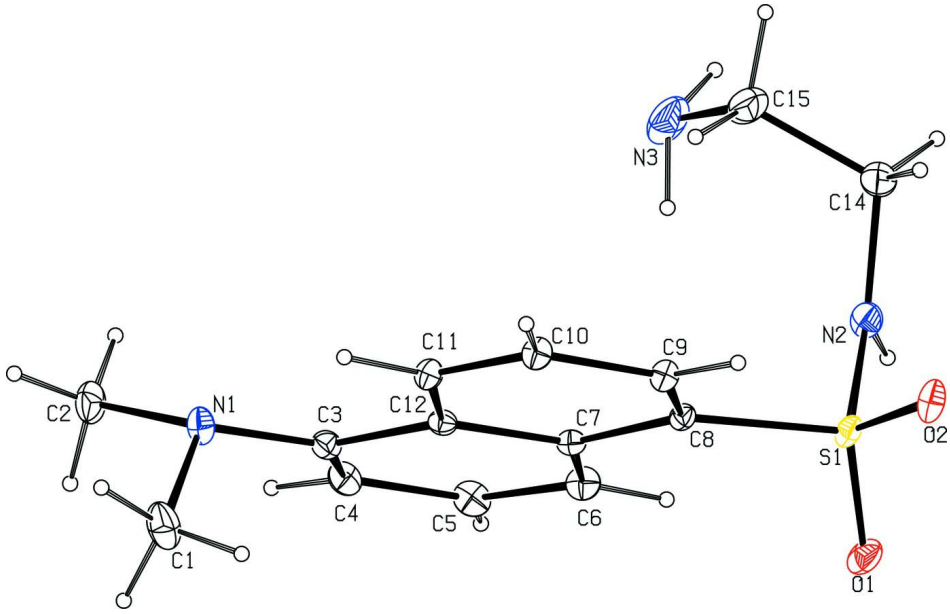

Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

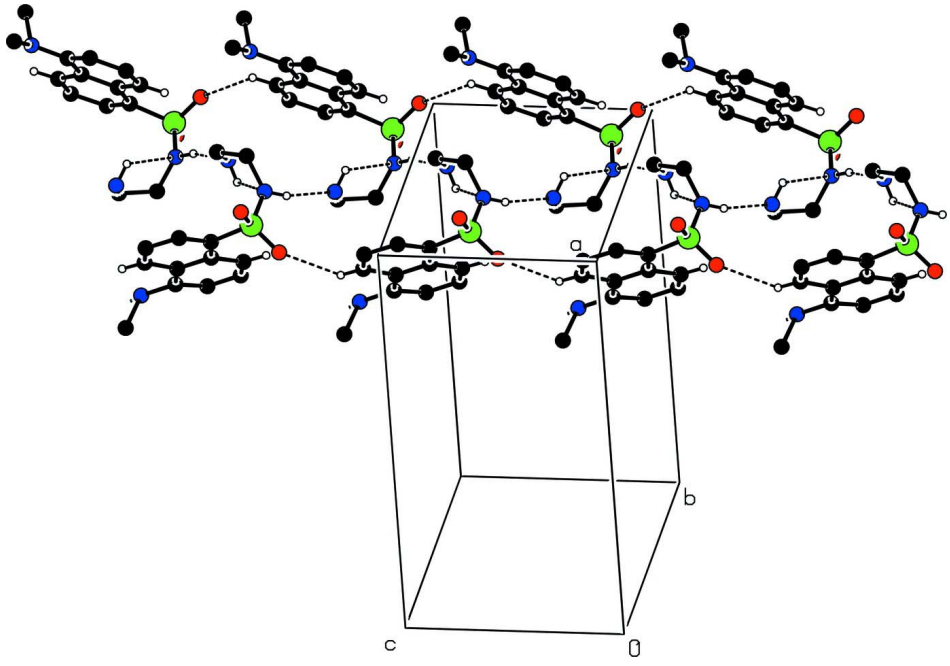

Figure 2

Part of the crystal structure of (I) showing weak hydrogen bonds as dashed lines. Only H atoms involved in hydrogen bonds are shown.

