

Bis(2-amino-3-carboxypyrazin-1-ium) sulfate dihydrate

Fadila Berrah,^{a,b,*} Amira Ouakkaf,^a Sofiane Bouacida^{b,c} and Thierry Roisnel^d

^aLaboratoire de Chimie Appliquée et Technologie des Matériaux LCATM, Université Larbi Ben M'Hidi, 04000 Oum El Bouaghi, Algeria, ^bDépartement Sciences de la Matière, Faculté des Sciences Exactes et Sciences de la Nature et de la Vie, Université Larbi Ben M'hidi, 04000 Oum El Bouaghi, Algeria, ^cUnité de Recherche de Chimie de l'Environnement et Moléculaire Structurale, CHEMS, Faculté des Sciences Exactes, Université Mentouri Constantine 25000, Algeria, and ^dCentre de Difractométrie X, UMR 6226 CNRS Unité Sciences Chimiques de Rennes, Université de Rennes I, 263 Avenue du Général Leclerc, 35042 Rennes, France
Correspondence e-mail: fadilaber@yahoo.fr

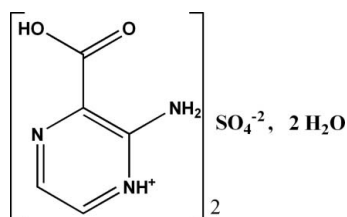
Received 7 February 2011; accepted 16 February 2011

Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.035; wR factor = 0.096; data-to-parameter ratio = 14.9.

The crystal structure of the title compound, $2\text{C}_5\text{H}_6\text{N}_3\text{O}_2^{+}\cdot\text{SO}_4^{2-}\cdot 2\text{H}_2\text{O}$, displays a variety of $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds in which all potential donors and acceptors are involved. In the crystal, cations and anions are interconnected, forming $R_3^3(10)$ and $R_2^2(8)$ ring motifs whereas the anions and water molecules form $R_3^3(10)$ rings, which develop in chains running along [100]. The resulting three-dimensional network exhibits undulating sheets parallel to (011), marked by the presence of $R_6^6(26)$ rings in which six cations are involved.

Related literature

For related compounds, see: Berrah *et al.* (2005*a,b*, 2011); Bouacida *et al.* (2005, 2009); Dobson & Gerkin (1996). For hydrogen-bond motifs, see: Bernstein *et al.* (1995); Etter *et al.* (1990). For similar intermolecular interactions, see: Dorn *et al.* (2005), Janiak (2000); Desiraju (2003).



Experimental

Crystal data

$2\text{C}_5\text{H}_6\text{N}_3\text{O}_2^{+}\cdot\text{SO}_4^{2-}\cdot 2\text{H}_2\text{O}$
 $M_r = 412.36$
Monoclinic, $P2_1/a$
 $a = 7.7214$ (4) Å
 $b = 20.7043$ (14) Å
 $c = 10.6398$ (7) Å
 $\beta = 109.299$ (2)°

$V = 1605.36$ (17) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.27$ mm⁻¹
 $T = 150$ K
 $0.55 \times 0.36 \times 0.15$ mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2002)
 $T_{\min} = 0.708$, $T_{\max} = 0.960$

13466 measured reflections
3675 independent reflections
3146 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.096$
 $S = 1.03$
3675 reflections

246 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.47$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.48$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1A}-\text{H1A1}\cdots\text{O4}$	0.88	1.92	2.7970 (18)	175
$\text{N1A}-\text{H1A2}\cdots\text{O6A}$	0.88	2.04	2.6741 (18)	128
$\text{N1A}-\text{H1A2}\cdots\text{O6B}^i$	0.88	2.30	3.0158 (18)	138
$\text{N2A}-\text{H2A}\cdots\text{O1}$	0.88	1.83	2.6915 (18)	167
$\text{N1B}-\text{H1B1}\cdots\text{O2}$	0.88	2.34	3.0827 (18)	142
$\text{N1B}-\text{H1B2}\cdots\text{O6B}$	0.88	2.08	2.7144 (19)	129
$\text{N1B}-\text{H1B2}\cdots\text{O6A}^{ii}$	0.88	2.10	2.8237 (18)	139
$\text{N2B}-\text{H2B}\cdots\text{O1W}$	0.88	1.81	2.6705 (18)	167
$\text{O5B}-\text{H5B}\cdots\text{O2W}$	0.84	1.67	2.5046 (17)	174
$\text{O5A}-\text{H5A}\cdots\text{O2}^i$	0.84	1.78	2.6192 (16)	175
$\text{O1W}-\text{H1W}\cdots\text{O3}$	0.85	1.95	2.7934 (19)	175
$\text{O1W}-\text{H2W}\cdots\text{O2}^{iii}$	0.85	2.06	2.8996 (18)	167
$\text{O1W}-\text{H2W}\cdots\text{O4}^{iii}$	0.85	2.65	3.2856 (17)	133
$\text{O2W}-\text{H3W}\cdots\text{O4}^{ii}$	0.85	1.88	2.7351 (17)	177
$\text{O2W}-\text{H4W}\cdots\text{O3}^{iv}$	0.86	1.91	2.7633 (17)	171

