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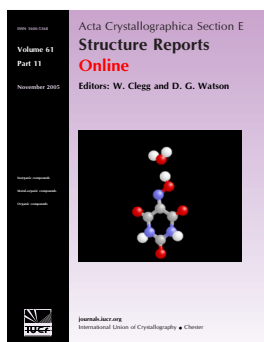
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Anilinium hydrogensulfate

Zina Boutobba, Amani Direm and Nourredine Benali-Cherif

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Anilinium hydrogensulfate

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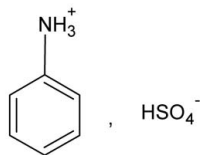
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.041; wR factor = 0.117; data-to-parameter ratio = 21.2.

The asymmetric unit of the title compound, $\text{C}_6\text{H}_8\text{N}^+\cdot\text{HSO}_4^-$, contains two cations and two anions which are linked to each other through $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, formed by all H atoms covalently bonded to the N atoms. In addition, strong $\text{O}-\text{H}\cdots\text{O}$ anion-anion hydrogen-bond interactions are also observed.

Related literature

For hydrogen bonding, see: Zimmerman & Corbin (2000); Brunsveld *et al.* (2001); Desiraju (2002); Desiraju & Steiner (1999); Steiner (2002); Etter *et al.* (1990); Bernstein *et al.* (1995). For related structures, see: Benali-Cherif, Boussekine *et al.* (2009); Messai *et al.* (2009); Benali-Cherif, Falek *et al.* (2009); Rademeyer (2004); Jayaraman *et al.* (2002); Smith *et al.* (2004); Paixão *et al.* (2000).



Experimental

Crystal data

 $\text{C}_6\text{H}_8\text{N}^+\cdot\text{HO}_4\text{S}^-$ $M_r = 191.20$ Orthorhombic, $Pca2_1$ $a = 14.3201$ (2) Å $b = 9.0891$ (3) Å $c = 12.8771$ (2) Å $V = 1676.04$ (7) Å³ $Z = 8$ Mo $K\alpha$ radiation $\mu = 0.36$ mm⁻¹ $T = 293$ K $0.2 \times 0.15 \times 0.1$ mm

Data collection

Nonius KappaCCD diffractometer

16963 measured reflections

4641 independent reflections

3108 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.049$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.041$ $wR(F^2) = 0.117$ $S = 1.02$

4641 reflections

219 parameters

1 restraint

H-atom parameters not refined

 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.47$ e Å⁻³

Absolute structure: Flack (1983),

2096 Friedel pairs

Flack parameter: 0.08 (9)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1A}-\text{H11}\cdots\text{O1A}^i$	0.89	1.95	2.821 (2)	167
$\text{N1A}-\text{H22}\cdots\text{O3B}$	0.89	1.95	2.817 (4)	163
$\text{N1A}-\text{H33}\cdots\text{O2B}^{iii}$	0.89	2.01	2.884 (3)	169
$\text{N1B}-\text{H1}\cdots\text{O1B}^{ii}$	0.89	1.94	2.828 (3)	175
$\text{N1B}-\text{H2}\cdots\text{O3A}^{ii}$	0.89	2.05	2.867 (3)	153
$\text{N1B}-\text{H3}\cdots\text{O1A}$	0.89	2.58	3.069 (3)	115
$\text{N1B}-\text{H3}\cdots\text{O2A}$	0.89	2.03	2.916 (3)	175
$\text{O4A}-\text{H4}\cdots\text{O3B}$	0.82	1.79	2.603 (4)	175
$\text{O4B}-\text{H44}\cdots\text{O3A}^i$	0.82	1.84	2.635 (4)	163

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

Data collection: *KappaCCD Server Software* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-32 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2534).

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supplementary materials

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Anilinium hydrogensulfate

Z. Boutobba, A. Direm and N. Benali-Cherif

Comment

The main purpose of this structural study was a determination of the arrangement of the cations and anions which are held together by two-dimensional hydrogen-bond networks.

Hydrogen bonding is one of the most versatile noncovalent forces in supramolecular chemistry and crystal engineering (Zimmerman & Corbin, 2000; Brunsveld *et al.*, 2001; Desiraju, 2002). Therefore, in the past decades assessment of discrete hydrogen bonding patterns had received great attention (Steiner, 2002; Desiraju & Steiner, 1999) because of its widespread occurrence in biological systems.

