# Crystal structure of 4,4′-(disulfanediyl)dibutanoic acid–4,4′-bipyridine (1/1)

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4,4′-(Disulfanediyl)dibutanoic acid (dtba) and 4,4′-bipyridine (4,4′-bpy) crystallize in an 1:1 ratio, leading to the title co-crystal with composition $C_8H_{14}O_4S_2 \cdot C_{10}H_8N_2$. A distinctive feature of the crystal structure is the geometry of the dtba moiety, which appears to be stretched [with a 9.98 (1) Å span between outermost carbons] and acts as an hydrogen-bonding connector, forming linear chains along [$\bar{2}$11] with the 4,4′-bpy moiety by way of O—H···N hydrogen bonds and C—H···O interactions. The influence of the molecular shape on the hydrogen-bonding pattern is analysed by comparing the title compound and two other 4,4′-bpy co-crystals with closely related molecules of similar formulation but different geometry, showing the way in which this correlates with the packing arrangement.

1. Chemical context

The object of the present study, the 4,4′-(disulfanediyl)dibutanoic acid molecule $C_8H_{12}O_4S_2$ (dtba), consists of a ten-membered $C(H_2)_4S_2C(H_2)_4$ chain setting apart the carboxylic acid groups at each end. This suggests that the molecule may be a good candidate for a 'spacer' in the design of compounds with metal-organic framework (MOF) structures, provided that the molecule connects the metal centres in an 'extended' fashion. However, the 'solid-state shape' of molecules such as dtba is not directly discernible from first principles, as the chain includes many sp^3 carbon atoms, which may possibly lead to twisted linkages.
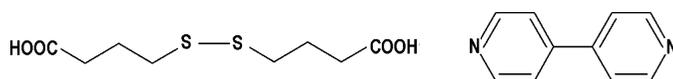
In addition, dtba is a rather uncommon ligand. The Cambridge Structure Database (Version 5.4, including June 2014 upgrades; Allen, 2002) does not at present include any entry whatsoever with the molecule, either in its coordinating or free forms, for which any direct evidence of its shape is available. We have been trying for a while to coordinate the acid to some transition metals; however, so far we have been unsuccessful. During one of these numerous attempts, a co-crystal of dtba with 4,4′-bipyridine (4,4′-bpy), $C_{10}H_8N_2$, was obtained instead. This serendipitous synthesis ended up being unique, since all subsequent attempts to obtain crystals with dtba ligand(s) in a more orthodox way have proved ineffective. We thus present herein the structural analysis of the 4,4′-bpy:dtba 1:1 co-crystal, $C_8H_{14}O_4S_2 \cdot C_{10}H_8N_2$ (I), which to our knowledge is the first crystal structure to be reported surveying the dtba group.

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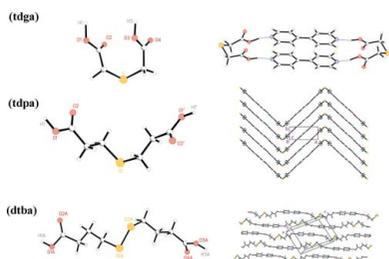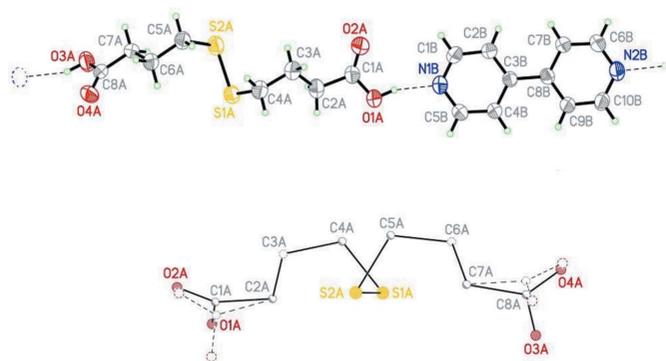
Figure 1
Top: the asymmetric unit of (I), showing the H—O···N linkages as dashed lines. Displacement ellipsoids are drawn at the 40% probability level. Bottom: the least-squares superposition of one dtba molecule and its $C2$ image, showing the pseudo-symmetry in its central core.

