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4-(2-Nitrobenzenesulfonamido)-pyridinium chloride hemihydrate

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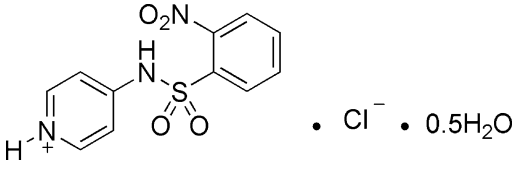
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.037; wR factor = 0.094; data-to-parameter ratio = 14.3.

In the crystal structure of the title compound, $C_{11}H_{10}N_3O_4S^+\cdot Cl^-\cdot 0.5H_2O$, there are two formula units in the asymmetric unit. A network of N—H···Cl, N—H···O and O—H···Cl hydrogen bonds helps to establish the crystal packing. The nitro group of one of the organic cations is disordered over two positions in a 0.57 (2):0.43 (2) ratio.

Related literature

For related literature, see: Yu & Li (2007).



Experimental

Crystal data

$C_{11}H_{10}N_3O_4S^+\cdot Cl^-\cdot 0.5H_2O$
M_r = 324.74
Monoclinic, $P2_1/c$
a = 8.5250 (9) Å
b = 23.686 (3) Å
c = 14.0227 (15) Å
β = 90.533 (2)°
V = 2831.4 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 294 (2) K
0.18 × 0.16 × 0.16 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.925, T_{max} = 0.933
16156 measured reflections
5788 independent reflections
4324 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.094
S = 1.04
5788 reflections
405 parameters
47 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···Cl1^{i}	0.878 (10)	2.321 (13)	3.1500 (17)	157 (2)
N3—H3A···Cl1	0.897 (10)	2.155 (11)	3.0472 (18)	173 (2)
N5—H5A···Cl2	0.897 (10)	2.253 (11)	3.1270 (17)	164 (2)
N6—H6A···O9^{i}	0.894 (10)	1.858 (13)	2.700 (2)	156 (2)
O9—H9A···Cl2^{ii}	0.86	2.39	3.1979 (18)	157
O9—H9B···Cl2	0.86	2.29	3.1332 (19)	167

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2493).

References

Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Yu, H.-J. & Li, J.-S. (2007). *Acta Cryst.* E**63**, o3399.

  doi:10.1107/S1600536807038378

supplementary materials

Acta Cryst. (2007). E**63**, o3766 [**doi:10.1107/S1600536807038378**]

4-(2-Nitrobenzenesulfonamido)pyridinium chloride hemihydrate

H.-J. Yu and J.-S. Li

Comment

As part of our ongoing studies of *N*-sulfonated derivatives of 4-aminopyridine (Yu & Li, 2007), which contain both an acid and a base centre, we had planned to obtain the neutral zwitterionic crystal structure, but failed. Thus, the crystal structure of the semihydrated HCl title salt, (I), is reported herein.

The asymmetric unit of (I) contains two cations, two anions, and a single water molecule (Fig. 1). The short C—N distances [C7—N2 = 1.385 (2), C18—N5 = 1.382 (3) Å] and planar conformations [S1/H2A/N2/C7 0.0012, S2/H5A/N5/C18 0.0039 Å] indicate that N2 and N5 are sp^2 hybridized to a large extent which can be related to tautomeric Lewis structures (Fig. 2). The benzene ring makes angles of 81.07 (12) and 74.80 (11)° with the pyridinium ring in the two asymmetric cations.

The crystal structure of (I) is stabilized by a series of intermolecular N—H···Cl, N—H···O and O—H···Cl hydrogen bonds (Table 1) with the water molecules making important contributions.

Experimental

2-Nitro-*N*-(4-pyridyl)benzenesulfonamide was obtained according to the method of Yu & Li (2007). Colourless blocks of (I) were grown by natural evaporation of an aqueous HCl solution of the amide.

Refinement

The N-bound H atoms were located in a difference map and refined with the constraint N—H = 0.90 (1) Å and their U_{iso} values were freely refined. The C-bound H atoms were positioned geometrically (C—H = 0.93 Å) and refined as riding atoms and the O-bound H atoms refined as riding in their as-found relative positions. The constraint $U_{iso}(H) = 1.2U_{eq}$(C or O) was applied. O1 and O2 are disordered over two positions and their occupancy ratio 0.57 (2):0.43 (2).

