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Sulfamerazine tetrahydrofuran monosolvate

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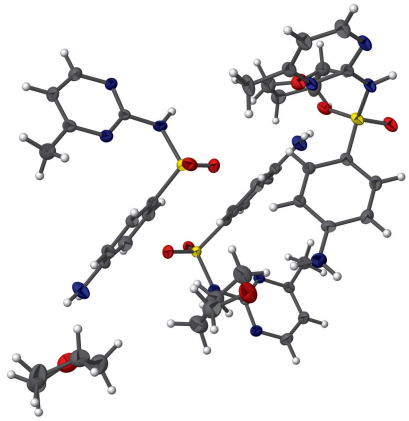
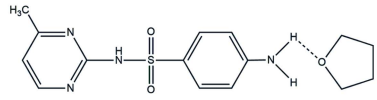
The title solvate, $C_{11}H_{12}N_4O_2S\cdot C_4H_8O$, crystallizes with three molecules of sulfamerazine [4-amino-*N*-(4-methylpyrimidin-2-yl)benzenesulfonamide] and three molecules of tetrahydrofuran solvent in the asymmetric unit. The dihedral angles between the aromatic rings in the sulfamerazine molecules are 83.40 (12), 87.40 (12) and 89.54 (12)°. In the crystal, molecules are linked by N—H···O and N—H···N hydrogen bonds, generating $(10\bar{6})$ sheets.

3D view



Chemical scheme



Structure description

This is the first crystal structure of solvated sulfamerazine. The structure of three polymorphs of sulfamerazine have been published in space groups *Pbca* (Acharya *et al.*, 1982), $Pna2_1$ (Caria & Mohamed, 1992) and $P2_1/c$ (Hossain, 2006).

The endocyclic N—C—N angles (pyrimidine ring) of 127.0 (2)–127.5 (2)° in the title compound (Fig. 1) are comparable with the corresponding values in the polymorphs of sulfamerazine; these angles are considerably larger than the value usually observed for a pyrimidine ring.

The planes of the benzene and pyrimidine rings are inclined to each other by 83.40 (12)–89.54 (12)°; these dihedral angles are larger than the corresponding values of 71 (1)° (Acharya *et al.*, 1982), 61.5 (5) and 58.5 (5)° (Caria & Mohamed, 1992) and 64.39 (2)° (Hossain, 2006) in the polymorphs of sulfamerazine. This may be due to the solvation of the tetrahydrofuran molecules hydrogen bonded to the sulfamerazine molecules. The torsion angles of the sulfamerazine molecule in this solvate indicate that the sulfamerazine molecules adopt a *gauche* conformation when viewed along the S—N vector.

In the crystal, molecules are linked by N—H···O and N—H···N hydrogen bonds (Table 1), generating $(10\bar{6})$ sheets.

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

D—H···A	D—H	H···A	D···A	D—H···A
N14—H14B···O32	0.88	2.11	2.968 (3)	166
N14—H14A···O41	0.88	2.40	3.051 (3)	131
N24—H24A···O42	0.88	2.29	2.998 (3)	137
N34—H34B···O43	0.88	2.30	2.983 (3)	134
N11—H11···N23^{i}	0.88	2.04	2.913 (3)	169
N21—H21···N13^{ii}	0.88	2.00	2.873 (3)	172
N24—H24B···O21^{iii}	0.88	2.17	2.999 (3)	158
N31—H31···N33^{iv}	0.88	2.02	2.893 (3)	174
N34—H34A···O11^{v}	0.88	2.15	3.009 (3)	167

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

Synthesis and crystallization

Solid sulfamerazine (0.5446 g, 2 mmol) was dissolved in 50 ml of hot methanol and a methanolic solution of $CuCl_2{\cdot}2H_2O$ (0.1705 g, 1 mmol) was added slowly with constant stirring on a hot plate. A red precipitate was formed and stirring was continued six hours. The precipitate was filtered and dried over silica gel. The red precipitate was dissolved in THF solution, filtered and left for crystallization at 273 K. Two weeks later, colorless blocks were filtered and dried over silica gel.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The mosaicity of the diffraction pattern was high and it did not diffract well so the R_{int} value is high.

