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4-Amino-2-methylquinolinium hydrogensulfate dihydrate

Mostafa M. Amini,* Gholamhossein Mohammadnezhad Sh. and Hamid Reza Khavasi

Department of Chemistry, Shahid Beheshti University, Evin, Tehran 1983963113, Iran
Correspondence e-mail: m-pouramini@cc.sbu.ac.ir

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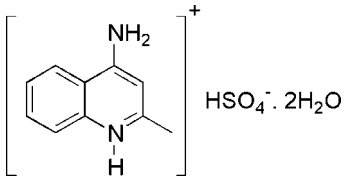
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.003 Å; R factor = 0.060; wR factor = 0.182; data-to-parameter ratio = 17.1.

In the title compound, $C_{10}H_{11}N_2^+\cdot HSO_4^-\cdot 2H_2O$, the asymmetric unit contains two protonated 4-aminoquinoline cations and two hydrogen sulfate anions with four water molecules. The crystal structure involves extensive N—H···O and O—H···O hydrogen bonding.

Related literature

For related literature, see: Amini *et al.* (2007*a*,*b*); Repicky *et al.* (2005).



Experimental

Crystal data

$C_{10}H_{11}N_2^+\cdot HSO_4^-\cdot 2H_2O$
M_r = 292.32
Triclinic, $P\overline{1}$
a = 10.1585 (9) Å
b = 11.2131 (9) Å
c = 13.3545 (11) Å
α = 68.283 (6)°
β = 76.355 (7)°
γ = 67.949 (6)°
V = 1301.51 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 120 (2) K
0.5 × 0.15 × 0.12 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 2005)
T_{min} = 0.950, T_{max} = 0.970
16357 measured reflections
7008 independent reflections
5729 reflections with $I > 2\sigma(I)$
R_{int} = 0.087

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.182
S = 1.06
7008 reflections
409 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.99 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O7^{i}	0.89 (3)	2.16 (3)	2.981 (3)	154 (3)
N1—H1C···O7^{ii}	0.88 (3)	2.26 (3)	3.049 (3)	149 (3)
N1—H1C···O8^{ii}	0.88 (3)	2.58 (3)	3.374 (3)	151 (3)
O1—H1D···O9	0.88 (4)	1.61 (4)	2.485 (3)	177 (5)
N2—H2B···O11^{iii}	0.95 (3)	1.88 (3)	2.819 (3)	172 (3)
N3—H3B···O3^{iv}	0.90 (3)	2.03 (3)	2.902 (3)	163 (3)
N3—H3C···O3	0.87 (3)	2.54 (3)	3.177 (3)	132 (3)
N3—H3C···O4	0.87 (3)	2.20 (3)	3.034 (3)	162 (3)
N4—H4B···O12	0.90 (3)	1.87 (3)	2.753 (3)	169 (3)
O5—H5···O10	0.94 (4)	1.58 (4)	2.523 (3)	174 (5)
O9—H9D···O8	0.84 (4)	1.91 (4)	2.736 (3)	169 (4)
O9—H9E···O6^{v}	0.81 (4)	1.98 (4)	2.783 (3)	178 (5)
O10—H10A···O4	0.89 (4)	1.81 (4)	2.695 (3)	175 (3)
O10—H10B···O2^{vi}	0.83 (4)	1.93 (4)	2.745 (3)	169 (4)
O11—H11B···O2^{vi}	0.82 (4)	2.04 (4)	2.843 (3)	167 (5)
O11—H11C···O7	0.85 (4)	1.94 (4)	2.787 (3)	172 (3)
O12—H12B···O3^{vii}	0.86 (4)	1.98 (4)	2.823 (3)	166 (4)
O12—H12C···O11^{ii}	0.92 (4)	1.90 (4)	2.823 (3)	177 (4)

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

Data collection: *X-RED32* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2193).