2. Structural commentary

Fig. 1 (top) presents an ellipsoid plot of the asymmetric unit of (I). The 'topological' (non-crystallographic) symmetry of the dtba molecule with a twofold rotation axis located at the center of the S1—S2 bond is obvious from inspection, and it is somehow reflected in the bond-length sequence, presented in Table 1 (corresponding bonds are presented in the same line). In fact, the pseudo-symmetry goes a bit further: the group presents a non-crystallographic C_{2v} symmetry involving the molecular core (C2–C7), which is reflected in the central torsion angles, *viz.* those involving S atoms (Table 1). Fig. 1 (bottom) shows the least-squares fit of this core and its C_{2v}-related image, with deviations falling in the tight range 0.011–0.015 Å. The outermost parts of the molecule (the carboxylic functions at the ends) deviate significantly from this trend, probably as a result of the strong O—H···N interactions with neighbouring 4,4′-bpy molecules (see discussion below), a fact also reflected in the torsion angles involved (last two lines in Table 1). The double bonds in the –COOH groups are non-delocalized, with the C—O(H) bonds being distinctly longer than the C=O bonds (Table 1).

In spite of the unavoidable twisting due to the individual sp^3 carbon atoms in the chain, the molecule can be considered to be stretched, with a C1···C8 span of 9.98 (1) Å and the terminal OH groups being almost anti-parallel to each other, subtending an angle of 175.5 (1)°. Thus, at least in the present

Table 1
Selected geometric parameters (Å, °).

C1A—C2A	1.513 (3)	C8A—C7A	1.497 (3)
C2A—C3A	1.487 (4)	C7A—C6A	1.507 (3)
C3A—C4A	1.522 (3)	C6A—C5A	1.522 (3)
C4A—S1A	1.811 (3)	S2A—C5A	1.805 (3)
C1A—O1A	1.309 (3)	O3A—C8A	1.325 (3)
C1A—O2A	1.198 (3)	O4A—C8A	1.197 (3)
S1A—S2A	2.0369 (14)		
C2A—C3A—C4A—S1A	−66.2 (3)	S2A—C5A—C6A—C7A	−67.6 (2)
C3A—C4A—S1A—S2A	−68.2 (2)	S1A—S2A—C5A—C6A	−67.67 (19)
O2A—C1A—C2A—C3A	−28.2 (4)	C6A—C7A—C8A—O4A	4.3 (4)
C1A—C2A—C3A—C4A	−165.0 (2)	C5A—C6A—C7A—C8A	178.3 (2)

Table 2
Hydrogen-bond geometry (Å, °).

| D—H···A	D—H	H···A	D···A	D—H···A
O1A—H1A···N1B	0.85 (1)	1.83 (1)	2.661 (3)	163 (3)
O3A—H3A···N2B^i	0.86 (1)	1.80 (1)	2.637 (3)	162 (3)
C9B—H9B···O4A^{ii}	0.93	2.49	3.404 (3)	167

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

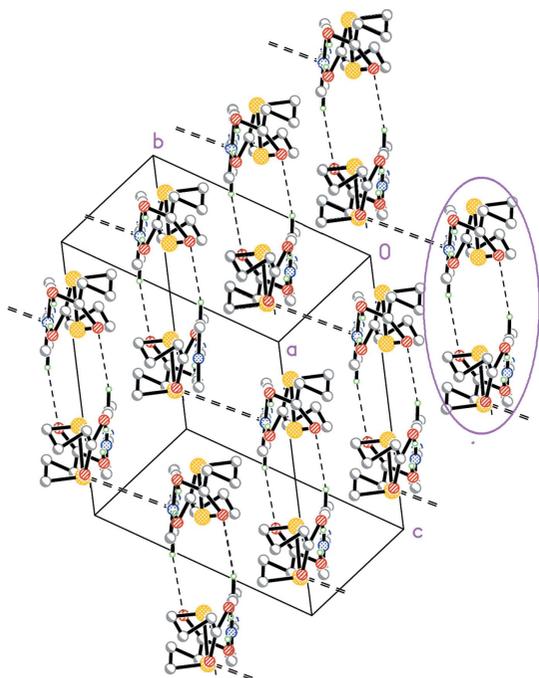structure, the molecule can be considered as a potentially adequate spacer for MOF construction.

The 4,4′-bpy molecule, in turn, is basically featureless, with slightly non-planar pyridine rings [maximum deviations from the least-squares planes: C2B 0.005 (3), N2B: 0.005 (3) Å], rotated to each other by 4.54 (13)°.