Figures

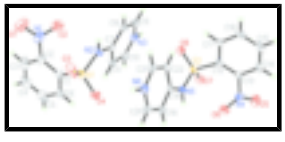


Fig. 1. A view of (I) with displacement ellipsoids drawn at the 30% probability level (arbitrary spheres for the H atoms). Only one of the two disordered components is shown. The water molecule and chloride counterions are omitted for clarity.

Fig. 2. The tautomerization in the title compound.

4-(2-Nitrobenzenesulfonamido)pyridinium chloride hemihydrate

Crystal data

$C_{11}H_{10}N_3O_4S^+\cdot Cl^-\cdot 0.5H_2O$	F_{000} = 1336
M_r = 324.74	D_x = 1.524 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5769 reflections
a = 8.5250 (9) Å	θ = 2.3–26.3º
b = 23.686 (3) Å	μ = 0.44 mm^{-1}
c = 14.0227 (15) Å	T = 294 (2) K
β = 90.533 (2)º	Block, colourless
V = 2831.4 (5) Å^3	0.18 × 0.16 × 0.16 mm
Z = 8	

Data collection

Bruker SMART 1000 CCD diffractometer	5788 independent reflections
Radiation source: fine-focus sealed tube	4324 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.032
T = 294(2) K	θ_{max} = 26.4º
ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −4→10
T_{min} = 0.925, T_{max} = 0.933	k = −29→29
16156 measured reflections	l = −17→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difmap and geom
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.094	$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.8236P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.002
5788 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
405 parameters	$\Delta\rho_{min}$ = −0.42 e Å^{-3}
47 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
S1	1.03471 (6)	0.62128 (2)	0.81960 (4)	0.03895 (14)	
S2	0.48816 (6)	0.39225 (2)	0.71431 (4)	0.03662 (14)	
O1	1.1118 (19)	0.7890 (4)	0.6842 (10)	0.121 (3)	0.57 (2)
O2	1.0429 (14)	0.7078 (3)	0.6537 (5)	0.076 (2)	0.57 (2)
O1'	1.1554 (13)	0.7854 (5)	0.7061 (11)	0.081 (3)	0.43 (2)
O2'	1.117 (2)	0.7006 (3)	0.6807 (11)	0.084 (4)	0.43 (2)
O3	1.19801 (18)	0.62714 (8)	0.80367 (14)	0.0666 (5)	
O4	0.9807 (2)	0.58378 (7)	0.89101 (11)	0.0608 (5)	
O5	0.3534 (3)	0.22474 (9)	0.78593 (15)	0.0826 (6)	
O6	0.4843 (2)	0.29184 (8)	0.85010 (12)	0.0700 (6)	
O7	0.33755 (17)	0.38247 (7)	0.75352 (12)	0.0521 (4)	
O8	0.5093 (2)	0.43636 (6)	0.64641 (11)	0.0495 (4)	
N1	1.0708 (3)	0.74400 (11)	0.71537 (18)	0.0639 (6)	
N2	0.96220 (19)	0.60102 (8)	0.71765 (12)	0.0375 (4)	
N3	0.5063 (2)	0.56738 (8)	0.63932 (14)	0.0424 (4)	
N4	0.4476 (2)	0.26339 (8)	0.78224 (14)	0.0469 (5)	
N5	0.6065 (2)	0.40375 (7)	0.80458 (12)	0.0359 (4)	
N6	1.0631 (2)	0.46045 (8)	0.81160 (13)	0.0410 (4)	
C1	0.9552 (2)	0.68867 (9)	0.85036 (15)	0.0378 (5)	
C2	0.9868 (3)	0.74034 (10)	0.80580 (17)	0.0464 (6)	
C3	0.9357 (3)	0.79051 (11)	0.8440 (2)	0.0681 (8)	
H3	0.9585	0.8245	0.8139	0.082*	
C4	0.8512 (4)	0.79018 (14)	0.9263 (3)	0.0779 (9)	
H4	0.8177	0.8241	0.9525	0.093*	
C5	0.8156 (3)	0.74069 (14)	0.9704 (2)	0.0696 (8)	
H5	0.7561	0.7410	1.0256	0.084*	
C6	0.8679 (3)	0.68969 (11)	0.93307 (16)	0.0512 (6)	
H6	0.8439	0.6561	0.9639	0.061*	
C7	0.8072 (2)	0.58892 (8)	0.69514 (13)	0.0298 (4)	
C8	0.7726 (2)	0.57271 (9)	0.60155 (14)	0.0368 (5)	
H8	0.8523	0.5691	0.5571	0.044*	
C9	0.6209 (3)	0.56218 (10)	0.57608 (16)	0.0433 (5)	
H9	0.5971	0.5513	0.5139	0.052*	