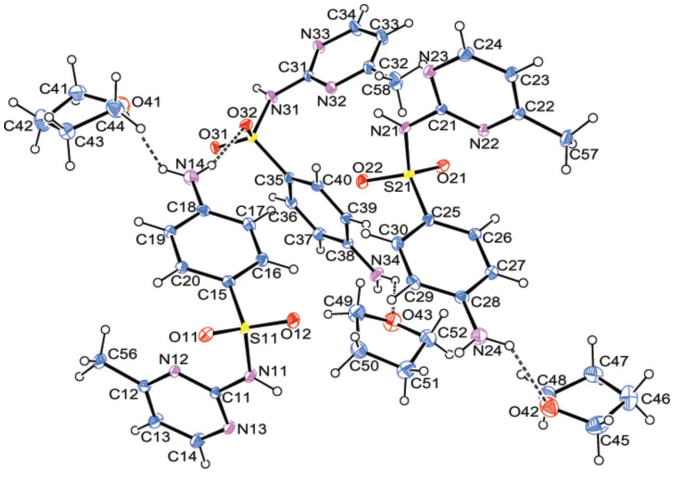


Figure 1
The molecular structure with 30% probability displacement ellipsoids for non-H atoms.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{12}N_4O_2S{\cdot}C_4H_8O$
M_r	336.41
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, b, c (Å)	10.5765 (2), 11.8088 (2), 39.1512 (7)
β (°)	92.618 (1)
V (Å^3)	4884.71 (15)
Z	12
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.22
Crystal size (mm)	0.25 × 0.22 × 0.20
Data collection	
Diffractometer	Nonius KappaCCD
Absorption correction	Multi-scan (*SORTAV*; Blessing, 1995)
T_{min}, T_{max}	0.947, 0.957
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	26393, 10636, 6070
R_{int}	0.119
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.063, 0.169, 1.01
No. of reflections	10636
No. of parameters	625
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.41, −0.58

Computer programs: *COLLECT* (Hooft, 1998; Nonius, 2004), (Otwinowski & Minor, 1997), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *ORTEP-3 for Windows* (Farrugia, 2012).

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full crystallographic data

IUCrData (2016). **1**, x160596 [doi:10.1107/S2414314616005964]

Sulfamerazine tetrahydrofuran monosolvate

G. M. Golzar Hossain

4-Amino-*N*-(4-methylpyrimidin-2-yl)benzenesulfonamide tetrahydrofuran monosolvate

Crystal data

$C_{11}H_{12}N_4O_2S·C_4H_8O$
M_r = 336.41
Monoclinic, $P2_1/c$
a = 10.5765 (2) Å
b = 11.8088 (2) Å
c = 39.1512 (7) Å
β = 92.618 (1)°
V = 4884.71 (15) Å^3
Z = 12
$F(000)$ = 2136
D_x = 1.372 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 10636 reflections
θ = 2.9–27.5°
μ = 0.22 mm^{-1}
T = 150 K
Block, colorless
0.25 × 0.22 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.947, T_{max} = 0.957
26393 measured reflections
10636 independent reflections
6070 reflections with $I > 2\sigma(I)$
R_{int} = 0.119
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −13→13
k = −15→12
l = −50→50

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.169
S = 1.01
10636 reflections
625 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0684P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S11	0.25291 (6)	0.32196 (6)	0.86011 (2)	0.02129 (18)
S21	0.73980 (6)	0.65993 (6)	0.80975 (2)	0.02120 (18)
S31	0.74417 (6)	0.83415 (6)	0.97168 (2)	0.02228 (18)
O11	0.22462 (17)	0.20954 (15)	0.87149 (4)	0.0262 (4)
O12	0.30284 (17)	0.33533 (16)	0.82686 (4)	0.0284 (5)
O21	0.78019 (16)	0.76950 (15)	0.79868 (4)	0.0268 (5)
O22	0.69177 (17)	0.64950 (16)	0.84348 (4)	0.0287 (5)
O31	0.68851 (17)	0.85232 (16)	1.00396 (4)	0.0292 (5)
O32	0.77429 (16)	0.72023 (15)	0.96177 (4)	0.0267 (5)
N11	0.12207 (19)	0.39811 (19)	0.85734 (5)	0.0247 (5)
H11	0.0974	0.4239	0.8370	0.030*
N12	0.08098 (19)	0.37871 (19)	0.91486 (5)	0.0218 (5)
N13	−0.0476 (2)	0.49571 (19)	0.87746 (5)	0.0241 (5)
N14	0.5984 (2)	0.5253 (2)	0.96566 (6)	0.0341 (6)
H14A	0.6044	0.4951	0.9862	0.041*
H14B	0.6432	0.5856	0.9610	0.041*
N21	0.86047 (19)	0.5717 (2)	0.81117 (5)	0.0257 (6)
H21	0.8837	0.5429	0.8312	0.031*
N22	0.89790 (19)	0.5881 (2)	0.75340 (5)	0.0237 (5)
N23	1.0164 (2)	0.46017 (19)	0.78991 (5)	0.0249 (5)
N24	0.3689 (2)	0.4854 (2)	0.70574 (6)	0.0393 (7)
H24A	0.3619	0.5175	0.6855	0.047*
H24B	0.3215	0.4267	0.7104	0.047*
N31	0.87487 (19)	0.90998 (19)	0.97536 (5)	0.0259 (6)
H31	0.8924	0.9426	0.9952	0.031*
N32	0.9370 (2)	0.87305 (19)	0.92019 (5)	0.0245 (5)
N33	1.0552 (2)	0.9978 (2)	0.95796 (5)	0.0272 (6)
N34	0.4198 (2)	1.0297 (2)	0.86005 (6)	0.0326 (6)
H34A	0.3732	1.0900	0.8637	0.039*
H34B	0.4186	0.9985	0.8396	0.039*
C11	0.0479 (2)	0.4246 (2)	0.88462 (6)	0.0200 (6)
C12	0.0132 (2)	0.4121 (2)	0.94139 (7)	0.0254 (7)
C13	−0.0859 (3)	0.4873 (3)	0.93699 (7)	0.0330 (8)
H13	−0.1335	0.5111	0.9557	0.040*
C14	−0.1132 (3)	0.5264 (3)	0.90433 (7)	0.0336 (8)
H14	−0.1818	0.5776	0.9007	0.040*
C15	0.3542 (2)	0.3840 (2)	0.89128 (6)	0.0205 (6)
C16	0.4259 (2)	0.4786 (2)	0.88334 (7)	0.0240 (6)
H16	0.4186	0.5109	0.8611	0.029*
C17	0.5076 (2)	0.5252 (2)	0.90798 (7)	0.0264 (7)
H17	0.5564	0.5898	0.9026	0.032*
C18	0.5193 (2)	0.4785 (2)	0.94098 (7)	0.0237 (6)
C19	0.4475 (2)	0.3824 (2)	0.94814 (7)	0.0230 (6)
H19	0.4558	0.3487	0.9702	0.028*
C20	0.3653 (2)	0.3361 (2)	0.92385 (6)	0.0212 (6)