3. Supramolecular features

There are only two strong hydrogen-bond donors (the dtba carboxylic acid OH functions) and two hydrogen-bond acceptors (the pyridine N atoms of 4,4′-bpy) present, defining the supramolecular organization (first two entries in Table 2) in the form of linear chains running along $[\bar{2}11]$ (Fig. 2) with graph-set descriptor $C_2^2(22)$ (for graph-set nomenclature, see Bernstein *et al.*, 1995). Neighbouring chains, in turn, are connected into strips along [001] by a (notably weaker) C—H···O contact involving the pyridyl C9A—H9A group and one of the two non-protonated carboxylato O atoms (third entry in Table 2, shown as nearly vertical broken lines in Fig. 2), giving rise to $R_4^4(16)$ centrosymmetric loops. The chains run parallel to each other, with no obvious second-order interactions linking them, either of the C—H···O, C—H···π or π–π types. There is, however, a different type of contact present, namely a C—O···π contact involving the non-protonated O atom [C1A—O2A···Cg^i where $Cg1$ is the centroid of atoms N1A, C1A–C5A, symmetry code (i): $1 − x$, $1 − y$, $1 − z$, with O2A···$Cg1^i$ = 3.619 (3) Å; O2A···$Cg1^i$, π: 165.25°], which helps in connecting the strips together into a three-dimensional supramolecular structure (drawn in double dashed lines in Fig. 3). Thus, all potentially expected actors for the supramolecular building (OH, O and N functionalities) end up fulfilling a relevant role in the overall organization.
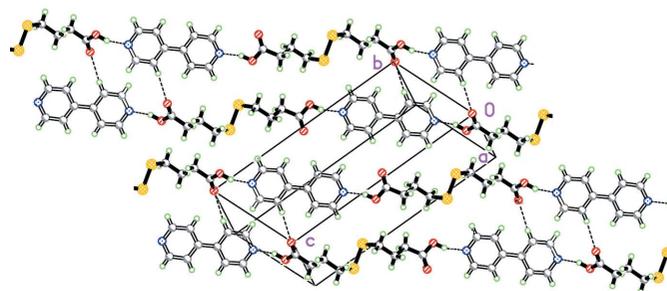
Figure 2
A packing view of (I), showing the slabs formed by neighbouring chains connected by C—H···O contacts (shown as dashed lines).

Many of these present 'extended' molecular shapes, generating chain structures with similar O—H···N synthons as in (I). Among these, alkane-types are relevant to the present discussion as they are made up of sp^3 C atoms. There are cases with $n = 5$ (glutaric acid) and $n = 6$ (adipic acid; Pedireddi *et al.*, 1998), $n = 7$ (heptane-1,7-dioic acid; Braga *et al.*, 2008) and $n = 10$ (sebacic acid; Yu *et al.*, 2006).

However, examples of adducts with thiodicarboxilyc acids are notably more rare and only two reported co-crystals of the sort can be found in the literature. These involve thiodicarboxilyc acids closely related to dtba (see scheme below): thiodiglycolic acid (tdga) and thiodipropionic acid (tdpa), *viz.* 4,4′-bpy:tdga and 4,4′-bpy:tdpa (Pedireddi *et al.*, 1998). Surprisingly, in these structures the linkers behave in a different way from dtba. Fig. 4 (left) shows the geometry of the three molecules under discussion, while Fig. 4 (right) presents the packing arrangements they give rise to.
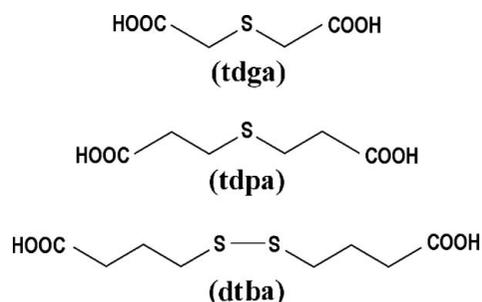

Figure 3
Packing view of (I) at right angles to the view in Fig. 2, showing the slabs in projection (one of them has been hightlighted). Single dashed lines denote the C—H···O bonds. The C—O···π contacts linking the slabs into a three-dimensional structure are shown as double dashed lines.
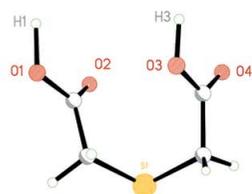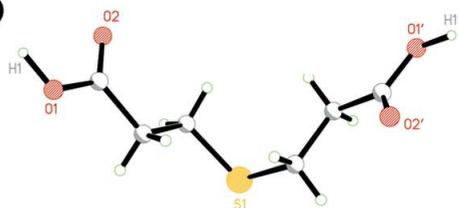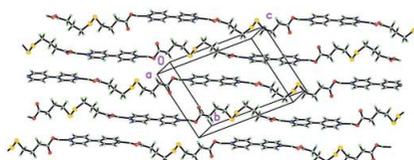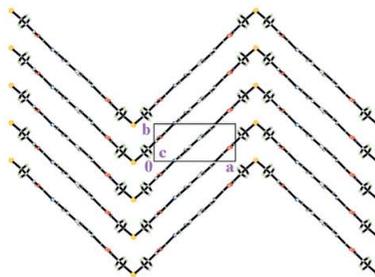
4. Database survey

A brief search of the CSD confirmed that 4,4′-bpy:dicarboxylic acid adducts are rather frequent; among the most populated families, the one derived from alkanes/alkenes ranks on top.