C10	0.5367 (2)	0.58106 (9)	0.72950 (16)	0.0417 (5)
H10	0.4544	0.5835	0.7724	0.050*
C11	0.6855 (2)	0.59159 (9)	0.76047 (14)	0.0369 (5)
H11	0.7056	0.6004	0.8241	0.044*
C12	0.5541 (2)	0.32936 (8)	0.65867 (14)	0.0337 (4)
C13	0.5231 (2)	0.27450 (9)	0.69050 (14)	0.0374 (5)
C14	0.5657 (3)	0.22817 (10)	0.63775 (17)	0.0490 (6)
H14	0.5416	0.1920	0.6590	0.059*
C15	0.6440 (3)	0.23546 (11)	0.55351 (18)	0.0565 (7)
H15	0.6737	0.2041	0.5181	0.068*
C16	0.6784 (3)	0.28830 (12)	0.52147 (17)	0.0574 (7)
H16	0.7331	0.2928	0.4649	0.069*
C17	0.6319 (3)	0.33548 (10)	0.57315 (15)	0.0449 (5)
H17	0.6533	0.3714	0.5500	0.054*
C18	0.7598 (2)	0.42265 (8)	0.80218 (13)	0.0315 (4)
C19	0.8394 (3)	0.42707 (9)	0.88944 (14)	0.0401 (5)
H19	0.7899	0.4169	0.9457	0.048*
C20	0.9895 (3)	0.44628 (10)	0.89160 (16)	0.0442 (5)
H20	1.0418	0.4496	0.9498	0.053*
C21	0.9919 (3)	0.45634 (9)	0.72684 (15)	0.0413 (5)
H21	1.0458	0.4663	0.6720	0.050*
C22	0.8408 (2)	0.43766 (9)	0.71951 (14)	0.0373 (5)
H22	0.7920	0.4349	0.6601	0.045*
Cl1	0.17769 (6)	0.55281 (3)	0.55557 (4)	0.04462 (15)
Cl2	0.47855 (7)	0.40063 (2)	1.01277 (4)	0.04624 (15)
H2A	1.033 (2)	0.5975 (9)	0.6730 (12)	0.046 (6)*
H3A	0.4067 (15)	0.5622 (11)	0.6200 (18)	0.067 (8)*
H5A	0.566 (3)	0.3957 (10)	0.8619 (10)	0.051 (7)*
H6A	1.1596 (16)	0.4746 (10)	0.8193 (16)	0.054 (7)*
O9	0.3370 (2)	0.49762 (7)	0.88677 (14)	0.0659 (5)
H9A	0.3647	0.5306	0.9052	0.079*
H9B	0.3606	0.4712	0.9262	0.079*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0333 (3)	0.0443 (3)	0.0391 (3)	0.0065 (2)	−0.0087 (2)	−0.0084 (2)
S2	0.0367 (3)	0.0363 (3)	0.0368 (3)	0.0019 (2)	−0.0038 (2)	−0.0046 (2)
O1	0.151 (7)	0.092 (4)	0.120 (6)	−0.017 (4)	0.020 (5)	0.040 (4)
O2	0.085 (5)	0.082 (3)	0.061 (3)	−0.002 (3)	0.022 (3)	0.007 (2)
O1'	0.059 (4)	0.081 (4)	0.102 (6)	−0.036 (3)	−0.004 (3)	0.041 (3)
O2'	0.103 (7)	0.073 (4)	0.075 (5)	−0.007 (4)	0.049 (5)	0.002 (3)
O3	0.0276 (8)	0.0855 (13)	0.0866 (13)	0.0101 (9)	−0.0136 (8)	−0.0393 (11)
O4	0.0938 (14)	0.0483 (10)	0.0400 (9)	0.0097 (10)	−0.0093 (9)	0.0078 (8)
O5	0.0980 (16)	0.0706 (13)	0.0795 (14)	−0.0444 (12)	0.0195 (12)	−0.0031 (11)
O6	0.0959 (15)	0.0742 (13)	0.0398 (9)	−0.0324 (11)	0.0064 (9)	−0.0037 (9)
O7	0.0333 (8)	0.0607 (10)	0.0623 (10)	0.0006 (8)	0.0023 (7)	−0.0177 (8)
O8	0.0672 (11)	0.0386 (8)	0.0426 (9)	0.0057 (8)	−0.0135 (8)	0.0036 (7)