N-(2-Aminoethyl)-5-(dimethylamino)naphthalene-1-sulfonamide

Crystal data

$C_{14}H_{19}N_3O_2S$
$M_r = 293.38$
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a = 15.5221$ (15) Å
$b = 11.5423$ (11) Å
$c = 8.1360$ (8) Å
$V = 1457.7$ (2) Å^3
$Z = 4$
$F(000) = 624$
$D_x = 1.337$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4085 reflections
$\theta = 2.2$–$28.0°$
$\mu = 0.23$ mm^{-1}
$T = 298$ K
Block, colorless
0.20 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min} = 0.956$, $T_{max} = 0.956$
7478 measured reflections
3140 independent reflections
3012 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.2°$
$h = -19 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.107$
$S = 1.11$
3140 reflections
192 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2 + 0.0989P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Absolute structure: Flack (1983), 1332 Friedel pairs
Absolute structure parameter: −0.03 (8)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.6783 (2)	0.2592 (3)	1.2600 (5)	0.0711 (9)
H1A	0.6195	0.2620	1.2236	0.107*
H1B	0.6797	0.2519	1.3775	0.107*

H1C	0.7065	0.1937	1.2111	0.107*
C2	0.67195 (17)	0.4676 (3)	1.2579 (4)	0.0615 (7)
H2A	0.7030	0.5368	1.2300	0.092*
H2B	0.6616	0.4659	1.3742	0.092*
H2C	0.6180	0.4665	1.2004	0.092*
C3	0.75553 (12)	0.36601 (19)	1.0498 (3)	0.0381 (5)
C4	0.72410 (14)	0.43583 (19)	0.9255 (3)	0.0451 (5)
H4	0.6796	0.4874	0.9477	0.054*
C5	0.75833 (15)	0.4299 (2)	0.7670 (3)	0.0480 (6)
H5	0.7350	0.4767	0.6853	0.058*
C6	0.82454 (15)	0.3580 (2)	0.7281 (3)	0.0445 (5)
H6	0.8451	0.3547	0.6208	0.053*
C7	0.86251 (12)	0.28748 (17)	0.8530 (3)	0.0323 (4)
C8	0.93538 (13)	0.21356 (16)	0.8264 (3)	0.0309 (4)
C9	0.96977 (14)	0.14947 (18)	0.9519 (3)	0.0366 (4)
H9	1.0158	0.0999	0.9311	0.044*
C10	0.93643 (14)	0.15783 (18)	1.1106 (3)	0.0379 (4)
H10	0.9610	0.1151	1.1954	0.045*
C11	0.86816 (12)	0.22823 (16)	1.1418 (3)	0.0363 (4)
H11	0.8473	0.2345	1.2486	0.044*
C12	0.82814 (12)	0.29230 (16)	1.0143 (3)	0.0322 (4)
C14	1.10343 (19)	0.3731 (3)	0.6827 (4)	0.0689 (9)
H14A	1.1354	0.4225	0.6080	0.083*
H14B	1.1400	0.3079	0.7107	0.083*
C15	1.0839 (2)	0.4406 (3)	0.8375 (5)	0.0833 (11)
H15A	1.0609	0.3874	0.9188	0.100*
H15B	1.1375	0.4712	0.8805	0.100*
N1	0.72278 (13)	0.36630 (17)	1.2108 (3)	0.0467 (5)
O1	0.92413 (13)	0.18510 (17)	0.5084 (2)	0.0558 (5)
O2	1.05529 (12)	0.12231 (15)	0.6510 (2)	0.0553 (4)
N2	1.02722 (14)	0.33007 (18)	0.5987 (3)	0.0495 (5)
H2D	1.0054 (19)	0.358 (3)	0.508 (5)	0.059*
N3	1.0237 (3)	0.5358 (3)	0.8182 (5)	0.0993 (13)
H3A	0.980 (3)	0.499 (5)	0.773 (8)	0.119*
H3B	1.044 (3)	0.588 (4)	0.752 (7)	0.119*
S1	0.98740 (3)	0.20448 (4)	0.63277 (8)	0.03840 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0693 (17)	0.0655 (18)	0.079 (2)	0.0010 (15)	0.0311 (15)	0.0156 (17)
C2	0.0547 (14)	0.0663 (17)	0.0635 (18)	0.0210 (13)	0.0108 (12)	−0.0083 (14)
C3	0.0340 (10)	0.0365 (11)	0.0439 (12)	0.0001 (8)	−0.0005 (9)	−0.0006 (9)
C4	0.0377 (10)	0.0406 (12)	0.0570 (14)	0.0071 (9)	−0.0008 (10)	0.0082 (11)
C5	0.0472 (12)	0.0486 (13)	0.0482 (14)	0.0062 (10)	−0.0097 (10)	0.0171 (11)
C6	0.0533 (12)	0.0461 (12)	0.0343 (11)	−0.0003 (10)	−0.0053 (9)	0.0120 (10)
C7	0.0331 (9)	0.0315 (9)	0.0323 (10)	−0.0037 (7)	−0.0041 (8)	0.0023 (8)
C8	0.0369 (10)	0.0294 (9)	0.0264 (10)	−0.0018 (8)	0.0014 (8)	−0.0008 (8)