In the first case, (tdga co-crystal) the molecule is shaped like a horseshoe, and the terminal OH functions end up being
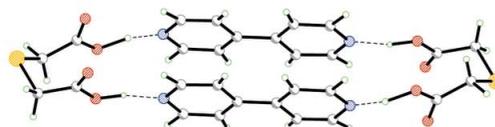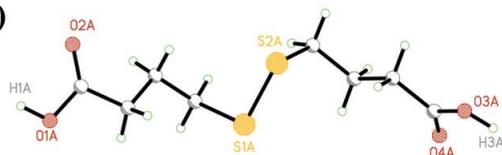
Figure 4
The three different molecular shapes for tdga, tdpa and dtba, and the packing arrangements they give rise to, as described in the text.

Table 3
Experimental details.

Crystal data	
Chemical formula	$C_8H_{14}O_4S_2 \cdot C_{10}H_8N_2$
M_r	394.49
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	297
a, b, c (Å)	5.154 (3), 11.124 (7), 17.256 (11)
α, β, γ (°)	79.096 (10), 87.126 (10), 85.030 (12)
V (Å^3)	967.3 (10)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.30
Crystal size (mm)	$0.23 \times 0.14 \times 0.11$
Data collection	
Diffractometer	Bruker *SMART* CCD area detector
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2008a)
T_{min}, T_{max}	0.94, 0.98
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	8306, 4185, 2376
R_{int}	0.031
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.656
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.131, 0.91
No. of reflections	4185
No. of parameters	243
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.26, −0.16

Computer programs: *SMART* (Bruker, 2001), *SAINT* (Bruker, 2002), *SHELXS97*, *SHELXL97* and *SHELXTL* (Sheldrick, 2008b) and *PLATON* (Spek, 2009).

almost parallel, subtending an angle of 12.5 (1)° to each other. The 4,4′-bpy aggregation motifs with this particular geometry give rise to isolated closed dimers as shown in Fig. 4 (upper right).

The tdpa molecule presents a shape somehow similar to, but noticeably more open than tdga, with H—O···O—H bonds almost at a right angle to each other [97.1 (1)°]. The resulting packing mimics this molecular geometry, in a tight herring-bone pattern (Fig. 4, mid-right). Finally, and as already discussed, the present dtba co-crystal displays a fully stretched geometry [H—O···O—H: 175.5 (1)°] and the basic packing unit is a linear chain.

From this analysis it can be concluded (at least for this type of terminal dicarboxilic acids) that the relative angular disposition of the outermost OH groups are relevant in defining the expected general aspect of the packing. In this context, dtba could be considered a potentially useful spacer for MOF construction, and further work to obtain transition-metal complexes with this ligand is in progress.

5. Synthesis and crystallization

The reported 4,4′-bpy:dtba co-crystal was obtained serendipitously from an unsuccessful synthesis of a holmium complex, prepared from an Ho_2O_3:dtba:4,4-bpy solution (in a 1:2:1 ratio), dissolved in a mixture of water (200 ml) and ethanol (20 ml). After a few days of slow evaporation at room temperature, colourless block-like crystals were obtained.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. All H atoms were originally found in a difference Fourier map, but treated differently in refinement: C—H H atoms were repositioned in their expected positions and thereafter allowed to ride with $U_{iso}(H) = 1.2U_{eq}$(host) ($d = 0.93$ Å for C—H$_{aromatic}$ and $d = 0.97$ Å for C—H$_{methylene}$), while OH H atoms were refined with a restrained distance of 0.85 (1) Å.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Braga, D., Palladino, G., Polito, M., Rubini, K., Grepioni, F., Chierotti, M. R. & Gobetto, R. (2008). *Chem. Eur. J.* **14**, 10149–10159.
Bruker (2001). *SMART*. Bruker Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Bruker (2002). *SAINT*. Bruker Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Pedireddi, V. R., Chatterjee, S., Ranganathan, A. & Rao, C. N. R. (1998). *Tetrahedron*, **54**, 9457–9474.
Sheldrick, G. M. (2008a). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008b). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Yu, B., Wang, X.-Q., Wang, R.-J., Shen, G.-Q. & Shen, D.-Z. (2006). *Acta Cryst.* E**62**, o2757–o2758.

supporting information

Crystal structure of 4,4′-(disulfanediyl)dibutanoic acid–4,4′-bipyridine (1/1)

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Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT* (Bruker, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008*b*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008*b*); molecular graphics: *SHELXTL* (Sheldrick, 2008*b*); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008*b*) and *PLATON* (Spek, 2009).