H20	0.3162	0.2717	0.9292	0.025*
C21	0.9280 (2)	0.5395 (2)	0.78351 (6)	0.0200 (6)
C22	0.9583 (2)	0.5492 (2)	0.72655 (7)	0.0258 (7)
C23	1.0500 (3)	0.4662 (3)	0.73016 (7)	0.0320 (7)
H23	1.0931	0.4390	0.7110	0.038*
C24	1.0766 (3)	0.4242 (3)	0.76258 (7)	0.0315 (7)
H24	1.1400	0.3677	0.7656	0.038*
C25	0.6292 (2)	0.6087 (2)	0.77938 (6)	0.0203 (6)
C26	0.6176 (2)	0.6598 (2)	0.74717 (6)	0.0220 (6)
H26	0.6705	0.7219	0.7420	0.026*
C27	0.5298 (2)	0.6203 (2)	0.72295 (7)	0.0253 (6)
H27	0.5209	0.6567	0.7013	0.030*
C28	0.4536 (2)	0.5268 (2)	0.73002 (7)	0.0249 (6)
C29	0.4649 (2)	0.4768 (2)	0.76266 (7)	0.0279 (7)
H29	0.4119	0.4150	0.7680	0.034*
C30	0.5528 (2)	0.5174 (2)	0.78696 (7)	0.0252 (6)
H30	0.5608	0.4827	0.8088	0.030*
C31	0.9596 (2)	0.9269 (2)	0.94987 (6)	0.0223 (6)
C32	1.0181 (3)	0.8950 (2)	0.89552 (7)	0.0278 (7)
C33	1.1182 (3)	0.9691 (3)	0.90116 (7)	0.0339 (7)
H33	1.1749	0.9852	0.8837	0.041*
C34	1.1330 (3)	1.0185 (3)	0.93276 (7)	0.0349 (8)
H34	1.2015	1.0695	0.9369	0.042*
C35	0.6488 (2)	0.8931 (2)	0.93866 (6)	0.0209 (6)
C36	0.5742 (2)	0.9872 (2)	0.94514 (7)	0.0263 (7)
H36	0.5759	1.0200	0.9673	0.032*
C37	0.4979 (2)	1.0324 (2)	0.91911 (7)	0.0271 (7)
H37	0.4472	1.0968	0.9234	0.032*
C38	0.4941 (2)	0.9845 (2)	0.88630 (7)	0.0233 (6)
C39	0.5698 (2)	0.8894 (2)	0.88041 (7)	0.0225 (6)
H39	0.5679	0.8558	0.8583	0.027*
C40	0.6465 (2)	0.8445 (2)	0.90619 (7)	0.0223 (6)
H40	0.6978	0.7805	0.9019	0.027*
C56	0.0545 (3)	0.3624 (3)	0.97532 (6)	0.0351 (8)
H56A	0.1384	0.3917	0.9823	0.053*
H56B	0.0583	0.2797	0.9734	0.053*
H56C	−0.0062	0.3832	0.9924	0.053*
C57	0.9215 (3)	0.6026 (3)	0.69277 (7)	0.0390 (8)
H57A	0.9127	0.6846	0.6957	0.059*
H57B	0.8407	0.5709	0.6841	0.059*
H57C	0.9870	0.5872	0.6765	0.059*
C58	0.9911 (3)	0.8349 (3)	0.86235 (7)	0.0419 (9)
H58A	0.9073	0.8571	0.8529	0.063*
H58B	0.9931	0.7529	0.8662	0.063*
H58C	1.0554	0.8555	0.8462	0.063*
O41	0.75615 (19)	0.36623 (19)	1.01187 (5)	0.0414 (6)
O42	0.2191 (2)	0.6315 (2)	0.65606 (6)	0.0490 (6)
O43	0.3115 (2)	0.84734 (18)	0.81484 (6)	0.0484 (6)