References

Amini, M. M., Mohammadnezhad, G. S. & Khavasi, H. R. (2007*b*). *Acta Cryst.* E**63**, m2516.
Amini, M. M., Nasiri, S. & Ng, S. W. (2007*a*). *Acta Cryst.* E**63**, o1361–o1362.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Repicky, A., Jantova, S., Theiszova, M. & Milata, V. (2005). *Biomed. Pap. Med. Fac. Univ. Palacky Olomouc Czech. Rep.* **49**, 345–347.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stoe & Cie (2005). *X-RED32* (Version 1.28b), *X-SHAPE* (Version 2.05) and *X-AREA* (Version 1.31). Stoe & Cie, Darmstadt, Germany.

supplementary materials

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4-Amino-2-methylquinolinium hydrogensulfate dihydrate

M. M. Amini, G. Mohammadnezhad Sh. and H. R. Khavasi

Comment

In continuation of our research on the structure determination of simple ammonium salts (Amini *et al.*, 2007*a*) as precursors for synthesis of double sulfate (Amini *et al.*, 2007*b*), we have found that the this compound (Fig. 1) forms structures with extended hydrogen bonding in the presence of water molecules. From the packing diagram (Fig. 2), it seems that the intermolecular and intramolecular N—H···O and O—H···O and O—H···O hydrogen bonds (Table 1) are effective in the stabilization of the crystal structure. These compounds could be classified as an outstanding model system for polymers with higher dimensional hydrogen bonding. 4-aminoquinoline derivatives have cytotoxic activity (Repicky *et al.*, 2005), so determination of molecular structure of these compounds could be beneficial. As it is shown in Fig. 2, the quinoline rings form π bonding stacks. The closest contact distance between adjacent aromatic rings is 3.328 (3) A°.

Experimental

2-methylquinolin-4-amine (1.58 g, 0.01 mol) was dissolved in 30 ml dichloromethane. Dropwise addition of concentrated sulfuric acid (0.98 g, 0.01 mol) resulted in a white precipitate which was filtered and dissolved in methanol. Needle shape crystals were grown by slow evaporation of a methanol solution at room temperature.

Refinement

The N-bond and O-bond H atoms were located in a difference map and their positions were freely refined. Other hydrogen atoms were refined using a riding model (C—H = 0.93–0.96 A°) with their displacement parameters set at 1.2 times U_{eq} of the parent atom.

Figures

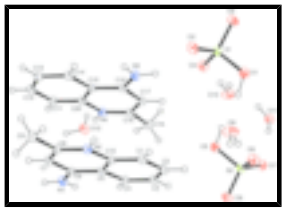

Fig. 1. Molecular structure. Displacement ellipsoids are drawn at the 30% probability level

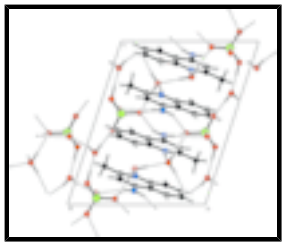

Fig. 2. Unit-cell packing diagram as viewed down the a-direction. Hydrogen bonds are shown as dashed lines.

4-Amino-2-methylquinolinium hydrogensulfate dihydrate

Crystal data

$C_{10}H_{11}N_2^+ \cdot HSO_4^- \cdot 2H_2O$	$Z = 4$
$M_r = 292.32$	$F_{000} = 616$
Triclinic, $P\overline{1}$	$D_x = 1.492$ Mg m^{-3}
$a = 10.1585$ (9) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 11.2131$ (9) Å	Cell parameters from 2500 reflections
$c = 13.3545$ (11) Å	$\theta = 1.7$–29.2º
$\alpha = 68.283$ (6)º	$\mu = 0.27$ mm^{-1}
$\beta = 76.355$ (7)º	$T = 120$ (2) K
$\gamma = 67.949$ (6)º	Needle, colorless
$V = 1301.51$ (19) Å^3	0.5 × 0.15 × 0.12 mm

Data collection

Stoe IPDSII diffractometer	$R_{int} = 0.087$
ω scans	$\theta_{max} = 29.2$º
Absorption correction: numerical (X-SHAPE; Stoe & Cie, 2005)	$\theta_{min} = 1.7$º
$T_{min} = 0.950$, $T_{max} = 0.970$	$h = -13 \rightarrow 13$
16357 measured reflections	$k = -15 \rightarrow 15$
7008 independent reflections	$l = -18 \rightarrow 16$
5729 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0853P)^2 + 1.1102P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.060$	$(\Delta/\sigma)_{max} = 0.019$
$wR(F^2) = 0.182$	$\Delta\rho_{max} = 0.39$ e Å^{-3}
$S = 1.06$	$\Delta\rho_{min} = -0.99$ e Å^{-3}
7008 reflections	Extinction correction: none
409 parameters	