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

Data collection: *APEX2* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg & Berndt, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We are grateful to the LCATM laboratory, Université Larbi Ben M'Hidi, Oum El Bouaghi, Algeria, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2656).

References

- Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Berrah, F., Benali-Cherif, N. & Lamraoui, H. (2005*b*). *Acta Cryst.* **E61**, o1517–o1519.
Berrah, F., Lamraoui, H. & Benali-Cherif, N. (2005*a*). *Acta Cryst.* **E61**, o210–o212.

- Berrah, F., Ouakkaf, A., Bouacida, S. & Roisnel, T. (2011). *Acta Cryst.* **E67**, o525–o526.
- Bouacida, S., Belhouas, R., Kechout, H., Merazig, H. & Bénard-Rocherullé, P. (2009). *Acta Cryst.* **E65**, o628–o629.
- Bouacida, S., Merazig, H., Beghidja, A. & Beghidja, C. (2005). *Acta Cryst.* **E61**, m1153–m1155.
- Brandenburg, K. & Berndt, M. (2001). *DIAMOND*. Crystal Impact, Bonn, Germany.
- Bruker (2001). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Burla, M. C., Camalli, M., Carrozzini, B., Cascarano, G. L., Giacovazzo, C., Polidori, G. & Spagna, R. (2003). *J. Appl. Cryst.* **36**, 1103.
- Desiraju, G. R. (2003). Editor. *Crystal Design: Structure and Function*. In *Perspectives in Supramolecular Chemistry*, Vol. 7. Chichester: John Wiley & Sons, Ltd.
- Dobson, A. J. & Gerkin, R. E. (1996). *Acta Cryst.* **C52**, 1512–1514.
- Dorn, T., Janiak, C. & Abu-Shandi, K. (2005). *CrystEngComm*, **7**, 633–641.
- Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Janiak, C. (2000). *J. Chem. Soc. Dalton Trans.* pp. 3885–3896.
- Sheldrick, G. M. (2002). *SADABS* Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2011). E67, o677-o678 [doi:10.1107/S1600536811005824]

Bis(2-amino-3-carboxypyrazin-1-ium) sulfate dihydrate

F. Berrah, A. Ouakkaf, S. Bouacida and T. Roisnel

Comment

Hydrogen bonds are object of several studies, that aim at elucidate their influence on crystal construction and compounds properties (Desiraju, 2003). N-heterocyclic compounds such as pyrazine and its derivatives may be interesting units to built new edifices involving original hydrogen-bonding scheme since they include a variety of potential hydrogen donors and acceptors. In this perspective and as a part of our search for new hybrid compounds based on protonated amines and imines (Berrah *et al.* 2011, 2005*a,b*; Bouacida *et al.* 2005,2009), we present here the structure of Bis (2-Amino-3-carboxypyrazin-1-ium) sulfate dihydrate.

The asymmetric units of (I) includes two symmetry- independent cations and water molecules, and one sulfate anion. Cations and anions are interconnected to form $R_3^3(10)$ and $R_2^2(8)$ ring motifs (Etter *et al.*, 1990; Bernstein *et al.*, 1995) (Fig 1). Bond lengths and angles are as expected (Berrah *et al.* 2011; Dobson & Gerkin, 1996).