4,4′-(Disulfanediyl)dibutanoic acid–4,4′-bipyridine (1/1)

Crystal data

$C_8H_{14}O_4S_2$·$C_{10}H_8N_2$	$Z = 2$
$M_r = 394.49$	$F(000) = 416$
Triclinic, $P\overline{1}$	$D_x = 1.354$ Mg m^{-3}
$a = 5.154$ (3) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 11.124$ (7) Å	Cell parameters from 1445 reflections
$c = 17.256$ (11) Å	$\theta = 2.4$–21.1°
$\alpha = 79.096$ (10)°	$\mu = 0.30$ mm^{-1}
$\beta = 87.126$ (10)°	$T = 297$ K
$\gamma = 85.030$ (12)°	Block, colourless
$V = 967.3$ (10) Å^3	$0.23 \times 0.14 \times 0.11$ mm

Data collection

Bruker SMART CCD area detector diffractometer	8306 measured reflections
Radiation source: fine-focus sealed tube	4185 independent reflections
Graphite monochromator	2376 reflections with $I > 2\sigma(I)$
CCD rotation images, thin slices scans	$R_{int} = 0.031$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008*a*)	$\theta_{max} = 27.8°$, $\theta_{min} = 1.2°$
	$h = -6 \rightarrow 6$
	$k = -14 \rightarrow 14$
$T_{min} = 0.94$, $T_{max} = 0.98$	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.048$	
$wR(F^2) = 0.131$	$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2]$
$S = 0.91$	where $P = (F_o^2 + 2F_c^2)/3$
4185 reflections	$(\Delta/\sigma)_{max} < 0.001$
243 parameters	$\Delta\rho_{max} = 0.26$ e Å^{-3}
2 restraints	$\Delta\rho_{min} = -0.16$ e Å^{-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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1A	1.04515 (14)	−0.04804 (7)	0.29204 (4)	0.0744 (3)
S2A	0.87636 (14)	0.02612 (6)	0.18771 (4)	0.0673 (2)
O1A	0.7237 (4)	0.23802 (19)	0.51914 (11)	0.0781 (6)
H1A	0.627 (5)	0.292 (2)	0.5388 (16)	0.105 (11)*
O2A	0.4101 (4)	0.26892 (19)	0.43314 (12)	0.0884 (7)
O3A	1.4882 (4)	−0.16886 (17)	−0.02741 (11)	0.0745 (6)
H3A	1.596 (5)	−0.222 (2)	−0.0447 (18)	0.127 (13)*
O4A	1.4099 (4)	−0.32979 (16)	0.06596 (10)	0.0775 (6)
C1A	0.6119 (6)	0.2170 (2)	0.45711 (15)	0.0640 (7)
C2A	0.7747 (6)	0.1205 (3)	0.42096 (16)	0.0794 (9)
H2AA	0.9044	0.1604	0.3846	0.095*
H2AB	0.8663	0.0643	0.4625	0.095*
C3A	0.6193 (6)	0.0492 (3)	0.37810 (16)	0.0738 (8)
H3AA	0.5658	0.1007	0.3287	0.089*
H3AB	0.4630	0.0276	0.4095	0.089*
C4A	0.7675 (6)	−0.0675 (2)	0.36052 (15)	0.0735 (8)
H4AA	0.6464	−0.1145	0.3398	0.088*
H4AB	0.8274	−0.1162	0.4100	0.088*
C5A	0.7684 (5)	−0.1061 (2)	0.15574 (15)	0.0623 (7)
H5AA	0.6608	−0.1495	0.1978	0.075*
H5AB	0.6594	−0.0777	0.1106	0.075*
C6A	0.9844 (5)	−0.1966 (2)	0.13303 (14)	0.0586 (6)
H6AA	1.1029	−0.2205	0.1762	0.070*
H6AB	0.9088	−0.2698	0.1247	0.070*
C7A	1.1351 (5)	−0.1431 (2)	0.05930 (13)	0.0550 (6)
H7AA	1.0154	−0.1211	0.0163	0.066*
H7AB	1.2036	−0.0682	0.0675	0.066*
C8A	1.3562 (5)	−0.2257 (2)	0.03466 (13)	0.0549 (6)
N1B	0.4619 (4)	0.38299 (19)	0.60732 (12)	0.0634 (6)
N2B	−0.2001 (4)	0.69792 (17)	0.89024 (12)	0.0619 (6)
C1B	0.2496 (6)	0.4565 (3)	0.58974 (15)	0.0752 (8)
H1B	0.1860	0.4652	0.5393	0.090*
C2B	0.1163 (5)	0.5213 (2)	0.64262 (14)	0.0657 (7)
H2B	−0.0307	0.5732	0.6270	0.079*
C3B	0.2016 (4)	0.50899 (19)	0.71826 (12)	0.0482 (6)
C4B	0.4247 (5)	0.4314 (2)	0.73679 (13)	0.0568 (6)
H4B	0.4919	0.4201	0.7869	0.068*
C5B	0.5455 (5)	0.3712 (2)	0.68026 (14)	0.0631 (7)
H5B	0.6942	0.3194	0.6939	0.076*