The aim of this paper is to discuss hydrogen patterns assuring the connection between anilinium and hydrogensulfate entities and to establish their different graph-set motifs (Bernstein *et al.*, 1995).

Bis(anilinium hydrogensulfate) is one of the hybrid compounds, rich in H-bonds (Benali-Cherif, Boussekine, *et al.*, 2009; Messai *et al.*, 2009; Benali-Cherif, Falek, *et al.*, 2009), which could have potential importance in constructing sophisticated assemblies from discrete ionic or molecular building blocks due to the strength and the directionality of hydrogen bonds (Steiner *et al.* 2002, Jayaraman *et al.*, 2002).

Recently, similar structures containing anilinium cations have been reported. Among examples, can be named the following ones: anilinium nitrate (Rademeyer, 2004), anilinium picrate (Smith *et al.*, 2004), anilinium hydrogenphosphite and anilinium hydrogenoxalate hemihydrate (Paixão *et al.*, 2000).

The structure of (I) may be described as formed by alternating sheets of cations and anions (Fig. 2) which are held together with four and five-centered N—H \cdots O H-bonds to form C₄⁴(10) infinite chains running through the *c* direction. Moreover, strong O—H \cdots O hydrogen bonds observed between bisulfate anions generate C₂²(8) chains in the *a* axis direction. The infinite chains resulting from anion-anion and anion-cation interactions can be described as zigzag layers parallel to the (*ac*) plane (Fig. 3). The crossing of these chains builds up different rings with R₃³(10) and R₅⁴(16) graph set motifs (Fig. 3) (Etter *et al.*, 1990; Bernstein *et al.*, 1995).

Experimental

Single crystals of the title compound are prepared by slow evaporation at room temperature of an aqueous solution of aniline and sulfuric acid.

Refinement

The title compound crystallizes in the centrosymmetric space group P *c* a 2₁. All non-H atoms were refined with anisotropic atomic displacement parameters. H atoms were located from Fourier difference maps and treated as riding with C—H =

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0.93 Å, N—H = 0.89 Å and O—H = 0.82 Å. Their isotropic displacement parameters were set equal to 1.2Ueq (C) and 1.5Ueq (N, O).

Figures

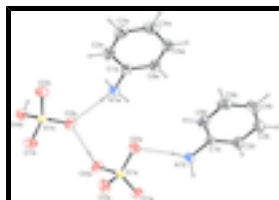


Fig. 1. ORTEP view of the asymmetric unit of (I) showing 10% probability displacement ellipsoids.

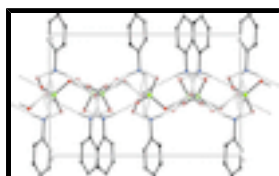


Fig. 2. Alternating cationic and anionic layers visualized through the (001) plane.

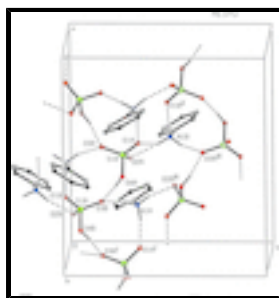


Fig. 3. Intermolecular hydrogen bonding patterns running parallel to (*bc*) plane. H atoms not involved in hydrogen bondings have been omitted for clarity. [Symmetry codes: (i) $x-1/2, -y+1, z$; (ii) $-x+1, -y+1, z+1/2$; (iii) $-x+1/2, y, z+1/2$.

Anilinium hydrogensulfate

Crystal data

$C_6H_8N^+ \cdot HO_4S^-$

$M_r = 191.20$

Orthorhombic, $Pca2_1$

Hall symbol: P 2c -2ac

$a = 14.3201$ (2) Å

$b = 9.0891$ (3) Å

$c = 12.8771$ (2) Å

$V = 1676.04$ (7) Å³

$Z = 8$

$F(000) = 800$

$D_x = 1.516$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 16963 reflections

$\theta = 2.7\text{--}30.0^\circ$

$\mu = 0.36$ mm⁻¹

$T = 293$ K

Prism, colourless

$0.2 \times 0.15 \times 0.1$ mm

Data collection

Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed tube

graphite

$\omega - \theta$ scans

3108 reflections with $I > 2\sigma(I)$

$R_{int} = 0.049$

$\theta_{max} = 30.0^\circ$, $\theta_{min} = 2.7^\circ$

$h = -19 \rightarrow 17$

16963 measured reflections $k = -9 \rightarrow 12$
 4641 independent reflections $l = -18 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters not refined
$wR(F^2) = 0.117$	$w = 1/[\sigma^2(F_o^2) + (0.0622P)^2 + 0.1354P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
4641 reflections	$(\Delta/\sigma)_{\max} = 0.001$
219 parameters	$\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\min} = -0.47 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 2096 Friedel pairs
	Flack parameter: 0.08 (9)