C9	0.0418 (10)	0.0344 (11)	0.0336 (10)	0.0061 (8)	−0.0015 (9)	0.0015 (8)
C10	0.0450 (10)	0.0387 (10)	0.0299 (11)	0.0066 (8)	−0.0045 (8)	0.0077 (8)
C11	0.0434 (10)	0.0367 (9)	0.0286 (9)	0.0018 (7)	0.0003 (9)	0.0037 (9)
C12	0.0328 (9)	0.0303 (10)	0.0335 (10)	−0.0049 (7)	−0.0015 (8)	0.0030 (8)
C14	0.0647 (16)	0.0609 (16)	0.081 (3)	−0.0208 (14)	0.0173 (15)	−0.0189 (15)
C15	0.099 (2)	0.077 (2)	0.075 (2)	−0.034 (2)	0.012 (2)	−0.0264 (19)
N1	0.0462 (10)	0.0458 (11)	0.0482 (11)	0.0089 (9)	0.0133 (9)	0.0015 (9)
O1	0.0778 (12)	0.0595 (11)	0.0302 (8)	−0.0089 (9)	−0.0003 (8)	−0.0045 (8)
O2	0.0729 (10)	0.0498 (9)	0.0432 (9)	0.0162 (8)	0.0174 (9)	−0.0028 (8)
N2	0.0641 (13)	0.0425 (10)	0.0420 (14)	−0.0092 (9)	0.0122 (9)	0.0037 (9)
N3	0.158 (4)	0.0527 (18)	0.087 (2)	−0.0261 (19)	0.036 (2)	−0.0248 (16)
S1	0.0538 (3)	0.0341 (2)	0.0274 (2)	−0.00129 (19)	0.0066 (3)	−0.0034 (3)

Geometric parameters (Å, º)

C1—N1	1.472 (3)	C8—S1	1.773 (2)
C1—H1A	0.9600	C9—C10	1.395 (3)
C1—H1B	0.9600	C9—H9	0.9300
C1—H1C	0.9600	C10—C11	1.359 (3)
C2—N1	1.461 (3)	C10—H10	0.9300
C2—H2A	0.9600	C11—C12	1.417 (3)
C2—H2B	0.9600	C11—H11	0.9300
C2—H2C	0.9600	C14—N2	1.454 (4)
C3—C4	1.382 (3)	C14—C15	1.511 (5)
C3—N1	1.405 (3)	C14—H14A	0.9700
C3—C12	1.441 (3)	C14—H14B	0.9700
C4—C5	1.396 (3)	C15—N3	1.450 (5)
C4—H4	0.9300	C15—H15A	0.9700
C5—C6	1.358 (3)	C15—H15B	0.9700
C5—H5	0.9300	O1—S1	1.4278 (19)
C6—C7	1.429 (3)	O2—S1	1.4255 (18)
C6—H6	0.9300	N2—S1	1.600 (2)
C7—C12	1.417 (3)	N2—H2D	0.87 (3)
C7—C8	1.433 (3)	N3—H3A	0.88 (5)
C8—C9	1.369 (3)	N3—H3B	0.87 (5)
N1—C1—H1A	109.5	C9—C10—H10	119.9
N1—C1—H1B	109.5	C10—C11—C12	121.1 (2)
H1A—C1—H1B	109.5	C10—C11—H11	119.4
N1—C1—H1C	109.5	C12—C11—H11	119.4
H1A—C1—H1C	109.5	C11—C12—C7	119.47 (18)
H1B—C1—H1C	109.5	C11—C12—C3	120.3 (2)
N1—C2—H2A	109.5	C7—C12—C3	120.20 (18)
N1—C2—H2B	109.5	N2—C14—C15	113.9 (3)
H2A—C2—H2B	109.5	N2—C14—H14A	108.8
N1—C2—H2C	109.5	C15—C14—H14A	108.8
H2A—C2—H2C	109.5	N2—C14—H14B	108.8
H2B—C2—H2C	109.5	C15—C14—H14B	108.8