C41	0.7635 (3)	0.3445 (3)	1.04792 (8)	0.0408 (8)
H41A	0.7407	0.4131	1.0608	0.049*
H41B	0.8501	0.3209	1.0555	0.049*
C42	0.6696 (3)	0.2502 (3)	1.05346 (8)	0.0472 (9)
H42A	0.5870	0.2811	1.0598	0.057*
H42B	0.7016	0.1981	1.0717	0.057*
C43	0.6576 (3)	0.1892 (3)	1.01893 (8)	0.0383 (8)
H43A	0.6858	0.1095	1.0211	0.046*
H43B	0.5692	0.1908	1.0095	0.046*
C44	0.7442 (3)	0.2564 (3)	0.99650 (8)	0.0444 (9)
H44A	0.8279	0.2193	0.9957	0.053*
H44B	0.7069	0.2623	0.9729	0.053*
C45	0.2034 (3)	0.6474 (3)	0.61990 (9)	0.0538 (10)
H45A	0.1132	0.6609	0.6133	0.065*
H45B	0.2324	0.5795	0.6077	0.065*
C46	0.2823 (4)	0.7491 (3)	0.61114 (9)	0.0549 (10)
H46A	0.2335	0.8017	0.5959	0.066*
H46B	0.3599	0.7257	0.5998	0.066*
C47	0.3146 (3)	0.8040 (3)	0.64550 (8)	0.0460 (9)
H47A	0.4052	0.7935	0.6522	0.055*
H47B	0.2952	0.8860	0.6449	0.055*
C48	0.2318 (3)	0.7430 (3)	0.66945 (9)	0.0500 (9)
H48A	0.2715	0.7415	0.6928	0.060*
H48B	0.1482	0.7803	0.6702	0.060*
C49	0.3219 (3)	0.7412 (3)	0.83223 (9)	0.0458 (9)
H49A	0.2973	0.7492	0.8562	0.055*
H49B	0.4099	0.7126	0.8322	0.055*
C50	0.2324 (3)	0.6605 (3)	0.81282 (8)	0.0390 (8)
H50A	0.1538	0.6489	0.8252	0.047*
H50B	0.2732	0.5862	0.8093	0.047*
C51	0.2048 (3)	0.7208 (3)	0.77892 (8)	0.0482 (9)
H51A	0.2204	0.6700	0.7594	0.058*
H51B	0.1159	0.7471	0.7770	0.058*
C52	0.2948 (3)	0.8194 (3)	0.77967 (8)	0.0448 (9)
H52A	0.3764	0.7979	0.7701	0.054*
H52B	0.2584	0.8841	0.7665	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S11	0.0219 (4)	0.0264 (4)	0.0156 (3)	0.0045 (3)	0.0009 (3)	−0.0011 (3)
S21	0.0205 (4)	0.0252 (4)	0.0177 (4)	0.0036 (3)	−0.0004 (3)	−0.0016 (3)
S31	0.0202 (4)	0.0277 (4)	0.0187 (4)	−0.0072 (3)	−0.0011 (3)	0.0012 (3)
O11	0.0297 (11)	0.0252 (11)	0.0233 (10)	0.0034 (9)	−0.0043 (8)	−0.0012 (8)
O12	0.0301 (11)	0.0396 (13)	0.0158 (10)	0.0056 (9)	0.0058 (8)	−0.0009 (8)
O21	0.0274 (10)	0.0243 (11)	0.0281 (11)	−0.0002 (9)	−0.0062 (8)	0.0018 (8)
O22	0.0297 (11)	0.0396 (13)	0.0170 (10)	0.0075 (9)	0.0042 (8)	−0.0020 (8)
O31	0.0264 (11)	0.0451 (13)	0.0163 (10)	−0.0120 (10)	0.0027 (8)	0.0018 (9)