The three-dimensional structure of (I), results from undulating sheets of cations dimmers parallel to (011) plane(Fig.2 and Fig.3)and sulfate-water chains extending along [100](Fig.3). An interesting hydrogen bonds network, in which all potential donors and acceptors are involved, and especially marked by the presence of $R_6^6(26)$ and $R_2^3(10)$ set-graph motifs (Etter *et al.*, 1990; Bernstein *et al.*, 1995), ensures the coherence of the structure(Fig.2 and Fig.3, table 1). This later is reinforced by the contribution of π - π , S—O \cdots π and C—O \cdots π interactions (Dorn *et al.* 2005; Janiak, 2000) (table 2,3).

Experimental

The title compound was synthesized by reacting 3-amino-pyrazine 2- carboxylic acid with some excess of sulphuric acid in aqueous solution. Slow evaporation leads to well crystallized yellow needles.

Refinement

All non-H atoms were refined with anisotropic atomic displacement parameters. H atoms of water molecule were located in difference Fourier maps and treated as riding on their parent oxygen atoms with O—H = 0.85, H \cdots H = 1.40 and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. The remaining H atoms were localized on Fourier maps but introduced in calculated positions and treated as riding on their parent atoms (C, N or O) with C—H = 0.95 Å, O—H = 0.84 Å and N—H = 0.88 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C or N})$ and $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$.

Figures

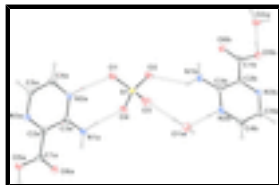


Fig. 1. The asymmetric unit of the title compound with the atomic labelling scheme. Displacements are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines.

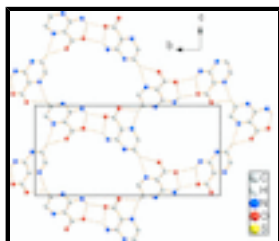


Fig. 2. Partial packing view showing undulating sheets parallel to (011) plane and $R_6^6(26)$ rings set motif. Hydrogen bonds are shown as dashed lines. Hydrogen atoms not involved in H-bonds have been omitted for clarity.

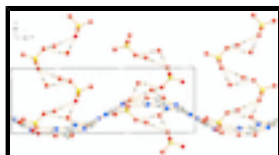
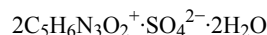


Fig. 3. Partial packing view showing sulfate-water chains extending along [100] direction and undulating sheets. Hydrogen bonds are shown as dashed lines. Hydrogen atoms not involved in H-bonds have been omitted for clarity.

Bis(2-amino-3-carboxypyrazin-1-ium) sulfate dihydrate

Crystal data



$M_r = 412.36$

Monoclinic, $P2_1/a$

$a = 7.7214$ (4) Å

$b = 20.7043$ (14) Å

$c = 10.6398$ (7) Å

$\beta = 109.299$ (2)°

$V = 1605.36$ (17) Å³

$Z = 4$

$F(000) = 856$

$D_x = 1.706$ Mg m⁻³

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

Cell parameters from 5179 reflections

$\theta = 2.8\text{--}27.5^\circ$

$\mu = 0.27$ mm⁻¹

$T = 150$ K

Prism, yellow

$0.55 \times 0.36 \times 0.15$ mm

Data collection

Bruker APEXII
diffractometer

graphite

CCD rotation images, thin slices scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2002)

$T_{\min} = 0.708$, $T_{\max} = 0.960$

13466 measured reflections

3675 independent reflections

3146 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 27.6^\circ$, $\theta_{\min} = 2.3^\circ$

