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Crystal structure of ethyl 2-(5-amino-1-benzenesulfonyl-3-oxo-2,3-dihydro-1*H*-pyrazol-2-yl)acetate

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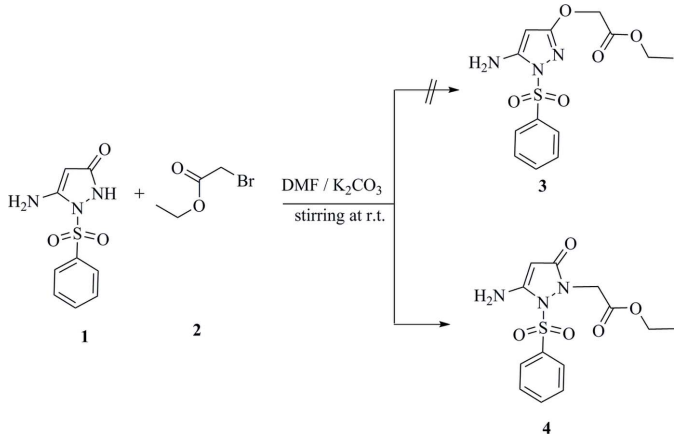
In the title compound, $C_{13}H_{15}N_3O_5S$, the two rings face each other in a 'V' form at the S atom, with one N—H···O=S and one C—H···O=S contact from the pyrazolyl substituents to the sulfonyl group. Two classical hydrogen bonds from the amine group, one of the form N—H···O=S and one N—H···O=C_{oxo}, link the molecules to form layers parallel to the *bc* plane.

1. Chemical context

We are interested in the development of innovative synthetic strategies for *N*-sulfonyl- and *N*-sulfonylamino-based heterocyclic ring systems that have found application as new antimicrobial and anti-viral agents (Azzam *et al.*, 2017, 2019*ab*; Elgemeie *et al.*, 2017, 2019; Zhu *et al.*, 2013). Michael *et al.* (2007) investigated the inhibition capabilities of a novel series of our reported *N*-sulfonylpyrazoles (Elgemeie *et al.*, 1998, 1999, 2013) towards the enzyme cathepsin B16. Shyama *et al.* (2009) also identified some of our reported *N*-arylsulfonylpyrazole series to be active inhibitors of the NS2B-NS3 virus. These promising results led our research group to investigate new approaches to other derivatives of *N*-sulfonylpyrazoles, thereby seeking alternative scaffolds for use as promising chemotherapeutics (Azzam & Elgemeie, 2019; Elgemeie & Jones, 2002; Zhang *et al.*, 2020). Accordingly, we synthesized the N-1-substituted derivative of *N*-sulfonylpyrazole **1**.



The reaction **1** with ethyl bromoacetate **2** in the presence of anhydrous potassium carbonate in dry *N*,*N*-dimethylformamide at room temperature produced an adduct for which two possible isomers, the *O*-alkylated or *N*-alkylated *N*-sulfonylpyrazole structures **3** or **4**, were considered. The ^{1}H NMR spectra of the product revealed the presence of an

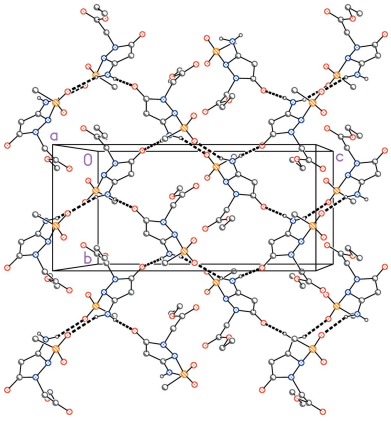


Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H01···O1	0.866 (19)	2.355 (19)	2.8296 (15)	114.8 (15)
N3—H01···O1^{i}	0.866 (19)	2.593 (19)	3.3644 (15)	148.8 (16)
N3—H02···O3^{ii}	0.871 (19)	1.961 (19)	2.8257 (15)	171.5 (17)
C12—H12A···O2	0.99	2.38	3.0214 (16)	122

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

amino group at 7.34 ppm and a pyrazole CH at 4.34 ppm, but spectroscopic data cannot differentiate between structures **3** and **4**. The crystal structure determination indicated unambiguously the formation of the *N*-alkylated *N*-sulfonylpyrazole **4** as the only product in the solid state.