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.2667 (2)	0.3412 (2)	0.37942 (17)	0.0230 (4)
H1A	−0.1789	0.3565	0.3583	0.028*
C2	−0.2725 (2)	0.2116 (2)	0.42028 (19)	0.0251 (4)
H2A	−0.1882	0.1389	0.4268	0.03*
C3	−0.4052 (2)	0.1875 (2)	0.45255 (19)	0.0251 (4)
H3A	−0.408	0.0991	0.48	0.03*
C4	−0.5305 (2)	0.2943 (2)	0.44357 (17)	0.0228 (4)
H4A	−0.6174	0.2772	0.4648	0.027*
C5	−0.5292 (2)	0.4296 (2)	0.40258 (16)	0.0206 (4)
C6	−0.6557 (2)	0.5461 (2)	0.39237 (17)	0.0208 (4)
C7	−0.6404 (2)	0.6757 (2)	0.34890 (17)	0.0228 (4)
H7	−0.7215	0.7519	0.3411	0.027*
C8	−0.5072 (2)	0.6914 (2)	0.31778 (17)	0.0217 (4)
C9	−0.4852 (3)	0.8274 (2)	0.27189 (19)	0.0274 (5)
H9C	−0.4342	0.8368	0.2005	0.033*
H9B	−0.4311	0.8351	0.3179	0.033*
H9A	−0.5764	0.8973	0.2679	0.033*
C10	−0.3942 (2)	0.4510 (2)	0.36953 (17)	0.0205 (4)
C11	−0.7437 (2)	0.6655 (2)	0.12158 (18)	0.0240 (4)
H11A	−0.8339	0.6553	0.1415	0.029*
C12	−0.7303 (2)	0.7924 (2)	0.08060 (19)	0.0268 (4)
H12A	−0.8116	0.8683	0.0736	0.032*
C13	−0.5935 (2)	0.8088 (2)	0.04894 (19)	0.0257 (4)
H13	−0.5854	0.8953	0.0208	0.031*
C14	−0.4726 (2)	0.6974 (2)	0.05956 (17)	0.0230 (4)
H14	−0.3832	0.7093	0.0386	0.028*
C15	−0.4823 (2)	0.5647 (2)	0.10199 (16)	0.0203 (4)
C16	−0.3599 (2)	0.4439 (2)	0.11442 (17)	0.0202 (4)
C17	−0.3833 (2)	0.3176 (2)	0.15913 (17)	0.0217 (4)
H17	−0.3055	0.2385	0.1688	0.026*
C18	−0.5200 (2)	0.3094 (2)	0.18876 (17)	0.0216 (4)
C19	−0.5493 (2)	0.1767 (2)	0.23516 (18)	0.0253 (4)
H19A	−0.4617	0.1044	0.253	0.03*
H19B	−0.5889	0.1641	0.1827	0.03*
H19C	−0.6159	0.1767	0.2995	0.03*
C20	−0.6205 (2)	0.5506 (2)	0.13349 (17)	0.0207 (4)
N1	−0.7861 (2)	0.5326 (2)	0.42328 (17)	0.0264 (4)
H1B	−0.799 (3)	0.452 (3)	0.450 (2)	0.035 (8)*
H1C	−0.863 (3)	0.604 (3)	0.421 (2)	0.034 (8)*
N2	−0.38895 (19)	0.58169 (19)	0.32826 (15)	0.0215 (4)
H2B	−0.297 (3)	0.591 (3)	0.304 (2)	0.033 (8)*
N3	−0.2264 (2)	0.4497 (2)	0.08604 (16)	0.0241 (4)
H3B	−0.206 (3)	0.528 (3)	0.056 (3)	0.041 (9)*
H3C	−0.156 (3)	0.375 (3)	0.090 (2)	0.030 (7)*
N4	−0.63363 (19)	0.42260 (19)	0.17560 (15)	0.0217 (4)