$h = -8 \rightarrow 9$

$k = -26 \rightarrow 26$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.096$	H-atom parameters constrained
$S = 1.03$	$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 0.7394P]$
3675 reflections	where $P = (F_o^2 + 2F_c^2)/3$
246 parameters	$(\Delta/\sigma)_{\max} = 0.001$
0 restraints	$\Delta\rho_{\max} = 0.47 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.48 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
O5A	0.17659 (16)	0.04617 (6)	0.03501 (11)	0.0221 (3)
H5A	0.1515	0.0732	-0.0273	0.033*
O6A	0.03846 (18)	0.11842 (6)	0.12557 (11)	0.0258 (3)
N1A	0.0567 (2)	0.10587 (7)	0.37979 (13)	0.0231 (3)
H1A1	0.0399	0.1167	0.4549	0.028*
H1A2	0.0122	0.1303	0.3087	0.028*
N2A	0.21592 (19)	0.01604 (6)	0.48490 (13)	0.0195 (3)
H2A	0.1989	0.0288	0.5588	0.023*
N3A	0.27826 (19)	-0.02410 (6)	0.25952 (13)	0.0194 (3)
C1A	0.1266 (2)	0.06905 (7)	0.13275 (15)	0.0185 (3)
C2A	0.1875 (2)	0.02980 (7)	0.25829 (15)	0.0177 (3)
C3A	0.1485 (2)	0.05305 (7)	0.37379 (15)	0.0183 (3)
C4A	0.3079 (2)	-0.03946 (8)	0.48622 (16)	0.0207 (3)
H4A	0.3518	-0.0644	0.5654	0.025*
C5A	0.3374 (2)	-0.05941 (8)	0.37264 (16)	0.0214 (3)
H5C	0.4006	-0.0988	0.3731	0.026*
O5B	-0.03794 (19)	0.32977 (6)	1.37046 (12)	0.0273 (3)
H5B	-0.0768	0.3055	1.4185	0.041*

supplementary materials

O6B	-0.03786 (17)	0.23824 (5)	1.25812 (11)	0.0241 (3)
N1B	0.0619 (2)	0.24559 (7)	1.03668 (14)	0.0238 (3)
H1B1	0.0821	0.2313	0.9649	0.029*
H1B2	0.0315	0.2184	1.0895	0.029*
N2B	0.12393 (19)	0.34886 (7)	0.98346 (13)	0.0205 (3)
H2B	0.1422	0.3332	0.912	0.025*
N3B	0.07073 (19)	0.39911 (6)	1.20349 (13)	0.0206 (3)
C1B	-0.0126 (2)	0.29617 (8)	1.27448 (15)	0.0196 (3)
C2B	0.0482 (2)	0.33663 (7)	1.17934 (14)	0.0178 (3)
C3B	0.0773 (2)	0.30786 (8)	1.06522 (15)	0.0187 (3)
C4B	0.1438 (2)	0.41269 (8)	1.00633 (16)	0.0227 (3)
H4B	0.1752	0.4403	0.9458	0.027*
C5B	0.1180 (2)	0.43737 (8)	1.11810 (16)	0.0233 (3)
H5D	0.1338	0.4824	1.1357	0.028*
S1	0.15317 (5)	0.131125 (18)	0.73117 (4)	0.01813 (11)
O1	0.2146 (2)	0.06509 (6)	0.71883 (12)	0.0337 (3)
O2	0.09536 (18)	0.13565 (6)	0.85065 (12)	0.0262 (3)
O3	0.30149 (17)	0.17773 (7)	0.74478 (12)	0.0306 (3)
O4	-0.00482 (16)	0.14708 (6)	0.61180 (11)	0.0236 (3)
O1W	0.23309 (17)	0.30737 (6)	0.78406 (12)	0.0262 (3)
H1W	0.2465	0.2676	0.7697	0.039*
H2W	0.3352	0.3253	0.7903	0.039*
O2W	-0.15818 (19)	0.26453 (6)	1.52193 (12)	0.0296 (3)
H3W	-0.1068	0.2285	1.5511	0.044*
H4W	-0.1653	0.2863	1.5886	0.044*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O5A	0.0332 (7)	0.0213 (6)	0.0140 (5)	0.0044 (5)	0.0109 (5)	0.0030 (4)
O6A	0.0400 (7)	0.0206 (6)	0.0184 (6)	0.0076 (5)	0.0115 (5)	0.0036 (4)
N1A	0.0340 (8)	0.0227 (7)	0.0140 (6)	0.0063 (6)	0.0100 (6)	0.0012 (5)
N2A	0.0269 (7)	0.0193 (6)	0.0128 (6)	-0.0002 (5)	0.0072 (5)	0.0001 (5)
N3A	0.0253 (7)	0.0168 (6)	0.0158 (6)	-0.0013 (5)	0.0063 (5)	-0.0003 (5)
C1A	0.0232 (8)	0.0172 (7)	0.0153 (7)	-0.0026 (6)	0.0065 (6)	-0.0007 (6)
C2A	0.0225 (8)	0.0159 (7)	0.0154 (7)	-0.0020 (6)	0.0072 (6)	-0.0008 (6)
C3A	0.0229 (8)	0.0175 (7)	0.0144 (7)	-0.0027 (6)	0.0062 (6)	-0.0007 (6)
C4A	0.0259 (8)	0.0177 (7)	0.0168 (7)	-0.0016 (6)	0.0048 (6)	0.0035 (6)
C5A	0.0279 (9)	0.0163 (7)	0.0186 (7)	0.0011 (6)	0.0056 (6)	0.0000 (6)
O5B	0.0480 (8)	0.0198 (6)	0.0201 (6)	-0.0009 (5)	0.0194 (5)	-0.0004 (5)
O6B	0.0355 (7)	0.0185 (6)	0.0193 (6)	-0.0015 (5)	0.0104 (5)	0.0003 (4)
N1B	0.0372 (8)	0.0188 (7)	0.0167 (6)	-0.0035 (6)	0.0105 (6)	-0.0037 (5)
N2B	0.0267 (7)	0.0221 (7)	0.0128 (6)	-0.0027 (5)	0.0069 (5)	-0.0024 (5)
N3B	0.0267 (7)	0.0172 (6)	0.0167 (6)	0.0005 (5)	0.0056 (5)	-0.0008 (5)
C1B	0.0231 (8)	0.0198 (8)	0.0141 (7)	0.0019 (6)	0.0039 (6)	0.0008 (6)
C2B	0.0216 (8)	0.0173 (7)	0.0127 (7)	0.0005 (6)	0.0033 (6)	0.0001 (5)
C3B	0.0203 (8)	0.0194 (7)	0.0142 (7)	-0.0010 (6)	0.0029 (6)	-0.0014 (6)
C4B	0.0276 (9)	0.0201 (8)	0.0195 (7)	-0.0035 (6)	0.0068 (6)	0.0020 (6)