2. Structural commentary

The structure analysis confirms the formation of compound **4** (Fig. 1). The molecule displays an intramolecular hydrogen bond of the form N—H···O═S, and the intramolecular contact H12A···O2 is also quite short at 2.38 Å (Table 1). Accordingly, the two rings face each other in a roughly 'V-shaped' form around the central SO_2 unit, with an interplanar angle of 53.45 (5)° and torsion angles C7—C6···N1—N2 = −13.10 (10) and C11—C6···N1—C5 = 21.26 (11)°. The corresponding angle N1—S1—C6 is the narrowest at S1 (the largest is, as expected, O1═S═O2). In the pyrazole ring, the bond C4—C5 is the shortest, consistent with a major contribution from the resonance form shown in the Scheme. The exocyclic C5—N3 bond is appreciably shorter than the two C—N bonds in the ring. The side-chain atom sequence C12—C13—O5—C14—C15 displays an extended conformation. See Table 2 for selected molecular dimensions.

3. Supramolecular features

Two classical hydrogen bonds (Table 1) are observed, one from each hydrogen atom of the amino group; the contact H01···O1^{i}, involving the same hydrogen atom that forms the intramolecular hydrogen bond, is however much longer than H02···O3^{ii}. The molecules are thereby connected to form layers parallel to the *bc* plane (Fig. 2).

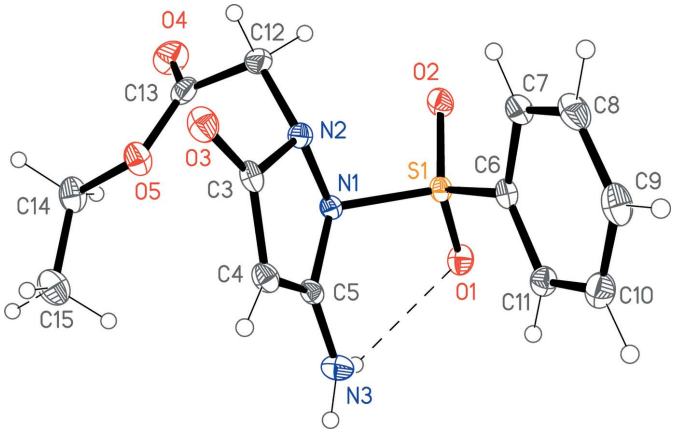


Figure 1
Structure of the title compound **4** in the crystal. Ellipsoids represent 50% probability levels. The dashed line indicates the intramolecular hydrogen bond.

Table 2
Selected geometric parameters (Å, °).

N1—C5	1.4305 (15)	N3—C5	1.3306 (16)
N1—N2	1.4313 (14)	C3—C4	1.4184 (18)
N2—C3	1.4139 (15)	C4—C5	1.3640 (17)
O2—S1—O1	120.63 (6)	N1—S1—C6	104.30 (5)
C14—O5—C13—C12	−175.81 (11)	C13—O5—C14—C15	158.95 (12)

4. Database survey

A search of the Cambridge Database (Version 5.4; Groom *et al.*, 2016) for the fragment Ar-SO_2 bonded to one nitrogen atom of an NNCCC ring (all atoms three-coordinate, any bond orders and any or no other substituents) gave only two hits, our previously reported structures NARCOY (Ar = Ph; Elgemeie *et al.*, 1998) and LERBIV (Ar = *p*-Tol; Elgemeie *et al.*, 2013). These are closely related, but the former is pseudosymmetric; for a detailed discussion, see Elgemeie *et al.* (2013). Both bear the same oxo and amino substituents as in the current structure; the latter is, however, substituted at N2,