H4B	−0.724 (3)	0.421 (3)	0.197 (2)	0.024 (7)*
O1	0.21598 (18)	0.09186 (18)	0.12957 (15)	0.0304 (4)
H1D	0.174 (4)	0.085 (4)	0.196 (3)	0.049 (10)*
O2	0.19440 (18)	0.16254 (17)	−0.05753 (13)	0.0285 (4)
O3	0.11031 (19)	0.33038 (17)	0.03498 (15)	0.0316 (4)
O4	−0.02002 (17)	0.17733 (17)	0.07138 (14)	0.0279 (4)
O5	−0.21953 (18)	−0.06596 (18)	0.35374 (15)	0.0299 (4)
H5	−0.169 (4)	−0.059 (4)	0.284 (3)	0.054 (10)*
O6	−0.20017 (18)	−0.15065 (18)	0.54201 (14)	0.0307 (4)
O7	−0.10350 (19)	−0.30542 (17)	0.43617 (14)	0.0306 (4)
O8	0.01481 (18)	−0.14707 (19)	0.41604 (15)	0.0328 (4)
O9	0.1039 (3)	0.0757 (2)	0.31884 (16)	0.0445 (5)
H9D	0.080 (4)	0.007 (4)	0.356 (3)	0.056 (11)*
H9E	0.133 (4)	0.098 (4)	0.358 (3)	0.047 (10)*
O10	−0.0982 (2)	−0.0478 (2)	0.16311 (16)	0.0396 (5)
H10A	−0.073 (4)	0.026 (4)	0.129 (3)	0.050 (10)*
H10B	−0.127 (4)	−0.073 (3)	0.124 (3)	0.042 (9)*
O11	−0.12512 (18)	−0.36765 (18)	0.25826 (15)	0.0276 (4)
H11C	−0.112 (3)	−0.346 (3)	0.309 (3)	0.038 (8)*
H11B	−0.137 (4)	−0.303 (4)	0.203 (3)	0.070 (13)*
O12	−0.90519 (19)	0.4039 (2)	0.21908 (16)	0.0316 (4)
H12B	−0.916 (4)	0.386 (4)	0.165 (3)	0.058 (11)*
H12C	−0.979 (4)	0.478 (4)	0.231 (3)	0.063 (11)*
S1	0.12067 (5)	0.19469 (5)	0.04176 (4)	0.02081 (14)
S2	−0.12161 (5)	−0.17207 (5)	0.44180 (4)	0.02107 (14)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0200 (10)	0.0298 (11)	0.0256 (10)	−0.0120 (8)	−0.0022 (8)	−0.0116 (8)
C2	0.0199 (10)	0.0276 (11)	0.0317 (11)	−0.0074 (8)	−0.0033 (8)	−0.0137 (9)
C3	0.0260 (11)	0.0250 (10)	0.0304 (11)	−0.0135 (9)	−0.0012 (8)	−0.0111 (8)
C4	0.0205 (10)	0.0282 (11)	0.0258 (10)	−0.0132 (8)	−0.0009 (8)	−0.0108 (8)
C5	0.0196 (9)	0.0262 (10)	0.0220 (9)	−0.0122 (8)	−0.0019 (7)	−0.0095 (8)
C6	0.0168 (9)	0.0281 (10)	0.0224 (9)	−0.0096 (8)	−0.0002 (7)	−0.0118 (8)
C7	0.0223 (10)	0.0237 (10)	0.0258 (10)	−0.0077 (8)	−0.0040 (8)	−0.0102 (8)
C8	0.0222 (10)	0.0240 (10)	0.0228 (9)	−0.0099 (8)	−0.0033 (7)	−0.0086 (8)
C9	0.0317 (12)	0.0254 (11)	0.0297 (11)	−0.0148 (9)	−0.0041 (9)	−0.0076 (8)
C10	0.0197 (9)	0.0247 (10)	0.0230 (9)	−0.0109 (8)	−0.0022 (7)	−0.0102 (8)
C11	0.0184 (9)	0.0310 (11)	0.0278 (10)	−0.0109 (8)	−0.0033 (8)	−0.0113 (8)
C12	0.0213 (10)	0.0281 (11)	0.0329 (11)	−0.0059 (8)	−0.0057 (8)	−0.0121 (9)
C13	0.0266 (11)	0.0245 (10)	0.0307 (11)	−0.0120 (9)	−0.0034 (9)	−0.0098 (8)
C14	0.0213 (10)	0.0275 (10)	0.0253 (10)	−0.0126 (8)	−0.0030 (8)	−0.0086 (8)
C15	0.0192 (9)	0.0264 (10)	0.0212 (9)	−0.0112 (8)	−0.0019 (7)	−0.0101 (8)
C16	0.0178 (9)	0.0268 (10)	0.0225 (9)	−0.0113 (8)	−0.0015 (7)	−0.0109 (8)
C17	0.0176 (9)	0.0252 (10)	0.0278 (10)	−0.0091 (8)	−0.0035 (8)	−0.0115 (8)
C18	0.0201 (9)	0.0260 (10)	0.0242 (10)	−0.0121 (8)	−0.0025 (7)	−0.0091 (8)
C19	0.0258 (10)	0.0272 (11)	0.0288 (11)	−0.0154 (9)	−0.0009 (8)	−0.0097 (8)