C5B	0.0324 (9)	0.0177 (7)	0.0193 (7)	-0.0019 (6)	0.0077 (7)	0.0004 (6)
S1	0.0259 (2)	0.01758 (19)	0.01276 (18)	0.00290 (14)	0.00883 (15)	0.00224 (13)
O1	0.0605 (9)	0.0245 (6)	0.0186 (6)	0.0188 (6)	0.0163 (6)	0.0047 (5)
O2	0.0429 (7)	0.0230 (6)	0.0193 (6)	0.0051 (5)	0.0192 (5)	0.0035 (5)
O3	0.0291 (7)	0.0384 (7)	0.0238 (6)	-0.0067 (5)	0.0080 (5)	0.0029 (5)
O4	0.0266 (6)	0.0246 (6)	0.0179 (6)	0.0031 (5)	0.0049 (5)	0.0017 (4)
O1W	0.0294 (6)	0.0273 (6)	0.0252 (6)	-0.0009 (5)	0.0136 (5)	-0.0005 (5)
O2W	0.0460 (8)	0.0244 (6)	0.0229 (6)	0.0086 (5)	0.0174 (6)	0.0061 (5)

Geometric parameters (Å, °)

O5A—C1A	1.3116 (19)	N1B—H1B1	0.88
O5A—H5A	0.84	N1B—H1B2	0.88
O6A—C1A	1.216 (2)	N2B—C4B	1.343 (2)
N1A—C3A	1.316 (2)	N2B—C3B	1.347 (2)
N1A—H1A1	0.88	N2B—H2B	0.88
N1A—H1A2	0.88	N3B—C2B	1.319 (2)
N2A—C4A	1.349 (2)	N3B—C5B	1.344 (2)
N2A—C3A	1.360 (2)	C1B—C2B	1.503 (2)
N2A—H2A	0.88	C2B—C3B	1.435 (2)
N3A—C2A	1.315 (2)	C4B—C5B	1.368 (2)
N3A—C5A	1.352 (2)	C4B—H4B	0.95
C1A—C2A	1.500 (2)	C5B—H5D	0.95
C2A—C3A	1.442 (2)	S1—O1	1.4670 (12)
C4A—C5A	1.365 (2)	S1—O3	1.4677 (13)
C4A—H4A	0.95	S1—O4	1.4786 (12)
C5A—H5C	0.95	S1—O2	1.4831 (12)
O5B—C1B	1.3028 (19)	O1W—H1W	0.8491
O5B—H5B	0.84	O1W—H2W	0.8542
O6B—C1B	1.218 (2)	O2W—H3W	0.8543
N1B—C3B	1.321 (2)	O2W—H4W	0.8582
C1A—O5A—H5A	109.5	C4B—N2B—C3B	122.81 (14)
C3A—N1A—H1A1	120	C4B—N2B—H2B	118.6
C3A—N1A—H1A2	120	C3B—N2B—H2B	118.6
H1A1—N1A—H1A2	120	C2B—N3B—C5B	119.63 (14)
C4A—N2A—C3A	122.57 (14)	O6B—C1B—O5B	125.38 (15)
C4A—N2A—H2A	118.7	O6B—C1B—C2B	121.55 (14)
C3A—N2A—H2A	118.7	O5B—C1B—C2B	113.05 (14)
C2A—N3A—C5A	119.32 (14)	N3B—C2B—C3B	121.62 (14)
O6A—C1A—O5A	123.99 (14)	N3B—C2B—C1B	117.76 (14)
O6A—C1A—C2A	121.03 (14)	C3B—C2B—C1B	120.62 (14)
O5A—C1A—C2A	114.98 (13)	N1B—C3B—N2B	119.31 (15)
N3A—C2A—C3A	122.39 (14)	N1B—C3B—C2B	124.91 (15)
N3A—C2A—C1A	118.54 (14)	N2B—C3B—C2B	115.78 (14)
C3A—C2A—C1A	119.06 (14)	N2B—C4B—C5B	118.98 (15)
N1A—C3A—N2A	118.94 (14)	N2B—C4B—H4B	120.5
N1A—C3A—C2A	125.91 (14)	C5B—C4B—H4B	120.5
N2A—C3A—C2A	115.15 (14)	N3B—C5B—C4B	121.17 (15)
N2A—C4A—C5A	119.33 (14)	N3B—C5B—H5D	119.4

supplementary materials