C6B	−0.2730 (5)	0.7174 (2)	0.81551 (15)	0.0655 (7)
H6B	−0.4152	0.7733	0.8012	0.079*
C7B	−0.1501 (5)	0.6598 (2)	0.75835 (14)	0.0578 (6)
H7B	−0.2093	0.6767	0.7071	0.069*
C8B	0.0639 (4)	0.57597 (19)	0.77791 (13)	0.0477 (6)
C9B	0.1404 (5)	0.5567 (2)	0.85557 (13)	0.0577 (6)
H9B	0.2827	0.5019	0.8715	0.069*
C10B	0.0063 (5)	0.6186 (2)	0.90922 (14)	0.0633 (7)
H10B	0.0622	0.6045	0.9608	0.076*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1A	0.0605 (5)	0.0877 (5)	0.0845 (5)	0.0148 (4)	−0.0181 (4)	−0.0456 (4)
S2A	0.0779 (5)	0.0572 (4)	0.0685 (4)	0.0008 (3)	0.0026 (4)	−0.0206 (3)
O1A	0.0807 (14)	0.0935 (14)	0.0625 (11)	0.0290 (11)	−0.0102 (10)	−0.0350 (11)
O2A	0.0810 (15)	0.1039 (15)	0.0861 (14)	0.0257 (12)	−0.0253 (12)	−0.0411 (12)
O3A	0.0912 (15)	0.0658 (11)	0.0640 (11)	0.0144 (10)	0.0107 (10)	−0.0185 (10)
O4A	0.0936 (15)	0.0618 (11)	0.0692 (12)	0.0252 (10)	−0.0073 (10)	−0.0048 (9)
C1A	0.0731 (19)	0.0640 (16)	0.0546 (15)	0.0076 (14)	0.0012 (14)	−0.0173 (13)
C2A	0.079 (2)	0.0869 (19)	0.0806 (19)	0.0143 (16)	−0.0110 (16)	−0.0441 (17)
C3A	0.075 (2)	0.0852 (19)	0.0631 (16)	0.0072 (15)	−0.0015 (14)	−0.0248 (15)
C4A	0.094 (2)	0.0654 (16)	0.0629 (16)	0.0057 (15)	−0.0097 (15)	−0.0197 (14)
C5A	0.0628 (17)	0.0658 (15)	0.0616 (15)	−0.0032 (13)	−0.0026 (13)	−0.0207 (13)
C6A	0.0675 (18)	0.0527 (13)	0.0574 (14)	−0.0010 (12)	−0.0042 (13)	−0.0154 (12)
C7A	0.0591 (16)	0.0546 (13)	0.0504 (13)	0.0096 (12)	−0.0096 (11)	−0.0114 (11)
C8A	0.0617 (16)	0.0589 (15)	0.0463 (13)	0.0086 (12)	−0.0143 (12)	−0.0183 (12)
N1B	0.0697 (15)	0.0664 (13)	0.0553 (12)	0.0062 (11)	0.0018 (11)	−0.0208 (11)
N2B	0.0729 (15)	0.0546 (12)	0.0586 (12)	−0.0003 (11)	0.0096 (11)	−0.0164 (10)
C1B	0.076 (2)	0.101 (2)	0.0514 (15)	0.0174 (17)	−0.0120 (14)	−0.0279 (15)
C2B	0.0632 (17)	0.0813 (18)	0.0520 (14)	0.0174 (14)	−0.0094 (12)	−0.0191 (13)
C3B	0.0533 (15)	0.0453 (12)	0.0456 (12)	−0.0007 (11)	−0.0001 (11)	−0.0089 (10)
C4B	0.0652 (17)	0.0558 (14)	0.0483 (13)	0.0105 (12)	−0.0059 (12)	−0.0125 (11)
C5B	0.0687 (18)	0.0602 (15)	0.0582 (15)	0.0149 (13)	−0.0047 (13)	−0.0136 (13)
C6B	0.0674 (18)	0.0630 (15)	0.0646 (16)	0.0115 (13)	−0.0004 (14)	−0.0158 (13)
C7B	0.0579 (16)	0.0642 (15)	0.0508 (13)	0.0078 (12)	−0.0047 (12)	−0.0140 (12)
C8B	0.0522 (14)	0.0415 (11)	0.0491 (12)	−0.0021 (10)	0.0021 (11)	−0.0092 (10)
C9B	0.0715 (18)	0.0498 (13)	0.0504 (13)	0.0080 (12)	−0.0070 (12)	−0.0103 (11)
C10B	0.086 (2)	0.0576 (14)	0.0460 (13)	0.0022 (14)	−0.0018 (13)	−0.0129 (12)