C20	0.0192 (9)	0.0251 (10)	0.0228 (9)	−0.0102 (8)	−0.0035 (7)	−0.0092 (8)
N1	0.0177 (9)	0.0285 (10)	0.0355 (10)	−0.0099 (8)	−0.0012 (7)	−0.0112 (8)
N2	0.0199 (8)	0.0259 (9)	0.0248 (8)	−0.0121 (7)	−0.0015 (7)	−0.0106 (7)
N3	0.0160 (9)	0.0269 (10)	0.0325 (10)	−0.0104 (8)	−0.0011 (7)	−0.0101 (8)
N4	0.0171 (8)	0.0292 (9)	0.0255 (9)	−0.0130 (7)	−0.0011 (6)	−0.0109 (7)
O1	0.0226 (8)	0.0384 (10)	0.0315 (9)	−0.0084 (7)	−0.0048 (7)	−0.0127 (7)
O2	0.0302 (8)	0.0337 (9)	0.0284 (8)	−0.0149 (7)	0.0030 (6)	−0.0162 (7)
O3	0.0305 (9)	0.0282 (8)	0.0435 (10)	−0.0172 (7)	0.0046 (7)	−0.0167 (7)
O4	0.0183 (7)	0.0330 (9)	0.0387 (9)	−0.0157 (7)	−0.0021 (6)	−0.0111 (7)
O5	0.0238 (8)	0.0329 (9)	0.0346 (9)	−0.0071 (7)	−0.0045 (7)	−0.0135 (7)
O6	0.0300 (9)	0.0380 (9)	0.0305 (8)	−0.0175 (7)	0.0027 (7)	−0.0148 (7)
O7	0.0325 (9)	0.0268 (8)	0.0367 (9)	−0.0127 (7)	−0.0027 (7)	−0.0121 (7)
O8	0.0206 (8)	0.0443 (10)	0.0398 (10)	−0.0194 (7)	−0.0011 (7)	−0.0124 (8)
O9	0.0656 (14)	0.0587 (13)	0.0308 (10)	−0.0463 (12)	0.0001 (9)	−0.0143 (9)
O10	0.0614 (13)	0.0439 (11)	0.0288 (9)	−0.0365 (10)	−0.0023 (8)	−0.0099 (8)
O11	0.0253 (8)	0.0320 (9)	0.0328 (9)	−0.0150 (7)	−0.0018 (7)	−0.0130 (7)
O12	0.0208 (8)	0.0380 (10)	0.0445 (10)	−0.0133 (7)	−0.0023 (7)	−0.0191 (8)
S1	0.0171 (3)	0.0242 (3)	0.0272 (3)	−0.0123 (2)	−0.00033 (19)	−0.0103 (2)
S2	0.0170 (3)	0.0253 (3)	0.0263 (3)	−0.0114 (2)	−0.00057 (19)	−0.0105 (2)

Geometric parameters (Å, °)