O32	0.0277 (10)	0.0265 (12)	0.0254 (11)	−0.0049 (9)	−0.0058 (8)	0.0028 (8)
N11	0.0234 (12)	0.0352 (15)	0.0153 (12)	0.0082 (11)	0.0003 (9)	0.0033 (10)
N12	0.0189 (12)	0.0307 (14)	0.0158 (12)	0.0014 (10)	0.0008 (9)	0.0031 (10)
N13	0.0239 (12)	0.0310 (14)	0.0175 (12)	0.0067 (11)	0.0008 (9)	0.0038 (10)
N14	0.0338 (14)	0.0362 (16)	0.0319 (14)	−0.0113 (12)	−0.0025 (11)	−0.0015 (11)
N21	0.0227 (12)	0.0367 (15)	0.0173 (12)	0.0098 (11)	−0.0023 (9)	0.0064 (10)
N22	0.0211 (12)	0.0346 (15)	0.0156 (12)	0.0039 (11)	0.0018 (9)	0.0030 (10)
N23	0.0240 (12)	0.0329 (15)	0.0179 (12)	0.0079 (11)	0.0005 (9)	0.0002 (10)
N24	0.0384 (15)	0.0485 (18)	0.0305 (15)	−0.0172 (13)	−0.0051 (12)	−0.0003 (12)
N31	0.0220 (12)	0.0370 (15)	0.0187 (12)	−0.0103 (11)	0.0010 (9)	−0.0049 (10)
N32	0.0222 (12)	0.0344 (15)	0.0168 (12)	−0.0044 (11)	−0.0003 (9)	−0.0016 (10)
N33	0.0246 (12)	0.0380 (15)	0.0191 (13)	−0.0137 (11)	0.0013 (10)	−0.0030 (10)
N34	0.0346 (14)	0.0367 (15)	0.0263 (14)	0.0110 (12)	−0.0017 (11)	0.0044 (11)
C11	0.0186 (13)	0.0267 (16)	0.0146 (14)	−0.0012 (12)	−0.0007 (10)	−0.0015 (11)
C12	0.0207 (14)	0.0381 (18)	0.0173 (14)	−0.0009 (13)	0.0012 (11)	−0.0010 (12)
C13	0.0306 (16)	0.050 (2)	0.0184 (15)	0.0117 (15)	0.0030 (12)	−0.0041 (13)
C14	0.0295 (16)	0.045 (2)	0.0266 (17)	0.0152 (15)	0.0021 (13)	0.0008 (14)
C15	0.0187 (14)	0.0221 (15)	0.0209 (14)	0.0045 (12)	0.0041 (11)	−0.0029 (11)
C16	0.0246 (15)	0.0255 (16)	0.0223 (15)	0.0047 (13)	0.0048 (12)	0.0026 (12)
C17	0.0264 (15)	0.0237 (16)	0.0296 (16)	−0.0037 (13)	0.0070 (12)	0.0020 (12)
C18	0.0181 (14)	0.0316 (17)	0.0215 (15)	0.0006 (13)	0.0028 (11)	−0.0045 (12)
C19	0.0217 (14)	0.0275 (16)	0.0197 (14)	0.0000 (12)	0.0003 (11)	0.0022 (12)
C20	0.0197 (14)	0.0224 (15)	0.0216 (15)	0.0020 (12)	0.0019 (11)	0.0006 (11)
C21	0.0165 (13)	0.0280 (16)	0.0155 (14)	0.0016 (12)	0.0002 (10)	0.0011 (11)
C22	0.0208 (14)	0.0369 (18)	0.0196 (15)	0.0012 (13)	0.0003 (11)	0.0008 (13)
C23	0.0311 (16)	0.048 (2)	0.0168 (15)	0.0102 (15)	0.0049 (12)	−0.0028 (13)
C24	0.0292 (16)	0.0389 (19)	0.0264 (17)	0.0117 (14)	0.0009 (13)	−0.0022 (14)
C25	0.0193 (14)	0.0220 (15)	0.0198 (14)	0.0029 (12)	0.0041 (11)	0.0004 (11)
C26	0.0223 (14)	0.0212 (15)	0.0226 (15)	0.0011 (12)	0.0005 (11)	0.0000 (11)
C27	0.0256 (15)	0.0283 (17)	0.0218 (15)	−0.0004 (13)	−0.0004 (12)	0.0011 (12)
C28	0.0210 (14)	0.0303 (17)	0.0231 (15)	−0.0015 (13)	0.0001 (11)	−0.0064 (12)
C29	0.0264 (15)	0.0262 (17)	0.0317 (17)	−0.0064 (13)	0.0072 (12)	0.0009 (13)
C30	0.0267 (15)	0.0279 (17)	0.0212 (15)	0.0032 (13)	0.0040 (12)	0.0036 (12)
C31	0.0211 (14)	0.0300 (17)	0.0159 (14)	−0.0023 (12)	0.0002 (11)	0.0024 (12)
C32	0.0289 (16)	0.0345 (18)	0.0202 (15)	−0.0019 (14)	0.0016 (12)	0.0014 (12)
C33	0.0320 (17)	0.048 (2)	0.0223 (16)	−0.0113 (15)	0.0063 (12)	−0.0003 (14)
C34	0.0311 (17)	0.048 (2)	0.0260 (17)	−0.0194 (15)	0.0070 (13)	−0.0046 (14)
C35	0.0200 (14)	0.0246 (16)	0.0183 (14)	−0.0065 (12)	0.0012 (11)	0.0007 (11)
C36	0.0267 (15)	0.0276 (17)	0.0250 (16)	−0.0047 (13)	0.0058 (12)	−0.0038 (12)
C37	0.0268 (15)	0.0249 (16)	0.0299 (17)	0.0029 (13)	0.0054 (12)	−0.0026 (12)
C38	0.0191 (14)	0.0269 (16)	0.0238 (15)	−0.0006 (12)	0.0000 (11)	0.0058 (12)
C39	0.0201 (14)	0.0267 (16)	0.0207 (14)	−0.0006 (12)	0.0004 (11)	−0.0017 (12)
C40	0.0194 (14)	0.0238 (16)	0.0236 (15)	−0.0007 (12)	0.0010 (11)	−0.0003 (12)
C56	0.0315 (17)	0.055 (2)	0.0188 (16)	0.0075 (16)	0.0040 (12)	0.0061 (14)
C57	0.0411 (19)	0.058 (2)	0.0182 (16)	0.0153 (17)	0.0023 (13)	0.0070 (14)
C58	0.051 (2)	0.055 (2)	0.0209 (17)	−0.0154 (18)	0.0092 (14)	−0.0115 (15)
O41	0.0397 (13)	0.0418 (14)	0.0435 (14)	−0.0011 (11)	0.0088 (10)	0.0051 (11)
O42	0.0471 (14)	0.0436 (15)	0.0571 (16)	0.0022 (12)	0.0099 (11)	0.0111 (12)