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. 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1A	0.21732 (15)	0.3085 (2)	0.3390 (2)	0.0432 (5)
H22	0.2392	0.3525	0.2823	0.065*
H33	0.2550	0.3273	0.3924	0.065*
H11	0.1603	0.3422	0.3530	0.065*
C1A	0.21307 (19)	0.1496 (3)	0.3217 (3)	0.0363 (7)
C2A	0.1721 (3)	0.0988 (4)	0.2311 (3)	0.0531 (8)
H2A	0.1482	0.1641	0.1823	0.064*
C3A	0.1676 (3)	-0.0512 (4)	0.2153 (3)	0.0626 (10)
H3A	0.1398	-0.0875	0.1553	0.075*
C4A	0.2039 (2)	-0.1480 (4)	0.2873 (4)	0.0640 (11)
H4A	0.2006	-0.2489	0.2760	0.077*
C5A	0.2449 (3)	-0.0940 (4)	0.3758 (3)	0.0555 (9)
H5A	0.2698	-0.1588	0.4243	0.067*
C6A	0.2493 (3)	0.0551 (4)	0.3931 (3)	0.0458 (7)
H6A	0.2770	0.0912	0.4533	0.055*

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N1B	0.52868 (13)	0.2975 (2)	0.5035 (2)	0.0403 (5)
H1	0.5861	0.3342	0.4989	0.061*
H2	0.4989	0.3397	0.5564	0.061*
H3	0.4978	0.3154	0.4448	0.061*
C1B	0.53385 (18)	0.1390 (3)	0.5207 (2)	0.0330 (6)
C2B	0.5767 (2)	0.0855 (4)	0.6081 (3)	0.0498 (8)
H2B	0.6015	0.1493	0.6573	0.060*
C3B	0.5824 (3)	-0.0655 (4)	0.6217 (4)	0.0622 (10)
H3B	0.6104	-0.1037	0.6810	0.075*
C4B	0.5467 (3)	-0.1590 (4)	0.5479 (4)	0.0622 (11)
H4B	0.5521	-0.2602	0.5568	0.075*
C5B	0.5030 (3)	-0.1046 (4)	0.4610 (4)	0.0599 (11)
H5B	0.4775	-0.1685	0.4122	0.072*
C6B	0.4973 (3)	0.0456 (4)	0.4468 (3)	0.0453 (7)
H6B	0.4688	0.0836	0.3877	0.054*
S1A	0.47642 (6)	0.50293 (6)	0.27815 (5)	0.0354 (3)
O1A	0.53917 (14)	0.5609 (3)	0.3548 (2)	0.0588 (5)
O2A	0.43955 (15)	0.3610 (2)	0.30490 (17)	0.0501 (5)
O3A	0.5129 (3)	0.5072 (3)	0.1721 (3)	0.0610 (11)
O4A	0.39386 (13)	0.6139 (2)	0.27837 (19)	0.0492 (5)
H4	0.3495	0.5784	0.2468	0.074*
S1B	0.22562 (6)	0.50860 (7)	0.06533 (5)	0.0340 (3)
O1B	0.29288 (13)	0.5696 (2)	-0.00469 (18)	0.0543 (5)
O2B	0.18378 (13)	0.3739 (2)	0.02858 (18)	0.0477 (5)
O3B	0.2609 (3)	0.4930 (2)	0.1701 (3)	0.0570 (10)
O4B	0.14771 (13)	0.6268 (2)	0.0699 (2)	0.0493 (5)
H44	0.1067	0.6000	0.1100	0.074*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1A	0.0488 (13)	0.0354 (12)	0.0452 (12)	0.0039 (9)	-0.0019 (9)	-0.0050 (10)
C1A	0.0319 (14)	0.0339 (15)	0.0431 (16)	0.0026 (11)	0.0016 (12)	-0.0062 (12)
C2A	0.0594 (19)	0.0502 (19)	0.0496 (18)	0.0050 (16)	-0.0107 (17)	-0.0079 (17)
C3A	0.069 (2)	0.055 (2)	0.063 (3)	-0.001 (2)	-0.0126 (19)	-0.023 (2)
C4A	0.0542 (19)	0.0386 (19)	0.099 (3)	-0.0010 (14)	0.007 (2)	-0.022 (2)
C5A	0.0558 (18)	0.0426 (17)	0.068 (3)	0.0087 (16)	-0.0002 (19)	0.0097 (17)
C6A	0.0451 (15)	0.045 (2)	0.0471 (18)	-0.0003 (18)	-0.0043 (14)	-0.0026 (16)
N1B	0.0429 (11)	0.0348 (12)	0.0433 (11)	-0.0024 (9)	0.0007 (9)	-0.0008 (10)
C1B	0.0320 (14)	0.0303 (14)	0.0366 (15)	-0.0033 (10)	0.0044 (11)	0.0006 (11)
C2B	0.0532 (18)	0.0478 (18)	0.0483 (17)	0.0025 (15)	-0.0088 (16)	0.0018 (15)
C3B	0.062 (2)	0.058 (3)	0.067 (2)	0.008 (2)	0.003 (2)	0.024 (2)
C4B	0.0561 (19)	0.0345 (18)	0.096 (3)	-0.0007 (15)	0.021 (2)	0.0099 (19)
C5B	0.0511 (18)	0.045 (2)	0.084 (3)	-0.0072 (17)	0.004 (2)	-0.0201 (19)
C6B	0.0410 (15)	0.0461 (19)	0.0487 (19)	-0.0017 (18)	-0.0040 (14)	-0.0100 (17)
S1A	0.0319 (6)	0.0352 (5)	0.0392 (6)	-0.0012 (2)	-0.0005 (5)	0.0038 (2)
O1A	0.0503 (12)	0.0554 (14)	0.0705 (14)	-0.0116 (11)	-0.0226 (10)	0.0051 (12)
O2A	0.0625 (13)	0.0365 (11)	0.0514 (12)	-0.0065 (10)	-0.0036 (10)	0.0067 (8)