C1—C2	1.369 (3)	C16—C17	1.407 (3)
C1—C10	1.405 (3)	C17—C18	1.380 (3)
C1—H1A	0.93	C17—H17	0.93
C2—C3	1.411 (3)	C18—N4	1.344 (3)
C2—H2A	0.93	C18—C19	1.499 (3)
C3—C4	1.374 (3)	C19—H19A	0.96
C3—H3A	0.93	C19—H19B	0.96
C4—C5	1.414 (3)	C19—H19C	0.96
C4—H4A	0.93	C20—N4	1.380 (3)
C5—C10	1.420 (3)	N1—H1B	0.89 (3)
C5—C6	1.437 (3)	N1—H1C	0.89 (3)
C6—N1	1.339 (3)	N2—H2B	0.95 (3)
C6—C7	1.406 (3)	N3—H3B	0.90 (3)
C7—C8	1.376 (3)	N3—H3C	0.86 (3)
C7—H7	0.93	N4—H4B	0.90 (3)
C8—N2	1.348 (3)	O1—S1	1.5293 (18)
C8—C9	1.500 (3)	O1—H1D	0.87 (4)
C9—H9C	0.96	O2—S1	1.4601 (17)
C9—H9B	0.96	O3—S1	1.4548 (16)
C9—H9A	0.96	O4—S1	1.4568 (15)
C10—N2	1.379 (3)	O5—S2	1.5543 (18)
C11—C12	1.370 (3)	O5—H5	0.94 (4)
C11—C20	1.408 (3)	O6—S2	1.4406 (17)
C11—H11A	0.93	O7—S2	1.4646 (17)
C12—C13	1.414 (3)	O8—S2	1.4536 (15)
C12—H12A	0.93	O9—H9D	0.84 (4)
C13—C14	1.374 (3)	O9—H9E	0.81 (4)

C13—H13	0.93	O10—H10A	0.88 (4)
C14—C15	1.415 (3)	O10—H10B	0.82 (3)
C14—H14	0.93	O11—H11C	0.85 (3)
C15—C20	1.419 (3)	O11—H11B	0.82 (4)
C15—C16	1.440 (3)	O12—H12B	0.86 (4)
C16—N3	1.339 (2)	O12—H12C	0.92 (4)
C2—C1—C10	119.60 (19)	N3—C16—C17	120.0 (2)
C2—C1—H1A	120.2	N3—C16—C15	121.66 (19)
C10—C1—H1A	120.2	C17—C16—C15	118.32 (18)
C1—C2—C3	120.6 (2)	C18—C17—C16	121.0 (2)
C1—C2—H2A	119.7	C18—C17—H17	119.5
C3—C2—H2A	119.7	C16—C17—H17	119.5
C4—C3—C2	120.19 (19)	N4—C18—C17	120.30 (19)
C4—C3—H3A	119.9	N4—C18—C19	117.20 (18)
C2—C3—H3A	119.9	C17—C18—C19	122.5 (2)
C3—C4—C5	121.01 (19)	C18—C19—H19A	109.5
C3—C4—H4A	119.5	C18—C19—H19B	109.5
C5—C4—H4A	119.5	H19A—C19—H19B	109.5
C4—C5—C10	117.63 (19)	C18—C19—H19C	109.5
C4—C5—C6	124.08 (18)	H19A—C19—H19C	109.5
C10—C5—C6	118.30 (18)	H19B—C19—H19C	109.5
N1—C6—C7	120.1 (2)	N4—C20—C11	119.97 (18)
N1—C6—C5	121.16 (19)	N4—C20—C15	119.33 (19)
C7—C6—C5	118.71 (18)	C11—C20—C15	120.70 (19)
C8—C7—C6	120.9 (2)	C6—N1—H1B	122 (2)
C8—C7—H7	119.6	C6—N1—H1C	120.9 (19)
C6—C7—H7	119.6	H1B—N1—H1C	117 (3)
N2—C8—C7	120.14 (19)	C8—N2—C10	122.78 (17)
N2—C8—C9	116.95 (18)	C8—N2—H2B	121.3 (18)
C7—C8—C9	122.9 (2)	C10—N2—H2B	115.9 (18)
C8—C9—H9C	109.5	C16—N3—H3B	123 (2)
C8—C9—H9B	109.5	C16—N3—H3C	118.9 (19)
H9C—C9—H9B	109.5	H3B—N3—H3C	118 (3)
C8—C9—H9A	109.5	C18—N4—C20	122.61 (17)
H9C—C9—H9A	109.5	C18—N4—H4B	122.8 (17)
H9B—C9—H9A	109.5	C20—N4—H4B	114.6 (17)
N2—C10—C1	119.84 (18)	S1—O1—H1D	114 (2)
N2—C10—C5	119.20 (19)	S2—O5—H5	111 (2)
C1—C10—C5	120.96 (19)	H9D—O9—H9E	109 (4)
C12—C11—C20	119.83 (19)	H10A—O10—H10B	114 (3)
C12—C11—H11A	120.1	H11C—O11—H11B	111 (3)
C20—C11—H11A	120.1	H12B—O12—H12C	111 (3)
C11—C12—C13	120.4 (2)	O3—S1—O4	110.45 (10)
C11—C12—H12A	119.8	O3—S1—O2	111.62 (10)
C13—C12—H12A	119.8	O4—S1—O2	112.86 (10)
C14—C13—C12	120.2 (2)	O3—S1—O1	108.74 (10)
C14—C13—H13	119.9	O4—S1—O1	108.47 (10)
C12—C13—H13	119.9	O2—S1—O1	104.41 (10)
C13—C14—C15	120.93 (19)	O6—S2—O8	113.87 (10)