N2A—C4A—H4A	120.3	C4B—C5B—H5D	119.4
C5A—C4A—H4A	120.3	O1—S1—O3	110.91 (9)
N3A—C5A—C4A	121.21 (15)	O1—S1—O4	109.31 (7)
N3A—C5A—H5C	119.4	O3—S1—O4	109.46 (7)
C4A—C5A—H5C	119.4	O1—S1—O2	109.47 (7)
C1B—O5B—H5B	109.5	O3—S1—O2	108.66 (7)
C3B—N1B—H1B1	120	O4—S1—O2	109.00 (7)
C3B—N1B—H1B2	120	H1W—O1W—H2W	105.7
H1B1—N1B—H1B2	120	H3W—O2W—H4W	108
C5A—N3A—C2A—C3A	0.1 (2)	C5B—N3B—C2B—C3B	1.6 (2)
C5A—N3A—C2A—C1A	178.70 (14)	C5B—N3B—C2B—C1B	-177.43 (14)
O6A—C1A—C2A—N3A	178.18 (15)	O6B—C1B—C2B—N3B	179.35 (15)
O5A—C1A—C2A—N3A	-2.2 (2)	O5B—C1B—C2B—N3B	0.9 (2)
O6A—C1A—C2A—C3A	-3.2 (2)	O6B—C1B—C2B—C3B	0.3 (2)
O5A—C1A—C2A—C3A	176.41 (14)	O5B—C1B—C2B—C3B	-178.12 (14)
C4A—N2A—C3A—N1A	178.56 (15)	C4B—N2B—C3B—N1B	-179.42 (15)
C4A—N2A—C3A—C2A	-1.9 (2)	C4B—N2B—C3B—C2B	0.5 (2)
N3A—C2A—C3A—N1A	-179.04 (16)	N3B—C2B—C3B—N1B	178.17 (15)
C1A—C2A—C3A—N1A	2.4 (2)	C1B—C2B—C3B—N1B	-2.9 (2)
N3A—C2A—C3A—N2A	1.5 (2)	N3B—C2B—C3B—N2B	-1.7 (2)
C1A—C2A—C3A—N2A	-177.09 (13)	C1B—C2B—C3B—N2B	177.27 (14)
C3A—N2A—C4A—C5A	0.8 (2)	C3B—N2B—C4B—C5B	0.8 (2)
C2A—N3A—C5A—C4A	-1.4 (2)	C2B—N3B—C5B—C4B	-0.2 (3)
N2A—C4A—C5A—N3A	0.9 (2)	N2B—C4B—C5B—N3B	-1.0 (3)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1A—H1A1 \cdots O4	0.88	1.92	2.7970 (18)	175.
N1A—H1A2 \cdots O6A	0.88	2.04	2.6741 (18)	128.
N1A—H1A2 \cdots O6B ⁱ	0.88	2.30	3.0158 (18)	138.
N2A—H2A \cdots O1	0.88	1.83	2.6915 (18)	167.
N1B—H1B1 \cdots O2	0.88	2.34	3.0827 (18)	142.
N1B—H1B2 \cdots O6B	0.88	2.08	2.7144 (19)	129.
N1B—H1B2 \cdots O6A ⁱⁱ	0.88	2.10	2.8237 (18)	139.
N2B—H2B \cdots O1W	0.88	1.81	2.6705 (18)	167.
O5B—H5B \cdots O2W	0.84	1.67	2.5046 (17)	174.
O5A—H5A \cdots O2 ⁱ	0.84	1.78	2.6192 (16)	175.
O1W—H1W \cdots O3	0.85	1.95	2.7934 (19)	175.
O1W—H2W \cdots O2 ⁱⁱⁱ	0.85	2.06	2.8996 (18)	167
O1W—H2W \cdots O4 ⁱⁱⁱ	0.85	2.65	3.2856 (17)	133.
O2W—H3W \cdots O4 ⁱⁱ	0.85	1.88	2.7351 (17)	177.
O2W—H4W \cdots O3 ^{iv}	0.86	1.91	2.7633 (17)	171.
C4A—H4A \cdots O5B ^v	0.95	2.58	3.320 (2)	134.
C4A—H4A \cdots N3B ^v	0.95	2.45	3.369 (2)	163.
C4B—H4B \cdots O5A ^{vi}	0.95	2.45	3.187 (2)	134.
C4B—H4B \cdots N3A ^{vi}	0.95	2.44	3.347 (2)	159