N1	0.0532 (14)	0.0618 (15)	0.0769 (17)	−0.0045 (13)	0.0097 (12)	0.0191 (14)
N2	0.0243 (9)	0.0565 (11)	0.0318 (9)	−0.0019 (8)	0.0041 (7)	−0.0112 (8)
N3	0.0274 (9)	0.0438 (11)	0.0559 (12)	−0.0043 (8)	−0.0067 (9)	0.0009 (9)
N4	0.0528 (12)	0.0417 (11)	0.0463 (11)	−0.0074 (10)	0.0006 (9)	0.0040 (9)
N5	0.0367 (10)	0.0442 (10)	0.0268 (9)	−0.0064 (8)	0.0039 (7)	−0.0023 (8)
N6	0.0338 (10)	0.0394 (10)	0.0498 (11)	−0.0044 (8)	0.0013 (9)	0.0022 (8)
C1	0.0299 (11)	0.0431 (12)	0.0403 (11)	0.0027 (9)	−0.0032 (9)	−0.0075 (10)
C2	0.0359 (12)	0.0466 (13)	0.0568 (14)	−0.0003 (10)	−0.0005 (11)	−0.0015 (11)
C3	0.0618 (17)	0.0426 (15)	0.100 (2)	0.0033 (13)	−0.0060 (17)	−0.0067 (15)
C4	0.072 (2)	0.062 (2)	0.099 (2)	0.0185 (17)	−0.0040 (18)	−0.0375 (18)
C5	0.0628 (18)	0.085 (2)	0.0612 (17)	0.0119 (16)	0.0072 (14)	−0.0286 (16)
C6	0.0494 (14)	0.0611 (16)	0.0431 (13)	0.0021 (12)	0.0029 (11)	−0.0110 (12)
C7	0.0256 (10)	0.0310 (10)	0.0327 (10)	0.0015 (8)	−0.0002 (8)	−0.0005 (8)
C8	0.0330 (11)	0.0477 (12)	0.0297 (10)	0.0017 (10)	0.0022 (8)	−0.0018 (9)
C9	0.0418 (12)	0.0504 (13)	0.0375 (12)	−0.0028 (11)	−0.0080 (10)	−0.0012 (10)
C10	0.0302 (11)	0.0442 (12)	0.0510 (13)	0.0005 (10)	0.0104 (10)	−0.0021 (10)
C11	0.0327 (11)	0.0458 (12)	0.0324 (10)	0.0001 (9)	0.0054 (9)	−0.0075 (9)
C12	0.0323 (10)	0.0376 (11)	0.0310 (10)	−0.0003 (9)	−0.0044 (8)	−0.0041 (8)
C13	0.0374 (11)	0.0390 (12)	0.0358 (11)	−0.0026 (9)	−0.0053 (9)	−0.0015 (9)
C14	0.0535 (14)	0.0382 (12)	0.0551 (15)	0.0002 (11)	−0.0077 (12)	−0.0071 (11)
C15	0.0609 (16)	0.0526 (15)	0.0560 (15)	0.0090 (13)	−0.0011 (13)	−0.0214 (12)
C16	0.0622 (16)	0.0704 (18)	0.0397 (13)	0.0004 (14)	0.0113 (12)	−0.0127 (12)
C17	0.0533 (14)	0.0478 (13)	0.0337 (11)	−0.0057 (11)	0.0024 (10)	−0.0022 (10)
C18	0.0379 (11)	0.0255 (10)	0.0311 (10)	0.0006 (8)	0.0030 (8)	−0.0008 (8)
C19	0.0441 (12)	0.0486 (13)	0.0278 (10)	−0.0071 (11)	0.0031 (9)	0.0021 (9)
C20	0.0468 (13)	0.0482 (13)	0.0374 (12)	−0.0068 (11)	−0.0066 (10)	0.0000 (10)
C21	0.0430 (12)	0.0410 (12)	0.0400 (12)	−0.0004 (10)	0.0097 (10)	0.0038 (10)
C22	0.0436 (12)	0.0390 (12)	0.0294 (10)	−0.0016 (10)	0.0017 (9)	0.0014 (9)
Cl1	0.0289 (3)	0.0648 (4)	0.0403 (3)	−0.0078 (2)	0.0057 (2)	−0.0085 (3)
Cl2	0.0581 (4)	0.0463 (3)	0.0345 (3)	−0.0068 (3)	0.0103 (2)	−0.0017 (2)
O9	0.0635 (11)	0.0456 (10)	0.0881 (14)	−0.0060 (9)	−0.0275 (10)	−0.0018 (9)