O3A	0.058 (2)	0.075 (2)	0.050 (2)	0.0183 (11)	0.0188 (19)	0.0205 (10)
O4A	0.0391 (10)	0.0430 (11)	0.0655 (13)	0.0065 (8)	-0.0047 (9)	-0.0081 (10)
S1B	0.0305 (6)	0.0341 (5)	0.0374 (5)	-0.0020 (2)	0.0013 (4)	-0.0020 (3)
O1B	0.0427 (11)	0.0621 (16)	0.0580 (13)	-0.0112 (10)	0.0141 (9)	0.0022 (12)
O2B	0.0499 (11)	0.0368 (10)	0.0564 (12)	-0.0074 (9)	0.0048 (9)	-0.0099 (9)
O3B	0.0476 (18)	0.073 (2)	0.050 (2)	-0.0086 (10)	-0.0119 (18)	0.0084 (10)
O4B	0.0470 (11)	0.0379 (11)	0.0630 (12)	0.0054 (9)	0.0082 (10)	0.0013 (10)

Geometric parameters (Å, °)

N1A—C1A	1.462 (4)	C1B—C6B	1.378 (4)
N1A—H22	0.8900	C2B—C3B	1.386 (6)
N1A—H33	0.8900	C2B—H2B	0.9300
N1A—H11	0.8900	C3B—C4B	1.373 (6)
C1A—C6A	1.361 (5)	C3B—H3B	0.9300
C1A—C2A	1.385 (5)	C4B—C5B	1.374 (6)
C2A—C3A	1.380 (5)	C4B—H4B	0.9300
C2A—H2A	0.9300	C5B—C6B	1.380 (4)
C3A—C4A	1.380 (6)	C5B—H5B	0.9300
C3A—H3A	0.9300	C6B—H6B	0.9300
C4A—C5A	1.372 (6)	S1A—O1A	1.435 (2)
C4A—H4A	0.9300	S1A—O2A	1.436 (2)
C5A—C6A	1.375 (4)	S1A—O3A	1.463 (4)
C5A—H5A	0.9300	S1A—O4A	1.554 (2)
C6A—H6A	0.9300	O4A—H4	0.8201
N1B—C1B	1.460 (3)	S1B—O1B	1.431 (2)
N1B—H1	0.8900	S1B—O2B	1.4430 (19)
N1B—H2	0.8900	S1B—O3B	1.448 (4)
N1B—H3	0.8900	S1B—O4B	1.550 (2)
C1B—C2B	1.371 (4)	O4B—H44	0.8200
C1A—N1A—H22	109.5	C2B—C1B—N1B	119.8 (3)
C1A—N1A—H33	109.5	C6B—C1B—N1B	119.0 (3)
H22—N1A—H33	109.5	C1B—C2B—C3B	118.8 (3)
C1A—N1A—H11	109.5	C1B—C2B—H2B	120.6
H22—N1A—H11	109.5	C3B—C2B—H2B	120.6
H33—N1A—H11	109.5	C4B—C3B—C2B	120.2 (3)
C6A—C1A—C2A	121.4 (3)	C4B—C3B—H3B	119.9
C6A—C1A—N1A	120.3 (3)	C2B—C3B—H3B	119.9
C2A—C1A—N1A	118.4 (3)	C3B—C4B—C5B	120.7 (4)
C3A—C2A—C1A	118.2 (3)	C3B—C4B—H4B	119.7
C3A—C2A—H2A	120.9	C5B—C4B—H4B	119.7