Geometric parameters (Å, º)

C1A—C2A	1.513 (3)	C6A—H6AB	0.9700
C2A—C3A	1.487 (4)	C7A—H7AA	0.9700
C3A—C4A	1.522 (3)	C7A—H7AB	0.9700
C4A—S1A	1.811 (3)	N1B—C1B	1.320 (3)
C1A—O1A	1.309 (3)	N1B—C5B	1.330 (3)
C1A—O2A	1.198 (3)	N2B—C6B	1.334 (3)

S1A—S2A	2.0369 (14)	N2B—C10B	1.334 (3)
C8A—C7A	1.497 (3)	C1B—C2B	1.388 (3)
C7A—C6A	1.507 (3)	C1B—H1B	0.9300
C6A—C5A	1.522 (3)	C2B—C3B	1.377 (3)
S2A—C5A	1.805 (3)	C2B—H2B	0.9300
O3A—C8A	1.325 (3)	C3B—C4B	1.389 (3)
O4A—C8A	1.197 (3)	C3B—C8B	1.498 (3)
O1A—H1A	0.851 (10)	C4B—C5B	1.379 (3)
O3A—H3A	0.863 (10)	C4B—H4B	0.9300
C2A—H2AA	0.9700	C5B—H5B	0.9300
C2A—H2AB	0.9700	C6B—C7B	1.375 (3)
C3A—H3AA	0.9700	C6B—H6B	0.9300
C3A—H3AB	0.9700	C7B—C8B	1.393 (3)
C4A—H4AA	0.9700	C7B—H7B	0.9300
C4A—H4AB	0.9700	C8B—C9B	1.388 (3)
C5A—H5AA	0.9700	C9B—C10B	1.380 (3)
C5A—H5AB	0.9700	C9B—H9B	0.9300
C6A—H6AA	0.9700	C10B—H10B	0.9300
C4A—S1A—S2A	102.70 (10)	C6A—C7A—H7AA	108.5
C5A—S2A—S1A	102.95 (9)	C8A—C7A—H7AB	108.5
C1A—O1A—H1A	109 (2)	C6A—C7A—H7AB	108.5
C8A—O3A—H3A	108 (2)	H7AA—C7A—H7AB	107.5
O2A—C1A—O1A	123.6 (2)	O4A—C8A—O3A	123.4 (2)
O2A—C1A—C2A	126.0 (3)	O4A—C8A—C7A	125.4 (2)
O1A—C1A—C2A	110.4 (2)	O3A—C8A—C7A	111.2 (2)
C3A—C2A—C1A	113.4 (2)	C1B—N1B—C5B	116.8 (2)
C3A—C2A—H2AA	108.9	C6B—N2B—C10B	117.1 (2)
C1A—C2A—H2AA	108.9	N1B—C1B—C2B	123.4 (2)
C3A—C2A—H2AB	108.9	N1B—C1B—H1B	118.3
C1A—C2A—H2AB	108.9	C2B—C1B—H1B	118.3
H2AA—C2A—H2AB	107.7	C3B—C2B—C1B	120.0 (2)
C2A—C3A—C4A	113.2 (2)	C3B—C2B—H2B	120.0
C2A—C3A—H3AA	108.9	C1B—C2B—H2B	120.0
C4A—C3A—H3AA	108.9	C2B—C3B—C4B	116.6 (2)
C2A—C3A—H3AB	108.9	C2B—C3B—C8B	122.2 (2)
C4A—C3A—H3AB	108.9	C4B—C3B—C8B	121.2 (2)
H3AA—C3A—H3AB	107.8	C5B—C4B—C3B	119.4 (2)
C3A—C4A—S1A	116.6 (2)	C5B—C4B—H4B	120.3
C3A—C4A—H4AA	108.1	C3B—C4B—H4B	120.3
S1A—C4A—H4AA	108.1	N1B—C5B—C4B	123.8 (2)
C3A—C4A—H4AB	108.1	N1B—C5B—H5B	118.1
S1A—C4A—H4AB	108.1	C4B—C5B—H5B	118.1
H4AA—C4A—H4AB	107.3	N2B—C6B—C7B	123.8 (2)
C6A—C5A—S2A	115.37 (18)	N2B—C6B—H6B	118.1
C6A—C5A—H5AA	108.4	C7B—C6B—H6B	118.1
S2A—C5A—H5AA	108.4	C6B—C7B—C8B	119.4 (2)
C6A—C5A—H5AB	108.4	C6B—C7B—H7B	120.3