Geometric parameters (Å, °)

S1—O4	1.4186 (18)	C4—H4	0.9300
S1—O3	1.4187 (17)	C5—C6	1.391 (4)
S1—N2	1.6248 (17)	C5—H5	0.9300
S1—C1	1.788 (2)	C6—H6	0.9300
S2—O7	1.4205 (16)	C7—C11	1.392 (3)
S2—O8	1.4262 (16)	C7—C8	1.396 (3)
S2—N5	1.6343 (17)	C8—C9	1.362 (3)
S2—C12	1.775 (2)	C8—H8	0.9300
O1—N1	1.204 (7)	C9—H9	0.9300
O2—N1	1.240 (6)	C10—C11	1.360 (3)
O1'—N1	1.225 (7)	C10—H10	0.9300
O2'—N1	1.206 (7)	C11—H11	0.9300
O5—N4	1.219 (2)	C12—C17	1.383 (3)
O6—N4	1.205 (2)	C12—C13	1.400 (3)
N1—C2	1.465 (3)	C13—C14	1.374 (3)

N2—C7	1.385 (2)	C14—C15	1.373 (3)
N2—H2A	0.878 (10)	C14—H14	0.9300
N3—C10	1.328 (3)	C15—C16	1.363 (4)
N3—C9	1.331 (3)	C15—H15	0.9300
N3—H3A	0.897 (10)	C16—C17	1.392 (3)
N4—C13	1.468 (3)	C16—H16	0.9300
N5—C18	1.382 (3)	C17—H17	0.9300
N5—H5A	0.897 (10)	C18—C19	1.398 (3)
N6—C21	1.333 (3)	C18—C22	1.401 (3)
N6—C20	1.333 (3)	C19—C20	1.358 (3)
N6—H6A	0.894 (10)	C19—H19	0.9300
C1—C6	1.384 (3)	C20—H20	0.9300
C1—C2	1.401 (3)	C21—C22	1.365 (3)
C2—C3	1.376 (3)	C21—H21	0.9300
C3—C4	1.366 (4)	C22—H22	0.9300
C3—H3	0.9300	O9—H9A	0.8562
C4—C5	1.361 (4)	O9—H9B	0.8575
O4—S1—O3	119.87 (12)	C1—C6—C5	120.5 (3)
O4—S1—N2	108.22 (10)	C1—C6—H6	119.8
O3—S1—N2	104.84 (10)	C5—C6—H6	119.8
O4—S1—C1	105.20 (10)	N2—C7—C11	123.75 (18)
O3—S1—C1	108.99 (11)	N2—C7—C8	117.63 (18)
N2—S1—C1	109.51 (10)	C11—C7—C8	118.62 (18)
O7—S2—O8	119.89 (11)	C9—C8—C7	119.3 (2)
O7—S2—N5	106.33 (9)	C9—C8—H8	120.4
O8—S2—N5	108.30 (9)	C7—C8—H8	120.4
O7—S2—C12	108.93 (10)	N3—C9—C8	120.6 (2)
O8—S2—C12	106.19 (10)	N3—C9—H9	119.7
N5—S2—C12	106.53 (9)	C8—C9—H9	119.7
O1—N1—O2'	120.7 (8)	N3—C10—C11	121.4 (2)
O1—N1—O1'	23.1 (9)	N3—C10—H10	119.3
O2'—N1—O1'	116.4 (10)	C11—C10—H10	119.3
O1—N1—O2	114.4 (9)	C10—C11—C7	118.70 (19)
O2'—N1—O2	35.9 (6)	C10—C11—H11	120.6
O1'—N1—O2	126.1 (8)	C7—C11—H11	120.6
O1—N1—C2	120.9 (7)	C17—C12—C13	117.85 (19)
O2'—N1—C2	117.6 (5)	C17—C12—S2	116.70 (16)
O1'—N1—C2	115.6 (8)	C13—C12—S2	125.25 (16)
O2—N1—C2	118.1 (4)	C14—C13—C12	121.2 (2)
C7—N2—S1	128.01 (14)	C14—C13—N4	116.7 (2)
C7—N2—H2A	118.6 (15)	C12—C13—N4	122.11 (18)
S1—N2—H2A	113.4 (15)	C15—C14—C13	119.7 (2)
C10—N3—C9	121.26 (19)	C15—C14—H14	120.1
C10—N3—H3A	119.8 (17)	C13—C14—H14	120.1
C9—N3—H3A	118.9 (17)	C16—C15—C14	120.5 (2)
O6—N4—O5	123.6 (2)	C16—C15—H15	119.8
O6—N4—C13	118.63 (19)	C14—C15—H15	119.8
O5—N4—C13	117.80 (19)	C15—C16—C17	120.2 (2)
C18—N5—S2	127.75 (14)	C15—C16—H16	119.9