C1A—C2A—H2A	120.9	C4B—C5B—C6B	119.4 (3)
C2A—C3A—C4A	120.9 (3)	C4B—C5B—H5B	120.3
C2A—C3A—H3A	119.6	C6B—C5B—H5B	120.3
C4A—C3A—H3A	119.6	C1B—C6B—C5B	119.7 (3)
C5A—C4A—C3A	119.4 (3)	C1B—C6B—H6B	120.2
C5A—C4A—H4A	120.3	C5B—C6B—H6B	120.2
C3A—C4A—H4A	120.3	O1A—S1A—O2A	113.28 (16)
C4A—C5A—C6A	120.5 (3)	O1A—S1A—O3A	114.1 (2)

supplementary materials

C4A—C5A—H5A	119.8	O2A—S1A—O3A	112.27 (15)
C6A—C5A—H5A	119.8	O1A—S1A—O4A	103.72 (14)
C1A—C6A—C5A	119.6 (3)	O2A—S1A—O4A	107.62 (12)
C1A—C6A—H6A	120.2	O3A—S1A—O4A	104.85 (16)
C5A—C6A—H6A	120.2	S1A—O4A—H4	109.5
C1B—N1B—H1	109.5	O1B—S1B—O2B	113.68 (16)
C1B—N1B—H2	109.5	O1B—S1B—O3B	113.0 (2)
H1—N1B—H2	109.5	O2B—S1B—O3B	111.55 (14)
C1B—N1B—H3	109.5	O1B—S1B—O4B	103.86 (13)
H1—N1B—H3	109.5	O2B—S1B—O4B	107.56 (12)
H2—N1B—H3	109.5	O3B—S1B—O4B	106.47 (17)
C2B—C1B—C6B	121.2 (3)	S1B—O4B—H44	109.5
C6A—C1A—C2A—C3A	0.9 (5)	C6B—C1B—C2B—C3B	-0.4 (4)
N1A—C1A—C2A—C3A	-179.6 (3)	N1B—C1B—C2B—C3B	-178.8 (3)
C1A—C2A—C3A—C4A	-0.7 (5)	C1B—C2B—C3B—C4B	0.9 (4)
C2A—C3A—C4A—C5A	0.0 (6)	C2B—C3B—C4B—C5B	-1.6 (5)
C3A—C4A—C5A—C6A	0.4 (6)	C3B—C4B—C5B—C6B	1.7 (6)
C2A—C1A—C6A—C5A	-0.4 (5)	C2B—C1B—C6B—C5B	0.5 (5)
N1A—C1A—C6A—C5A	-180.0 (3)	N1B—C1B—C6B—C5B	178.9 (3)
C4A—C5A—C6A—C1A	-0.2 (5)	C4B—C5B—C6B—C1B	-1.1 (5)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1A—H11 \cdots O1A ⁱ	0.89	1.95	2.821 (2)	167
N1B—H2 \cdots O3A ⁱⁱ	0.89	2.05	2.867 (3)	153
N1A—H33 \cdots O2B ⁱⁱⁱ	0.89	2.01	2.884 (3)	169
N1B—H3 \cdots O1A	0.89	2.58	3.069 (3)	115
N1B—H3 \cdots O2A	0.89	2.03	2.916 (3)	175
N1A—H22 \cdots O3B	0.89	1.95	2.817 (4)	163
O4A—H4 \cdots O3B	0.82	1.79	2.603 (4)	175
O4B—H44 \cdots O3A ⁱ	0.82	1.84	2.635 (4)	163
N1B—H1 \cdots O1B ⁱⁱ	0.89	1.94	2.828 (3)	175