O43	0.0591 (16)	0.0386 (14)	0.0471 (15)	−0.0102 (12)	−0.0034 (12)	−0.0045 (11)
C41	0.0356 (19)	0.047 (2)	0.039 (2)	0.0022 (16)	−0.0001 (15)	−0.0040 (16)
C42	0.064 (2)	0.037 (2)	0.041 (2)	−0.0001 (18)	0.0180 (17)	−0.0022 (15)
C43	0.0362 (18)	0.041 (2)	0.0378 (19)	−0.0004 (16)	0.0049 (14)	0.0029 (15)
C44	0.0406 (19)	0.053 (2)	0.040 (2)	−0.0023 (17)	0.0086 (15)	−0.0059 (17)
C45	0.049 (2)	0.053 (3)	0.058 (3)	0.012 (2)	−0.0105 (18)	−0.0118 (19)
C46	0.080 (3)	0.040 (2)	0.045 (2)	0.009 (2)	0.0156 (19)	0.0022 (17)
C47	0.0361 (19)	0.051 (2)	0.051 (2)	0.0009 (17)	0.0007 (16)	−0.0005 (17)
C48	0.053 (2)	0.058 (3)	0.040 (2)	0.0099 (19)	0.0065 (17)	0.0012 (18)
C49	0.052 (2)	0.039 (2)	0.046 (2)	−0.0055 (18)	−0.0035 (16)	0.0034 (17)
C50	0.0393 (19)	0.036 (2)	0.042 (2)	−0.0022 (15)	0.0063 (15)	−0.0017 (15)
C51	0.049 (2)	0.048 (2)	0.046 (2)	−0.0012 (18)	−0.0072 (17)	−0.0008 (17)
C52	0.044 (2)	0.044 (2)	0.047 (2)	0.0001 (17)	0.0059 (16)	0.0026 (17)

Geometric parameters (Å, º)

S11—O11	1.4361 (19)	C29—C30	1.386 (3)
S11—O12	1.4359 (19)	C29—H29	0.9500
S11—N11	1.650 (2)	C30—H30	0.9500
S11—C15	1.747 (3)	C32—C33	1.384 (4)
S21—O21	1.4358 (19)	C32—C58	1.495 (4)
S21—O22	1.4417 (19)	C33—C34	1.370 (4)
S21—N21	1.646 (2)	C33—H33	0.9500
S21—C25	1.738 (3)	C34—H34	0.9500
S31—O31	1.4346 (19)	C35—C36	1.393 (4)
S31—O32	1.440 (2)	C35—C40	1.394 (3)
S31—N31	1.648 (2)	C36—C37	1.378 (3)
S31—C35	1.748 (2)	C36—H36	0.9500
N11—C11	1.390 (3)	C37—C38	1.403 (3)
N11—H11	0.8800	C37—H37	0.9500
N12—C11	1.334 (3)	C38—C39	1.404 (4)
N12—C12	1.348 (3)	C39—C40	1.372 (3)
N13—C11	1.333 (3)	C39—H39	0.9500
N13—C14	1.337 (3)	C40—H40	0.9500
N14—C18	1.365 (3)	C56—H56A	0.9800
N14—H14A	0.8800	C56—H56B	0.9800
N14—H14B	0.8800	C56—H56C	0.9800
N21—C21	1.378 (3)	C57—H57A	0.9800
N21—H21	0.8800	C57—H57B	0.9800
N22—C21	1.336 (3)	C57—H57C	0.9800
N22—C22	1.336 (3)	C58—H58A	0.9800
N23—C24	1.339 (3)	C58—H58B	0.9800
N23—C21	1.339 (3)	C58—H58C	0.9800
N24—C28	1.366 (3)	O41—C41	1.433 (3)
N24—H24A	0.8800	O41—C44	1.433 (4)
N24—H24B	0.8800	O42—C48	1.422 (4)
N31—C31	1.386 (3)	O42—C45	1.430 (4)
N31—H31	0.8800	O43—C52	1.419 (3)

N32—C31	1.337 (3)	O43—C49	1.429 (4)
N32—C32	1.346 (3)	C41—C42	1.515 (4)
N33—C34	1.336 (3)	C41—H41A	0.9900
N33—C31	1.339 (3)	C41—H41B	0.9900
N34—C38	1.373 (3)	C42—C43	1.532 (4)
N34—H34A	0.8800	C42—H42A	0.9900
N34—H34B	0.8800	C42—H42B	0.9900
C12—C13	1.378 (4)	C43—C44	1.520 (4)
C12—C56	1.499 (3)	C43—H43A	0.9900
C13—C14	1.378 (4)	C43—H43B	0.9900
C13—H13	0.9500	C44—H44A	0.9900
C14—H14	0.9500	C44—H44B	0.9900
C15—C16	1.394 (4)	C45—C46	1.511 (5)
C15—C20	1.395 (3)	C45—H45A	0.9900
C16—C17	1.379 (3)	C45—H45B	0.9900
C16—H16	0.9500	C46—C47	1.518 (4)
C17—C18	1.405 (4)	C46—H46A	0.9900
C17—H17	0.9500	C46—H46B	0.9900
C18—C19	1.401 (4)	C47—C48	1.496 (4)
C19—C20	1.372 (3)	C47—H47A	0.9900
C19—H19	0.9500	C47—H47B	0.9900
C20—H20	0.9500	C48—H48A	0.9900
C22—C23	1.382 (4)	C48—H48B	0.9900
C22—C57	1.500 (3)	C49—C50	1.521 (4)
C23—C24	1.379 (3)	C49—H49A	0.9900
C23—H23	0.9500	C49—H49B	0.9900
C24—H24	0.9500	C50—C51	1.522 (4)
C25—C30	1.387 (4)	C50—H50A	0.9900
C25—C26	1.398 (3)	C50—H50B	0.9900
C26—C27	1.378 (3)	C51—C52	1.504 (4)
C26—H26	0.9500	C51—H51A	0.9900
C27—C28	1.403 (4)	C51—H51B	0.9900
C27—H27	0.9500	C52—H52A	0.9900
C28—C29	1.407 (4)	C52—H52B	0.9900