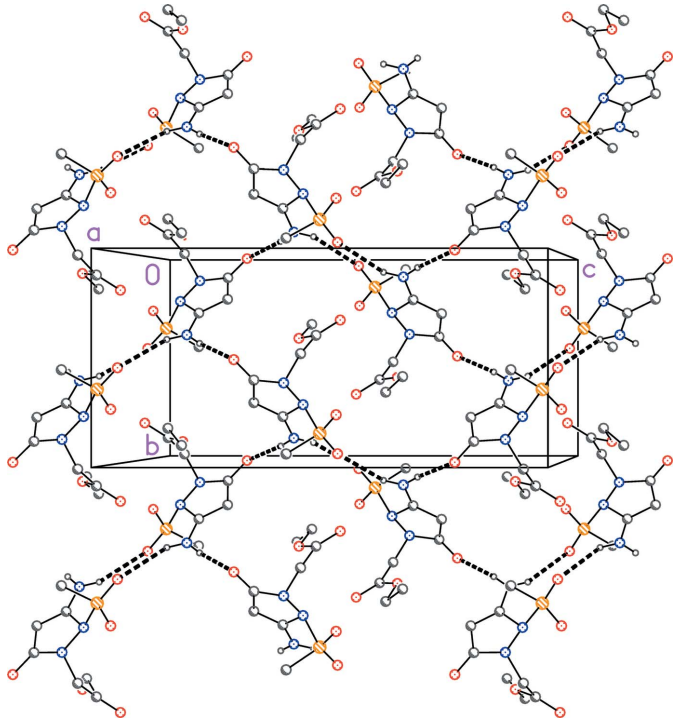


Figure 2
Packing diagram of **4** projected parallel to the *bc* plane. Dashed lines indicate intermolecular hydrogen bonds (intramolecular H bonds are omitted). Hydrogen atoms not involved in this hydrogen bonding system are omitted.

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{15}N_3O_5S$
M_r	325.34
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	9.2139 (4), 8.8122 (4), 18.3486 (7)
β (°)	104.521 (4)
V (Å^3)	1442.22 (11)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.25
Crystal size (mm)	0.35 × 0.30 × 0.15
Data collection	
Diffractometer	Oxford Diffraction Xcalibur Eos
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.964, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	74051, 4193, 3708
R_{int}	0.044
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.704
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.035, 0.085, 1.11
No. of reflections	4193
No. of parameters	208
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.47, −0.31

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXS97* (Sheldrick, 2008), *SHELXL2017* (Sheldrick, 2015) and *XP* (Siemens, 1994).

so that one fewer hydrogen-bond donor is available and the packing is different from those of the previous structures.

5. Synthesis and crystallization

A mixture of compound **1** (0.01 mol), ethyl bromoacetate **2** (0.01 mol) and anhydrous potassium carbonate (0.01 mol) in *N*,*N*-dimethylformamide (5 mL) was stirred at room temperature for 2 h. The mixture was poured onto ice–water; the solid thus formed was filtered off and recrystallized from ethanol to give pale yellow crystals in 60% yield, m.p. = 394 K. IR (KBr, cm^{-1}): ν 3330, 3250 (NH_2), 1730 (ester C=O), 1690 (ring C=O); ^{1}H NMR (DMSO-d_6): δ = 1.17 (*t*, 3H, *J* = 7.2 Hz, CH_3), 4.07 (*q*, 2H, *J* = 7.2 Hz, CH_2), 4.34 (*s*, 1H, CH), 4.43 (*s*, 2H, CH_2), 7.34 (*s*, 2H, NH_2), 7.63–7.88 (*m*, 5H, Ar). Analysis calculated $C_{13}H_{15}N_3O_5S$ (325.34); C, 47.99; H, 4.65; N, 12.92; S, 9.85. Found: C, 48.17; H, 4.84; N, 13.15; S, 9.67%.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The NH hydrogen atoms were refined freely. The methyl group was refined as an idealized rigid group allowed to rotate but not tip ('AFIX 137'; C—H 0.98 Å, H—C—H 109.5°). Other hydrogen atoms were included using a riding model starting from calculated positions (C—$H_{aromatic}$ = 0.95, C—$H_{methylene}$ = 0.99 Å). The U(H) values were fixed at 1.5 (for the methyl H) or 1.2 times the equivalent U_{iso} value of the parent carbon atoms.

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Crystal structure of ethyl 2-(5-amino-1-benzenesulfonyl-3-oxo-2,3-dihydro-1*H*-pyrazol-2-yl)acetate

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Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2015); cell refinement: *CrysAlis PRO* (Rigaku OD, 2015); data reduction: *CrysAlis PRO* (Rigaku OD, 2015); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2017* (Sheldrick, 2015); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL2017* (Sheldrick, 2015).

Ethyl 2-(5-amino-1-benzenesulfonyl-3-oxo-2,3-dihydro-1*H*-pyrazol-2-yl)acetate

Crystal data

$C_{13}H_{15}N_3O_5S$
$M_r = 325.34$
Monoclinic, $P2_1/c$
$a = 9.2139$ (4) Å
$b = 8.8122$ (4) Å
$c = 18.3486$ (7) Å
$\beta = 104.521$ (4)°
$V = 1442.22$ (11) Å^3
$Z = 4$
$F(000) = 680$
$D_x = 1.498$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 16307 reflections
$\theta = 2.6$–30.3°
$\mu = 0.25$ mm^{-1}
$T = 100$ K
Tablet, colourless
0.35 × 0.30 × 0.15 mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer
Radiation source: fine-focus sealed X-ray tube
Detector resolution: 16.1419 pixels mm^{-1}
ω–scan
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2015)
$T_{min} = 0.964$, $T_{max} = 1.000$
74051 measured reflections
4193 independent reflections
3708 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 30.0°$, $\theta_{min} = 2.3°$
$h = -12\rightarrow12$
$k = -12\rightarrow12$
$l = -25\rightarrow25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.085$
$S = 1.11$
4193 reflections
208 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0304P)^2 + 0.8673P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}