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

Fig. 1

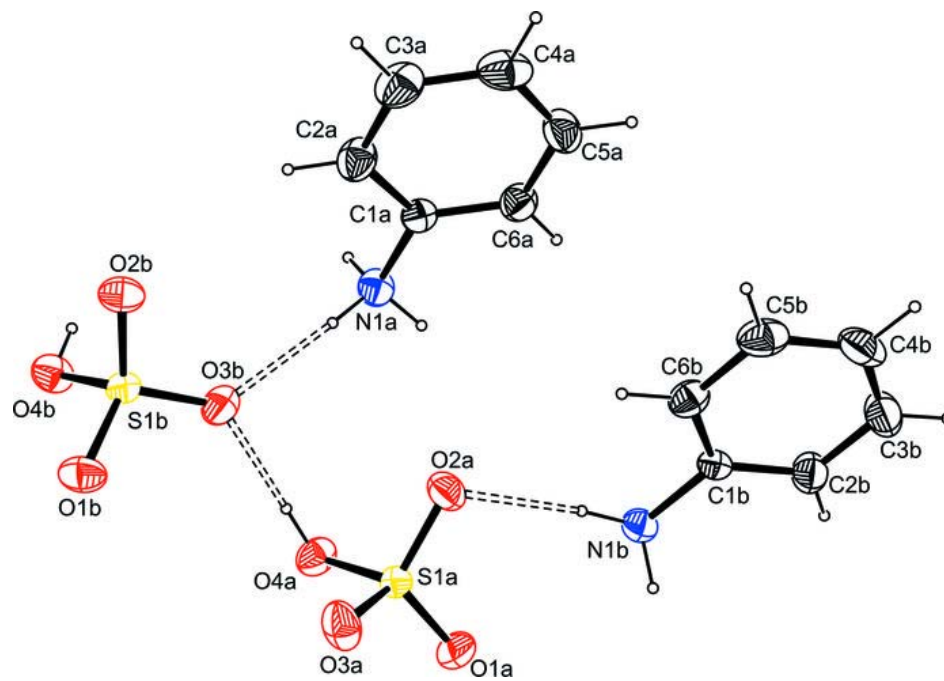


Fig. 2

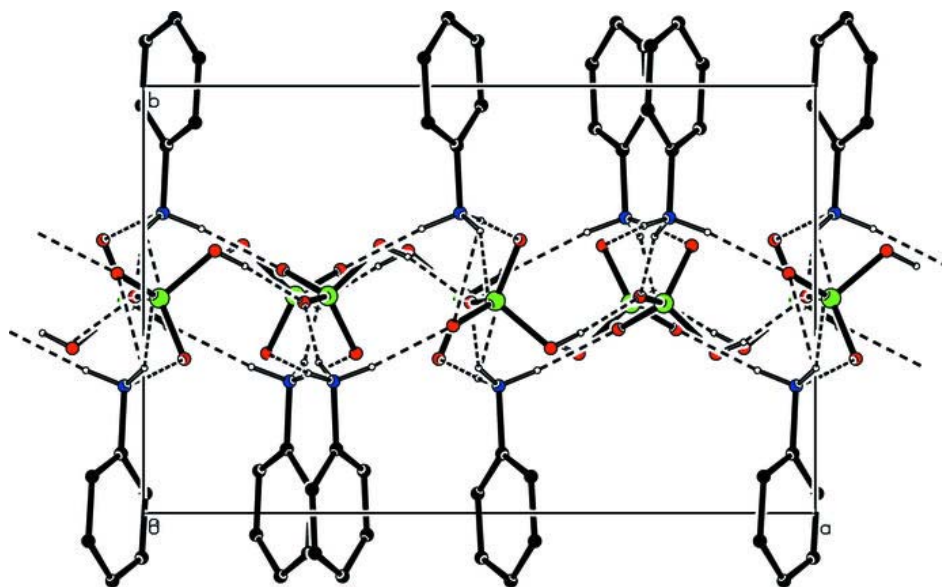


Fig. 3