O11—S11—O12	118.25 (11)	C33—C34—H34	118.7
O11—S11—N11	109.77 (12)	C36—C35—C40	120.5 (2)
O12—S11—N11	102.86 (11)	C36—C35—S31	119.7 (2)
O12—S11—C15	110.37 (12)	C40—C35—S31	119.7 (2)
O11—S11—C15	107.49 (12)	C37—C36—C35	119.5 (3)
N11—S11—C15	107.65 (12)	C37—C36—H36	120.3
O21—S21—O22	118.40 (11)	C35—C36—H36	120.3
O21—S21—N21	109.83 (12)	C36—C37—C38	120.8 (3)
O22—S21—N21	102.72 (11)	C36—C37—H37	119.6
O21—S21—C25	107.90 (12)	C38—C37—H37	119.6
O22—S21—C25	110.03 (12)	N34—C38—C37	121.3 (3)
N21—S21—C25	107.43 (12)	N34—C38—C39	120.0 (2)
O31—S31—O32	118.81 (12)	C37—C38—C39	118.7 (2)

O31—S31—N31	102.59 (11)	C40—C39—C38	120.7 (2)
O32—S31—N31	109.65 (12)	C40—C39—H39	119.7
O31—S31—C35	110.23 (12)	C38—C39—H39	119.7
O32—S31—C35	107.49 (12)	C39—C40—C35	119.8 (3)
N31—S31—C35	107.56 (12)	C39—C40—H40	120.1
C11—N11—S11	125.00 (17)	C35—C40—H40	120.1
C11—N11—H11	117.5	C12—C56—H56A	109.5
S11—N11—H11	117.5	C12—C56—H56B	109.5
C11—N12—C12	116.0 (2)	H56A—C56—H56B	109.5
C11—N13—C14	114.9 (2)	C12—C56—H56C	109.5
C18—N14—H14A	120.0	H56A—C56—H56C	109.5
C18—N14—H14B	120.0	H56B—C56—H56C	109.5
H14A—N14—H14B	120.0	C22—C57—H57A	109.5
C21—N21—S21	125.26 (17)	C22—C57—H57B	109.5
C21—N21—H21	117.4	H57A—C57—H57B	109.5
S21—N21—H21	117.4	C22—C57—H57C	109.5
C21—N22—C22	116.3 (2)	H57A—C57—H57C	109.5
C24—N23—C21	115.2 (2)	H57B—C57—H57C	109.5
C28—N24—H24A	120.0	C32—C58—H58A	109.5
C28—N24—H24B	120.0	C32—C58—H58B	109.5
H24A—N24—H24B	120.0	H58A—C58—H58B	109.5
C31—N31—S31	125.66 (18)	C32—C58—H58C	109.5
C31—N31—H31	117.2	H58A—C58—H58C	109.5
S31—N31—H31	117.2	H58B—C58—H58C	109.5
C31—N32—C32	116.0 (2)	C41—O41—C44	104.6 (2)
C34—N33—C31	115.1 (2)	C48—O42—C45	104.3 (2)
C38—N34—H34A	120.0	C52—O43—C49	105.2 (2)
C38—N34—H34B	120.0	O41—C41—C42	105.4 (2)
H34A—N34—H34B	120.0	O41—C41—H41A	110.7
N12—C11—N13	127.5 (2)	C42—C41—H41A	110.7
N13—C11—N11	115.4 (2)	O41—C41—H41B	110.7
N12—C11—N11	117.1 (2)	C42—C41—H41B	110.7
N12—C12—C13	121.4 (2)	H41A—C41—H41B	108.8
N12—C12—C56	115.1 (2)	C41—C42—C43	104.4 (3)
C13—C12—C56	123.5 (3)	C41—C42—H42A	110.9
C14—C13—C12	117.2 (3)	C43—C42—H42A	110.9
C14—C13—H13	121.4	C41—C42—H42B	110.9
C12—C13—H13	121.4	C43—C42—H42B	110.9
N13—C14—C13	123.0 (3)	H42A—C42—H42B	108.9
N13—C14—H14	118.5	C44—C43—C42	103.7 (3)
C13—C14—H14	118.5	C44—C43—H43A	111.0
C16—C15—C20	120.3 (2)	C42—C43—H43A	111.0
C16—C15—S11	120.2 (2)	C44—C43—H43B	111.0
C20—C15—S11	119.5 (2)	C42—C43—H43B	111.0
C17—C16—C15	119.6 (2)	H43A—C43—H43B	109.0
C17—C16—H16	120.2	O41—C44—C43	105.9 (3)
C15—C16—H16	120.2	O41—C44—H44A	110.6
C16—C17—C18	120.8 (3)	C43—C44—H44A	110.6