S2A—C5A—H5AB	108.4	C8B—C7B—H7B	120.3
H5AA—C5A—H5AB	107.5	C9B—C8B—C7B	116.6 (2)
C7A—C6A—C5A	112.23 (19)	C9B—C8B—C3B	121.9 (2)
C7A—C6A—H6AA	109.2	C7B—C8B—C3B	121.5 (2)
C5A—C6A—H6AA	109.2	C10B—C9B—C8B	120.3 (2)
C7A—C6A—H6AB	109.2	C10B—C9B—H9B	119.9
C5A—C6A—H6AB	109.2	C8B—C9B—H9B	119.9
H6AA—C6A—H6AB	107.9	N2B—C10B—C9B	122.8 (2)
C8A—C7A—C6A	115.2 (2)	N2B—C10B—H10B	118.6
C8A—C7A—H7AA	108.5	C9B—C10B—H10B	118.6
C2A—C3A—C4A—S1A	−66.2 (3)	C1B—N1B—C5B—C4B	−0.4 (4)
C3A—C4A—S1A—S2A	−68.2 (2)	C3B—C4B—C5B—N1B	0.3 (4)
O2A—C1A—C2A—C3A	−28.2 (4)	C10B—N2B—C6B—C7B	0.8 (4)
C1A—C2A—C3A—C4A	−165.0 (2)	N2B—C6B—C7B—C8B	−0.2 (4)
S2A—C5A—C6A—C7A	−67.6 (2)	C6B—C7B—C8B—C9B	−0.4 (4)
S1A—S2A—C5A—C6A	−67.67 (19)	C6B—C7B—C8B—C3B	178.8 (2)
C6A—C7A—C8A—O4A	4.3 (4)	C2B—C3B—C8B—C9B	175.5 (3)
C5A—C6A—C7A—C8A	178.3 (2)	C4B—C3B—C8B—C9B	−4.9 (4)
C6A—C7A—C8A—O3A	−175.9 (2)	C2B—C3B—C8B—C7B	−3.7 (4)
C5B—N1B—C1B—C2B	0.9 (5)	C4B—C3B—C8B—C7B	175.9 (2)
N1B—C1B—C2B—C3B	−1.2 (5)	C7B—C8B—C9B—C10B	0.3 (4)
C1B—C2B—C3B—C4B	1.0 (4)	C3B—C8B—C9B—C10B	−179.0 (2)
C1B—C2B—C3B—C8B	−179.5 (2)	C6B—N2B—C10B—C9B	−0.9 (4)
C2B—C3B—C4B—C5B	−0.5 (4)	C8B—C9B—C10B—N2B	0.4 (4)
C8B—C3B—C4B—C5B	179.9 (2)	O1A—C1A—C2A—C3A	152.4 (3)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
O1A—H1A···N1B	0.85 (1)	1.83 (1)	2.661 (3)	163 (3)
O3A—H3A···N2B[i]	0.86 (1)	1.80 (1)	2.637 (3)	162 (3)
C9B—H9B···O4A[ii]	0.93	2.49	3.404 (3)	167

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