Special details

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

Refinement. The NH hydrogens were refined freely. The methyl was refined as an idealized rigid group allowed to rotate but not tip. Other hydrogens were included using a riding model starting from calculated positions.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.21827 (3)	0.35269 (3)	0.02872 (2)	0.01371 (8)
N1	0.33782 (11)	0.22191 (12)	0.08270 (6)	0.01304 (19)
N2	0.25215 (11)	0.11750 (12)	0.11476 (6)	0.0142 (2)
N3	0.54212 (13)	0.38935 (14)	0.13239 (7)	0.0187 (2)
H01	0.547 (2)	0.411 (2)	0.0871 (11)	0.029 (5)*
H02	0.602 (2)	0.431 (2)	0.1716 (11)	0.027 (4)*
O1	0.31374 (10)	0.45322 (11)	0.00110 (5)	0.01845 (19)
O2	0.10901 (10)	0.26285 (11)	−0.02197 (5)	0.01864 (19)
O3	0.27967 (10)	0.00832 (11)	0.23139 (5)	0.0201 (2)
O4	0.32627 (13)	−0.17922 (12)	−0.00343 (6)	0.0282 (2)
O5	0.46016 (11)	−0.09414 (11)	0.10922 (5)	0.0206 (2)
C3	0.32177 (13)	0.10511 (15)	0.19250 (7)	0.0151 (2)
C4	0.43781 (13)	0.21500 (15)	0.20999 (7)	0.0159 (2)
H4	0.497420	0.238013	0.258957	0.019*
C5	0.44943 (13)	0.28208 (14)	0.14470 (7)	0.0139 (2)
C6	0.13579 (13)	0.44475 (14)	0.09267 (7)	0.0148 (2)
C7	0.00733 (14)	0.38207 (15)	0.10759 (7)	0.0187 (2)
H7	−0.038852	0.294967	0.081123	0.022*
C8	−0.05126 (15)	0.45024 (17)	0.16212 (8)	0.0219 (3)
H8	−0.137945	0.408731	0.173735	0.026*
C9	0.01580 (15)	0.57863 (17)	0.19982 (8)	0.0223 (3)
H9	−0.025685	0.624603	0.236866	0.027*
C10	0.14298 (15)	0.64049 (16)	0.18388 (7)	0.0206 (3)
H10	0.187588	0.728962	0.209625	0.025*
C11	0.20494 (14)	0.57298 (15)	0.13031 (7)	0.0173 (2)
H11	0.292829	0.613492	0.119539	0.021*
C12	0.20730 (14)	−0.01978 (15)	0.07046 (7)	0.0189 (2)
H12A	0.136124	0.008368	0.022367	0.023*
H12B	0.153689	−0.087254	0.098087	0.023*
C13	0.33651 (15)	−0.10706 (15)	0.05324 (7)	0.0185 (2)
C14	0.59637 (16)	−0.16389 (17)	0.09781 (8)	0.0225 (3)
H14A	0.602432	−0.149340	0.045151	0.027*
H14B	0.596692	−0.274125	0.108255	0.027*
C15	0.72666 (16)	−0.08824 (19)	0.15109 (8)	0.0263 (3)
H15A	0.724485	0.020857	0.140513	0.039*
H15B	0.820526	−0.131624	0.144566	0.039*
H15C	0.720047	−0.104563	0.202995	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01461 (14)	0.01428 (15)	0.01147 (13)	0.00241 (10)	0.00185 (10)	0.00165 (10)
N1	0.0123 (4)	0.0133 (5)	0.0126 (4)	0.0006 (4)	0.0014 (3)	0.0012 (4)
N2	0.0141 (5)	0.0141 (5)	0.0136 (4)	−0.0002 (4)	0.0021 (4)	0.0024 (4)
N3	0.0174 (5)	0.0223 (6)	0.0158 (5)	−0.0045 (4)	0.0029 (4)	−0.0011 (4)
O1	0.0216 (4)	0.0181 (5)	0.0170 (4)	0.0021 (4)	0.0074 (3)	0.0038 (3)
O2	0.0191 (4)	0.0197 (5)	0.0137 (4)	0.0022 (4)	−0.0021 (3)	−0.0003 (3)
O3	0.0190 (4)	0.0245 (5)	0.0177 (4)	0.0022 (4)	0.0060 (3)	0.0066 (4)
O4	0.0372 (6)	0.0253 (5)	0.0202 (5)	−0.0032 (4)	0.0037 (4)	−0.0071 (4)
O5	0.0211 (5)	0.0230 (5)	0.0167 (4)	0.0060 (4)	0.0029 (3)	−0.0030 (4)
C3	0.0134 (5)	0.0183 (6)	0.0136 (5)	0.0055 (4)	0.0035 (4)	0.0017 (4)
C4	0.0150 (5)	0.0199 (6)	0.0120 (5)	0.0025 (5)	0.0017 (4)	−0.0010 (4)
C5	0.0112 (5)	0.0154 (6)	0.0144 (5)	0.0025 (4)	0.0019 (4)	−0.0025 (4)
C6	0.0145 (5)	0.0155 (6)	0.0143 (5)	0.0042 (4)	0.0033 (4)	0.0020 (4)
C7	0.0147 (5)	0.0191 (6)	0.0210 (6)	0.0019 (5)	0.0023 (5)	0.0017 (5)
C8	0.0157 (6)	0.0273 (7)	0.0243 (6)	0.0040 (5)	0.0078 (5)	0.0047 (5)
C9	0.0226 (6)	0.0265 (7)	0.0187 (6)	0.0086 (5)	0.0070 (5)	0.0018 (5)
C10	0.0238 (6)	0.0186 (6)	0.0186 (6)	0.0035 (5)	0.0039 (5)	−0.0007 (5)
C11	0.0177 (6)	0.0155 (6)	0.0184 (6)	0.0016 (5)	0.0039 (4)	0.0020 (5)
C12	0.0183 (6)	0.0160 (6)	0.0194 (6)	−0.0026 (5)	−0.0010 (5)	0.0006 (5)
C13	0.0250 (6)	0.0130 (6)	0.0163 (6)	−0.0026 (5)	0.0029 (5)	0.0018 (4)
C14	0.0249 (7)	0.0235 (7)	0.0206 (6)	0.0084 (5)	0.0086 (5)	−0.0015 (5)
C15	0.0221 (6)	0.0333 (8)	0.0242 (7)	0.0047 (6)	0.0074 (5)	0.0012 (6)