C16—C17—H17	119.6	O41—C44—H44B	110.6
C18—C17—H17	119.6	C43—C44—H44B	110.6
N14—C18—C19	120.4 (2)	H44A—C44—H44B	108.7
N14—C18—C17	121.1 (3)	O42—C45—C46	106.8 (3)
C19—C18—C17	118.4 (2)	O42—C45—H45A	110.4
C20—C19—C18	121.1 (2)	C46—C45—H45A	110.4
C20—C19—H19	119.5	O42—C45—H45B	110.4
C18—C19—H19	119.5	C46—C45—H45B	110.4
C19—C20—C15	119.8 (3)	H45A—C45—H45B	108.6
C19—C20—H20	120.1	C45—C46—C47	104.0 (3)
C15—C20—H20	120.1	C45—C46—H46A	110.9
N22—C21—N23	127.0 (2)	C47—C46—H46A	110.9
N22—C21—N21	117.6 (2)	C45—C46—H46B	110.9
N23—C21—N21	115.4 (2)	C47—C46—H46B	110.9
N22—C22—C23	121.4 (2)	H46A—C46—H46B	109.0
N22—C22—C57	115.8 (2)	C48—C47—C46	103.6 (3)
C23—C22—C57	122.8 (3)	C48—C47—H47A	111.0
C24—C23—C22	117.5 (3)	C46—C47—H47A	111.0
C24—C23—H23	121.3	C48—C47—H47B	111.0
C22—C23—H23	121.3	C46—C47—H47B	111.0
N23—C24—C23	122.5 (3)	H47A—C47—H47B	109.0
N23—C24—H24	118.8	O42—C48—C47	105.2 (3)
C23—C24—H24	118.8	O42—C48—H48A	110.7
C30—C25—C26	120.0 (2)	C47—C48—H48A	110.7
C30—C25—S21	120.3 (2)	O42—C48—H48B	110.7
C26—C25—S21	119.7 (2)	C47—C48—H48B	110.7
C27—C26—C25	120.2 (3)	H48A—C48—H48B	108.8
C27—C26—H26	119.9	O43—C49—C50	106.2 (2)
C25—C26—H26	119.9	O43—C49—H49A	110.5
C26—C27—C28	120.4 (2)	C50—C49—H49A	110.5
C26—C27—H27	119.8	O43—C49—H49B	110.5
C28—C27—H27	119.8	C50—C49—H49B	110.5
N24—C28—C27	120.6 (3)	H49A—C49—H49B	108.7
N24—C28—C29	120.5 (3)	C49—C50—C51	103.5 (3)
C27—C28—C29	118.9 (2)	C49—C50—H50A	111.1
C30—C29—C28	120.3 (3)	C51—C50—H50A	111.1
C30—C29—H29	119.8	C49—C50—H50B	111.1
C28—C29—H29	119.8	C51—C50—H50B	111.1
C29—C30—C25	120.1 (2)	H50A—C50—H50B	109.0
C29—C30—H30	120.0	C52—C51—C50	104.4 (2)
C25—C30—H30	120.0	C52—C51—H51A	110.9
N32—C31—N33	127.3 (2)	C50—C51—H51A	110.9
N32—C31—N31	117.5 (2)	C52—C51—H51B	110.9
N33—C31—N31	115.2 (2)	C50—C51—H51B	110.9
N32—C32—C33	121.0 (3)	H51A—C51—H51B	108.9
N32—C32—C58	115.5 (2)	O43—C52—C51	104.5 (3)
C33—C32—C58	123.6 (3)	O43—C52—H52A	110.9
C34—C33—C32	117.9 (3)	C51—C52—H52A	110.9

C34—C33—H33	121.0	O43—C52—H52B	110.9
C32—C33—H33	121.0	C51—C52—H52B	110.9
N33—C34—C33	122.7 (3)	H52A—C52—H52B	108.9
N33—C34—H34	118.7		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N14—H14*B*···O32	0.88	2.11	2.968 (3)	166
N14—H14*A*···O41	0.88	2.40	3.051 (3)	131
N24—H24*A*···O42	0.88	2.29	2.998 (3)	137
N34—H34*B*···O43	0.88	2.30	2.983 (3)	134
N11—H11···N23^{i}	0.88	2.04	2.913 (3)	169
N21—H21···N13^{ii}	0.88	2.00	2.873 (3)	172
N24—H24*B*···O21^{iii}	0.88	2.17	2.999 (3)	158
N31—H31···N33^{iv}	0.88	2.02	2.893 (3)	174
N34—H34*A*···O11^{v}	0.88	2.15	3.009 (3)	167

Symmetry codes: (i) $x-1, y, z$; (ii) $x+1, y, z$; (iii) $-x+1, y-1/2, -z+3/2$; (iv) $-x+2, -y+2, -z+2$; (v) $x, y+1, z$.