C18—N5—H5A	117.5 (15)	C17—C16—H16	119.9
S2—N5—H5A	114.8 (15)	C12—C17—C16	120.6 (2)
C21—N6—C20	121.20 (19)	C12—C17—H17	119.7
C21—N6—H6A	123.1 (15)	C16—C17—H17	119.7
C20—N6—H6A	115.6 (15)	N5—C18—C19	117.04 (17)
C6—C1—C2	117.7 (2)	N5—C18—C22	125.15 (18)
C6—C1—S1	115.20 (18)	C19—C18—C22	117.82 (19)
C2—C1—S1	126.65 (17)	C20—C19—C18	119.63 (19)
C3—C2—C1	121.2 (2)	C20—C19—H19	120.2
C3—C2—N1	116.4 (2)	C18—C19—H19	120.2
C1—C2—N1	122.4 (2)	N6—C20—C19	121.1 (2)
C4—C3—C2	119.7 (3)	N6—C20—H20	119.5
C4—C3—H3	120.2	C19—C20—H20	119.5
C2—C3—H3	120.2	N6—C21—C22	120.8 (2)
C5—C4—C3	120.7 (3)	N6—C21—H21	119.6
C5—C4—H4	119.7	C22—C21—H21	119.6
C3—C4—H4	119.7	C21—C22—C18	119.47 (19)
C4—C5—C6	120.2 (3)	C21—C22—H22	120.3
C4—C5—H5	119.9	C18—C22—H22	120.3
C6—C5—H5	119.9	H9A—O9—H9B	114.1
O4—S1—N2—C7	50.4 (2)	C7—C8—C9—N3	0.2 (3)
O3—S1—N2—C7	179.47 (19)	C9—N3—C10—C11	1.6 (3)
C1—S1—N2—C7	−63.7 (2)	N3—C10—C11—C7	1.3 (3)
O7—S2—N5—C18	−170.52 (18)	N2—C7—C11—C10	177.3 (2)
O8—S2—N5—C18	−40.4 (2)	C8—C7—C11—C10	−3.2 (3)
C12—S2—N5—C18	73.41 (19)	O7—S2—C12—C17	140.21 (16)
O4—S1—C1—C6	1.64 (19)	O8—S2—C12—C17	9.81 (19)
O3—S1—C1—C6	−128.10 (18)	N5—S2—C12—C17	−105.48 (17)
N2—S1—C1—C6	117.74 (17)	O7—S2—C12—C13	−34.6 (2)
O4—S1—C1—C2	174.05 (19)	O8—S2—C12—C13	−164.97 (17)
O3—S1—C1—C2	44.3 (2)	N5—S2—C12—C13	79.74 (19)
N2—S1—C1—C2	−69.9 (2)	C17—C12—C13—C14	−1.2 (3)
C6—C1—C2—C3	1.4 (3)	S2—C12—C13—C14	173.48 (17)
S1—C1—C2—C3	−170.88 (19)	C17—C12—C13—N4	177.22 (19)
C6—C1—C2—N1	−176.7 (2)	S2—C12—C13—N4	−8.1 (3)
S1—C1—C2—N1	11.1 (3)	O6—N4—C13—C14	138.6 (2)
O1—N1—C2—C3	10.3 (10)	O5—N4—C13—C14	−39.6 (3)
O2'—N1—C2—C3	179.7 (13)	O6—N4—C13—C12	−39.9 (3)
O1'—N1—C2—C3	35.9 (7)	O5—N4—C13—C12	141.8 (2)
O2—N1—C2—C3	−139.5 (7)	C12—C13—C14—C15	1.8 (3)
O1—N1—C2—C1	−171.6 (10)	N4—C13—C14—C15	−176.7 (2)
O2'—N1—C2—C1	−2.2 (13)	C13—C14—C15—C16	−0.6 (4)
O1'—N1—C2—C1	−145.9 (7)	C14—C15—C16—C17	−1.2 (4)
O2—N1—C2—C1	38.6 (8)	C13—C12—C17—C16	−0.5 (3)
C1—C2—C3—C4	−0.7 (4)	S2—C12—C17—C16	−175.69 (18)
N1—C2—C3—C4	177.5 (3)	C15—C16—C17—C12	1.7 (4)
C2—C3—C4—C5	−0.7 (5)	S2—N5—C18—C19	−177.51 (16)
C3—C4—C5—C6	1.4 (5)	S2—N5—C18—C22	2.6 (3)
C2—C1—C6—C5	−0.7 (3)	N5—C18—C19—C20	−178.9 (2)