Geometric parameters (Å, º)

S1—O2	1.4268 (9)	C6—C7	1.3941 (17)
S1—O1	1.4280 (10)	C7—C8	1.3871 (19)
S1—N1	1.7249 (10)	C7—H7	0.9500
S1—C6	1.7491 (12)	C8—C9	1.388 (2)
N1—C5	1.4305 (15)	C8—H8	0.9500
N1—N2	1.4313 (14)	C9—C10	1.388 (2)
N2—C3	1.4139 (15)	C9—H9	0.9500
N2—C12	1.4583 (16)	C10—C11	1.3883 (18)
N3—C5	1.3306 (16)	C10—H10	0.9500
N3—H01	0.866 (19)	C11—H11	0.9500
N3—H02	0.871 (19)	C12—C13	1.5156 (19)
O3—C3	1.2353 (16)	C12—H12A	0.9900
O4—C13	1.2023 (16)	C12—H12B	0.9900
O5—C13	1.3338 (16)	C14—C15	1.501 (2)
O5—C14	1.4589 (16)	C14—H14A	0.9900
C3—C4	1.4184 (18)	C14—H14B	0.9900
C4—C5	1.3640 (17)	C15—H15A	0.9800
C4—H4	0.9500	C15—H15B	0.9800
C6—C11	1.3926 (18)	C15—H15C	0.9800