C4—C3—N1	123.6 (2)	H14A—C14—H14B	107.7
C4—C3—C12	118.3 (2)	N3—C15—C14	115.5 (3)
N1—C3—C12	118.11 (19)	N3—C15—H15A	108.4
C3—C4—C5	120.8 (2)	C14—C15—H15A	108.4
C3—C4—H4	119.6	N3—C15—H15B	108.4
C5—C4—H4	119.6	C14—C15—H15B	108.4
C6—C5—C4	122.3 (2)	H15A—C15—H15B	107.5
C6—C5—H5	118.9	C3—N1—C2	116.2 (2)
C4—C5—H5	118.9	C3—N1—C1	114.9 (2)
C5—C6—C7	119.6 (2)	C2—N1—C1	110.3 (2)
C5—C6—H6	120.2	C14—N2—S1	122.8 (2)
C7—C6—H6	120.2	C14—N2—H2D	126 (2)
C12—C7—C6	118.73 (19)	S1—N2—H2D	109 (2)
C12—C7—C8	117.42 (17)	C15—N3—H3A	100 (3)
C6—C7—C8	123.8 (2)	C15—N3—H3B	111 (3)
C9—C8—C7	121.08 (19)	H3A—N3—H3B	111 (5)
C9—C8—S1	116.94 (16)	O2—S1—O1	118.56 (12)
C7—C8—S1	121.94 (15)	O2—S1—N2	109.58 (12)
C8—C9—C10	120.57 (19)	O1—S1—N2	106.55 (12)
C8—C9—H9	119.7	O2—S1—C8	106.46 (10)
C10—C9—H9	119.7	O1—S1—C8	109.01 (10)
C11—C10—C9	120.2 (2)	N2—S1—C8	106.04 (10)
C11—C10—H10	119.9		

N1—C3—C4—C5	−178.7 (2)	C4—C3—C12—C11	173.49 (19)
C12—C3—C4—C5	3.9 (3)	N1—C3—C12—C11	−4.0 (3)
C3—C4—C5—C6	−1.4 (4)	C4—C3—C12—C7	−3.6 (3)
C4—C5—C6—C7	−1.5 (4)	N1—C3—C12—C7	178.89 (18)
C5—C6—C7—C12	1.7 (3)	N2—C14—C15—N3	−53.6 (4)
C5—C6—C7—C8	−176.60 (19)	C4—C3—N1—C2	−18.4 (3)
C12—C7—C8—C9	0.6 (3)	C12—C3—N1—C2	159.0 (2)
C6—C7—C8—C9	178.9 (2)	C4—C3—N1—C1	112.6 (3)
C12—C7—C8—S1	−177.20 (14)	C12—C3—N1—C1	−70.0 (3)
C6—C7—C8—S1	1.1 (3)	C15—C14—N2—S1	−91.9 (3)
C7—C8—C9—C10	−2.3 (3)	C14—N2—S1—O2	−40.1 (2)
S1—C8—C9—C10	175.56 (16)	C14—N2—S1—O1	−169.58 (19)
C8—C9—C10—C11	1.3 (3)	C14—N2—S1—C8	74.4 (2)
C9—C10—C11—C12	1.5 (3)	C9—C8—S1—O2	3.06 (19)
C10—C11—C12—C7	−3.2 (3)	C7—C8—S1—O2	−179.09 (16)
C10—C11—C12—C3	179.68 (19)	C9—C8—S1—O1	132.05 (17)
C6—C7—C12—C11	−176.28 (19)	C7—C8—S1—O1	−50.11 (19)
C8—C7—C12—C11	2.1 (3)	C9—C8—S1—N2	−113.59 (18)
C6—C7—C12—C3	0.8 (3)	C7—C8—S1—N2	64.25 (19)
C8—C7—C12—C3	179.24 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O1	0.93	2.48	3.093 (3)	123
N3—H3*A*···N2	0.88 (5)	2.52 (6)	2.972 (4)	113 (4)
C11—H11···O1^{i}	0.93	2.49	3.146 (3)	128
N2—H2*D*···N3^{ii}	0.87 (3)	2.02 (4)	2.869 (4)	163 (3)

Symmetry codes: (i) *x*, *y*, *z*+1; (ii) −*x*+2, −*y*+1, *z*−1/2.