C5A—H5C···O1W ^{vii}	0.95	2.55	3.175 (2)	124.
C5B—H5D···O1 ^{viii}	0.95	2.35	3.192 (2)	148.

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

Table 2

π - π stacking interactions (\AA , $^\circ$)

Cg1 is the centroid of the N2A–C4A ring.

CgI	CgJ	CgI···CgJ ^a	α	β	γ	CgI···P(J) ^b	CgJ···P(I) ^c	Slippage
Cg1	Cg1 ⁱ	3.9678 (9)	0	34.94	34.94	3.2528 (6)	3.2527 (6)	2.272

Symmetry codes: (i) $1-x, -y, 1-z$ Notes: a : Distance between centroids b : Perpendicular distance of CgI on ring plan J c : Perpendicular distance of CgJ on ring plan I α = Dihedral Angle between the ring planes β = Angle between the centroid vector CgI···CgJ and the normal to the plane I. γ = Angle between the centroid vector CgI···CgJ and the normal to the plane J. Slippage = vertical displacement between ring centroids.

Table 3

$S-O\cdots\pi$ and $C-O\cdots\pi$ interactions (\AA , $^\circ$).

Cg1 and Cg2 are the centroids of the N2A–C4A and N2B–C4B rings, respectively.

X	I	J	I···J	X–I···J	X···J
S1	O1	Cg1 ⁱ	3.5922 (17)	91.83 (7)	3.9233 (8)
S1	O2	Cg1 ⁱ	3.9845 (14)	76.88 (5)	3.9233 (8)
S1	O2	Cg2 ⁱⁱ	3.8831 (15)	92.77 (6)	4.2231 (8)
C1A	O6A	Cg2 ⁱⁱⁱ	3.3136 (16)	125.62 (11)	4.1418 (18)