C13—C14—H14	119.5	O6—S2—O7	112.78 (10)
C15—C14—H14	119.5	O8—S2—O7	110.74 (11)
C14—C15—C20	117.89 (19)	O6—S2—O5	104.17 (10)
C14—C15—C16	123.65 (18)	O8—S2—O5	108.13 (10)
C20—C15—C16	118.47 (18)	O7—S2—O5	106.56 (10)
C10—C1—C2—C3	0.0 (3)	C13—C14—C15—C16	179.5 (2)
C1—C2—C3—C4	−0.2 (3)	C14—C15—C16—N3	0.0 (3)
C2—C3—C4—C5	−0.3 (3)	C20—C15—C16—N3	179.79 (19)
C3—C4—C5—C10	1.0 (3)	C14—C15—C16—C17	179.10 (19)
C3—C4—C5—C6	−179.1 (2)	C20—C15—C16—C17	−1.2 (3)
C4—C5—C6—N1	0.8 (3)	N3—C16—C17—C18	−179.9 (2)
C10—C5—C6—N1	−179.22 (19)	C15—C16—C17—C18	1.0 (3)
C4—C5—C6—C7	−179.12 (19)	C16—C17—C18—N4	−0.2 (3)
C10—C5—C6—C7	0.8 (3)	C16—C17—C18—C19	179.12 (19)
N1—C6—C7—C8	179.3 (2)	C12—C11—C20—N4	179.5 (2)
C5—C6—C7—C8	−0.7 (3)	C12—C11—C20—C15	−0.9 (3)
C6—C7—C8—N2	0.3 (3)	C14—C15—C20—N4	−179.74 (18)
C6—C7—C8—C9	−179.5 (2)	C16—C15—C20—N4	0.5 (3)
C2—C1—C10—N2	−179.89 (19)	C14—C15—C20—C11	0.7 (3)
C2—C1—C10—C5	0.8 (3)	C16—C15—C20—C11	−179.10 (19)
C4—C5—C10—N2	179.44 (18)	C7—C8—N2—C10	0.1 (3)
C6—C5—C10—N2	−0.5 (3)	C9—C8—N2—C10	179.83 (18)
C4—C5—C10—C1	−1.3 (3)	C1—C10—N2—C8	−179.24 (19)
C6—C5—C10—C1	178.79 (19)	C5—C10—N2—C8	0.1 (3)
C20—C11—C12—C13	0.8 (3)	C17—C18—N4—C20	−0.5 (3)
C11—C12—C13—C14	−0.4 (3)	C19—C18—N4—C20	−179.86 (18)
C12—C13—C14—C15	0.1 (3)	C11—C20—N4—C18	179.95 (19)
C13—C14—C15—C20	−0.2 (3)	C15—C20—N4—C18	0.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1B···O7^{i}	0.89 (3)	2.16 (3)	2.981 (3)	154 (3)
N1—H1C···O7^{ii}	0.88 (3)	2.26 (3)	3.049 (3)	149 (3)
N1—H1C···O8^{ii}	0.88 (3)	2.58 (3)	3.374 (3)	151 (3)
O1—H1D···O9	0.88 (4)	1.61 (4)	2.485 (3)	177 (5)
N2—H2B···O11^{iii}	0.95 (3)	1.88 (3)	2.819 (3)	172 (3)
N3—H3B···O3^{iv}	0.90 (3)	2.03 (3)	2.902 (3)	163 (3)
N3—H3C···O3	0.87 (3)	2.54 (3)	3.177 (3)	132 (3)
N3—H3C···O4	0.87 (3)	2.20 (3)	3.034 (3)	162 (3)
N4—H4B···O12	0.90 (3)	1.87 (3)	2.753 (3)	169 (3)
O5—H5···O10	0.94 (4)	1.58 (4)	2.523 (3)	174 (5)
O9—H9D···O8	0.84 (4)	1.91 (4)	2.736 (3)	169 (4)
O9—H9E···O6^{v}	0.81 (4)	1.98 (4)	2.783 (3)	178 (5)
O10—H10A···O4	0.89 (4)	1.81 (4)	2.695 (3)	175 (3)
O10—H10B···O2^{vi}	0.83 (4)	1.93 (4)	2.745 (3)	169 (4)
O11—H11B···O2^{vi}	0.82 (4)	2.04 (4)	2.843 (3)	167 (5)