O2—S1—O1	120.63 (6)	C7—C8—H8	119.8
O2—S1—N1	104.37 (5)	C9—C8—H8	119.8
O1—S1—N1	104.88 (5)	C8—C9—C10	120.59 (12)
O2—S1—C6	109.90 (6)	C8—C9—H9	119.7
O1—S1—C6	111.12 (6)	C10—C9—H9	119.7
N1—S1—C6	104.30 (5)	C11—C10—C9	119.97 (13)
C5—N1—N2	105.78 (9)	C11—C10—H10	120.0
C5—N1—S1	115.93 (8)	C9—C10—H10	120.0
N2—N1—S1	109.08 (7)	C10—C11—C6	118.73 (12)
C3—N2—N1	107.87 (9)	C10—C11—H11	120.6
C3—N2—C12	119.46 (10)	C6—C11—H11	120.6
N1—N2—C12	114.36 (10)	N2—C12—C13	114.18 (10)
C5—N3—H01	120.9 (12)	N2—C12—H12A	108.7
C5—N3—H02	117.4 (12)	C13—C12—H12A	108.7
H01—N3—H02	121.5 (17)	N2—C12—H12B	108.7
C13—O5—C14	116.94 (10)	C13—C12—H12B	108.7
O3—C3—N2	120.48 (12)	H12A—C12—H12B	107.6
O3—C3—C4	131.97 (12)	O4—C13—O5	125.29 (13)
N2—C3—C4	107.53 (10)	O4—C13—C12	123.59 (12)
C5—C4—C3	108.53 (11)	O5—C13—C12	111.11 (11)
C5—C4—H4	125.7	O5—C14—C15	107.20 (11)
C3—C4—H4	125.7	O5—C14—H14A	110.3
N3—C5—C4	130.69 (12)	C15—C14—H14A	110.3
N3—C5—N1	119.50 (11)	O5—C14—H14B	110.3
C4—C5—N1	109.80 (11)	C15—C14—H14B	110.3
C11—C6—C7	121.97 (12)	H14A—C14—H14B	108.5
C11—C6—S1	119.17 (9)	C14—C15—H15A	109.5
C7—C6—S1	118.76 (10)	C14—C15—H15B	109.5
C8—C7—C6	118.25 (12)	H15A—C15—H15B	109.5
C8—C7—H7	120.9	C14—C15—H15C	109.5
C6—C7—H7	120.9	H15A—C15—H15C	109.5
C7—C8—C9	120.49 (12)	H15B—C15—H15C	109.5

O2—S1—N1—C5	−172.95 (9)	O2—S1—C6—C11	−159.28 (10)
O1—S1—N1—C5	59.29 (9)	O1—S1—C6—C11	−23.16 (12)
C6—S1—N1—C5	−57.61 (10)	N1—S1—C6—C11	89.33 (10)
O2—S1—N1—N2	−53.74 (9)	O2—S1—C6—C7	24.42 (12)
O1—S1—N1—N2	178.50 (8)	O1—S1—C6—C7	160.54 (10)
C6—S1—N1—N2	61.60 (9)	N1—S1—C6—C7	−86.97 (10)
C5—N1—N2—C3	−5.93 (12)	C11—C6—C7—C8	−0.47 (19)
S1—N1—N2—C3	−131.28 (8)	S1—C6—C7—C8	175.72 (10)
C5—N1—N2—C12	−141.43 (10)	C6—C7—C8—C9	0.85 (19)
S1—N1—N2—C12	93.23 (10)	C7—C8—C9—C10	−0.3 (2)
N1—N2—C3—O3	−171.44 (11)	C8—C9—C10—C11	−0.6 (2)
C12—N2—C3—O3	−38.61 (17)	C9—C10—C11—C6	0.97 (19)
N1—N2—C3—C4	7.23 (13)	C7—C6—C11—C10	−0.44 (19)
C12—N2—C3—C4	140.06 (11)	S1—C6—C11—C10	−176.61 (10)
O3—C3—C4—C5	172.70 (13)	C3—N2—C12—C13	−74.54 (14)

N2—C3—C4—C5	−5.75 (14)	N1—N2—C12—C13	55.45 (14)
C3—C4—C5—N3	−179.13 (13)	C14—O5—C13—O4	5.0 (2)
C3—C4—C5—N1	2.02 (14)	C14—O5—C13—C12	−175.81 (11)
N2—N1—C5—N3	−176.57 (11)	N2—C12—C13—O4	−147.96 (13)
S1—N1—C5—N3	−55.56 (13)	N2—C12—C13—O5	32.79 (15)
N2—N1—C5—C4	2.43 (13)	C13—O5—C14—C15	158.95 (12)
S1—N1—C5—C4	123.44 (10)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H01···O1	0.866 (19)	2.355 (19)	2.8296 (15)	114.8 (15)
N3—H01···O1^{i}	0.866 (19)	2.593 (19)	3.3644 (15)	148.8 (16)
N3—H02···O3^{ii}	0.871 (19)	1.961 (19)	2.8257 (15)	171.5 (17)
C12—H12*A*···O2	0.99	2.38	3.0214 (16)	122

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