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

Fig. 1

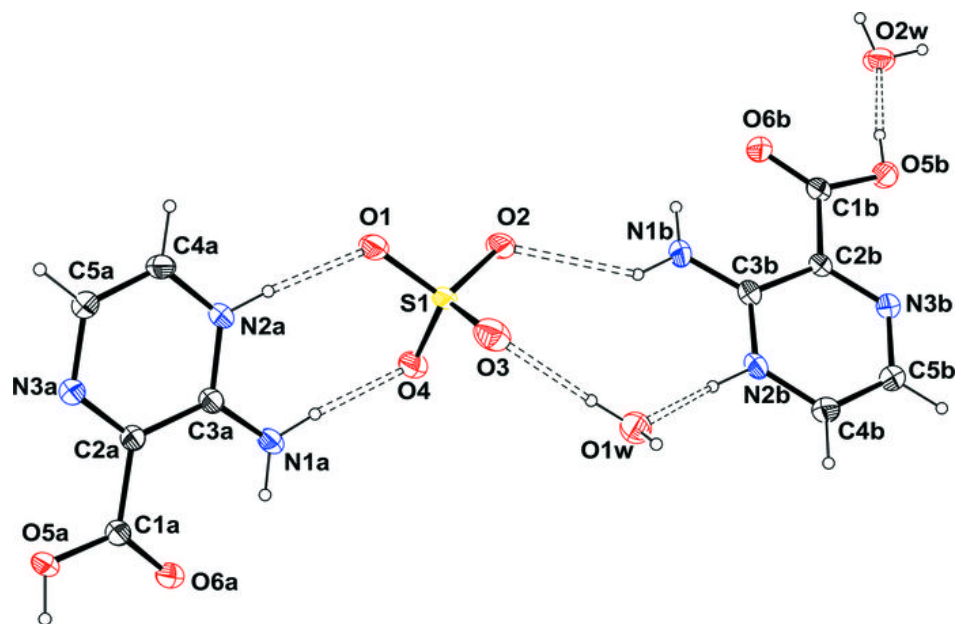


Fig. 2

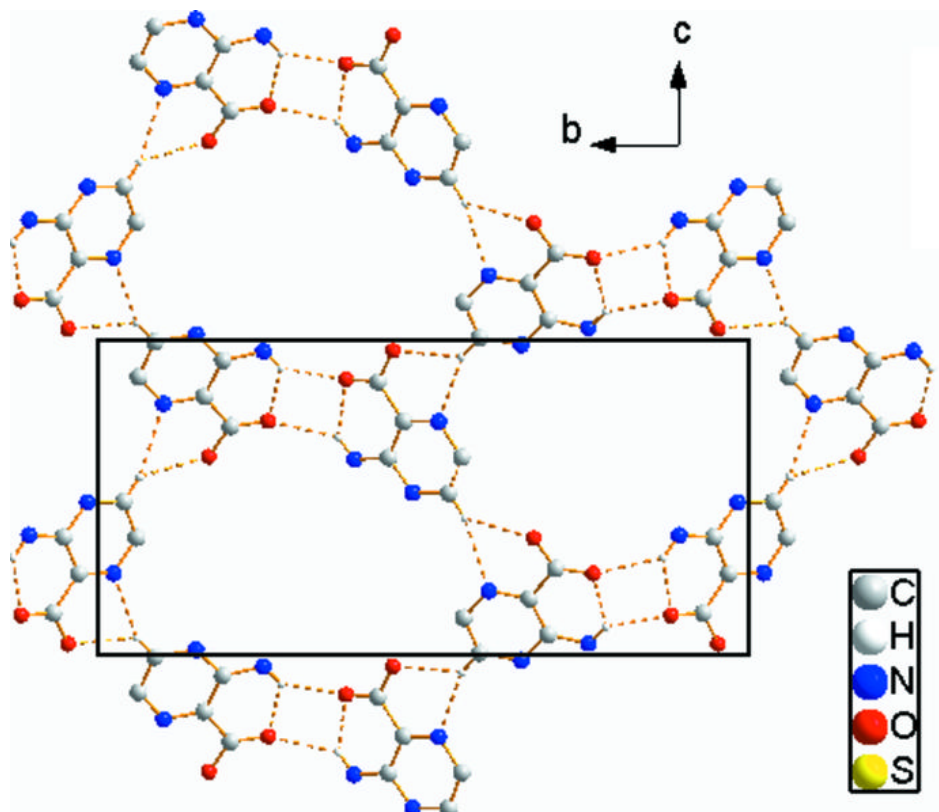


Fig. 3