O11—H11C···O7	0.85 (4)	1.94 (4)	2.787 (3)	172 (3)
O12—H12B···O3^{vii}	0.86 (4)	1.98 (4)	2.823 (3)	166 (4)
O12—H12C···O11^{ii}	0.92 (4)	1.90 (4)	2.823 (3)	177 (4)

Symmetry codes: (i) −*x*−1, −*y*, −*z*+1; (ii) *x*−1, *y*+1, *z*; (iii) *x*, *y*+1, *z*; (iv) −*x*, −*y*+1, −*z*; (v) −*x*, −*y*, −*z*+1; (vi) −*x*, −*y*, −*z*; (vii) *x*−1, *y*, *z*.

Fig. 1

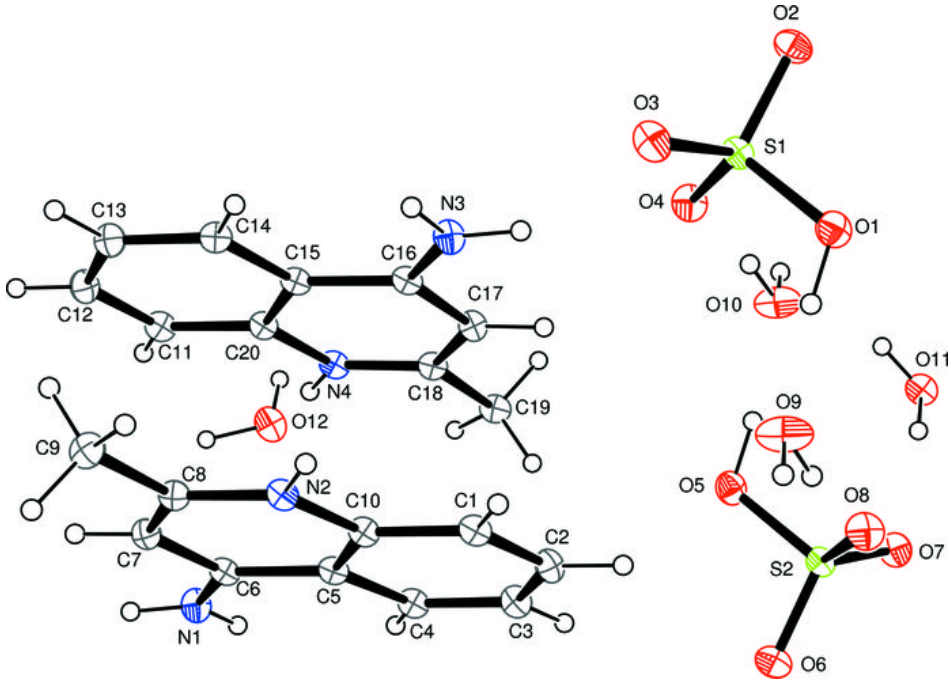
O2
O3
S1
O4
O1
O10
O11
O9
O5
O8
O7
S2
O6
C13
C14
N3
C15
C16
C17
C12
C11
C20
N4
C18
C19
O12
C9
C8
N2
C10
C1
C2
C7
C6
C5
C4
C3
N1

Fig. 2