S1—C1—C6—C5	172.4 (2)	C22—C18—C19—C20	1.0 (3)
C4—C5—C6—C1	−0.6 (4)	C21—N6—C20—C19	0.1 (3)
S1—N2—C7—C11	−1.2 (3)	C18—C19—C20—N6	−0.8 (3)
S1—N2—C7—C8	179.33 (16)	C20—N6—C21—C22	0.3 (3)
N2—C7—C8—C9	−178.0 (2)	N6—C21—C22—C18	0.0 (3)
C11—C7—C8—C9	2.5 (3)	N5—C18—C22—C21	179.3 (2)
C10—N3—C9—C8	−2.3 (3)	C19—C18—C22—C21	−0.6 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···Cl1^{i}	0.878 (10)	2.321 (13)	3.1500 (17)	157 (2)
N3—H3A···Cl1	0.897 (10)	2.155 (11)	3.0472 (18)	173 (2)
N5—H5A···Cl2	0.897 (10)	2.253 (11)	3.1270 (17)	164 (2)
N6—H6A···O9^{i}	0.894 (10)	1.858 (13)	2.700 (2)	156 (2)
O9—H9A···Cl2^{ii}	0.86	2.39	3.1979 (18)	157
O9—H9B···Cl2	0.86	2.29	3.1332 (19)	167

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

Fig. 1

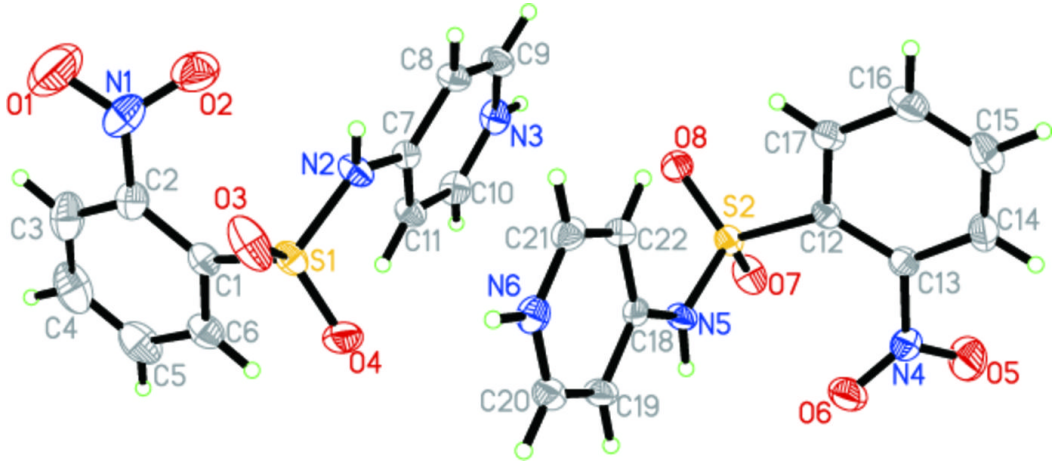

O1
N1
O2
C2
C3
C4
C5
C6
C1
O3
S1
O4
N2
C7
C8
C9
N3
C10
C11
O8
S2
O7
C21
C22
N6
C18
N5
C20
C19
C12
C16
C17
C15
C14
C13
N4
O5
O6

Fig. 2