

2-(4-Bromobenzenesulfonamido)acetic acid

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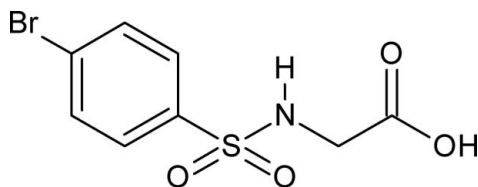
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.058; wR factor = 0.168; data-to-parameter ratio = 18.7.

The title compound, $\text{C}_8\text{H}_8\text{BrNO}_4\text{S}$, a halogenated sulfonamide, was prepared by basic hydrolysis of the methyl ester. In the crystal, molecules form centrosymmetric hydrogen-bonded dimers *via* the carboxyl groups. These dimers are further linked by $\text{N}-\text{H}\cdots\text{O}$ interactions involving the carbonyl O and amide H atoms, forming a ribbon-like structure propagating in [010]. These ribbons are further linked *via* $\text{C}-\text{H}\cdots\text{O}$ interactions, forming a three-dimensional network.

Related literature

For details of the crystal structure of the methyl ester of the title compound, see: Arshad *et al.* (2008*b*). For related structures, see: Arshad *et al.* (2008*a*); Arshad *et al.* (2009). For related thiazine heterocycles, see: Arshad *et al.* (2008*c*). For hydrogen-bonding patterns, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_8\text{H}_8\text{BrNO}_4\text{S}$
 $M_r = 294.12$
Triclinic, $P\bar{1}$

$a = 5.0042$ (4) Å
 $b = 7.9997$ (6) Å
 $c = 13.2289$ (11) Å

$\alpha = 79.691$ (4)°
 $\beta = 88.667$ (5)°
 $\gamma = 81.404$ (4)°
 $V = 515.18$ (7) Å³
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 4.18$ mm⁻¹
 $T = 296$ K
 $0.28 \times 0.17 \times 0.11$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2007)
 $T_{\min} = 0.612$, $T_{\max} = 0.632$

10359 measured reflections
2557 independent reflections
1243 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.056$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$
 $wR(F^2) = 0.168$
 $S = 0.95$
2557 reflections

137 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.15$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.38$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O3}-\text{H3O}\cdots\text{O4}^{\text{i}}$	0.82	1.85	2.671 (5)	174
$\text{N1}-\text{H1}\cdots\text{O4}^{\text{ii}}$	0.86	2.38	3.124 (5)	146
$\text{C2}-\text{H2}\cdots\text{O1}^{\text{iii}}$	0.93	2.53	3.384 (7)	153
$\text{C3}-\text{H3}\cdots\text{O3}^{\text{iv}}$	0.93	2.50	3.423 (7)	170

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

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2009) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2493).

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supplementary materials

Acta Cryst. (2009). E65, o1816 [doi:10.1107/S160053680902604X]

2-(4-Bromobenzenesulfonamido)acetic acid

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Comment

The title compound, (I), was prepared by basic hydrolysis of methyl (4-bromobenzenesulfonamido)acetate (II) (Arshad *et al.*, 2008b), in a continuation of our studies on the synthesis of thiazine related heterocycles (Arshad *et al.*, 2008c). We have previously reported the crystal structures of 2-(benzenesulfonamido)acetic acid (III) (Arshad *et al.*, 2008a) and 2-(2-iodobenzenesulfonamido)acetic acid (IV) (Arshad *et al.*, 2009).

The molecular structure of (I), Fig. 1, reveals the bond lengths and angles are similar to those found for compounds (II), (III) and (IV).

The presence of the carboxylic acid group leads to the formation of characteristic O—H \cdots O hydrogen-bonded centrosymmetric dimers (Table 1 and Fig. 2). These dimers are linked *via* N1—H1 \cdots O4 interactions, involving the carbonyl O-atom and the H-atom of the amido group, to form a ribbon-like structure propagating in the [010] direction (Table 1 and Fig. 2). The ribbons are further linked by C—H \cdots O interactions to form a 3-D network (Table 1 and Fig. 3).

It is interesting to compare the hydrogen bonding patterns in the three acids; (I), (III) and (IV). The formation of the hydrogen bonded carboxylic acid dimers is the same in all three compounds, i.e. $R^2_2(8)$ (Bernstein *et al.*, 1995). The N—H \cdots O hydrogen-bonding involves the sulfonamido groups in (III) and (IV) [$R^2_2(8)$], while in (I) it involves the carbonyl O-atom (O4) and the H-atom of the amido group (Table 1). This leads to a larger hydrogen-bonded ring of the form [$R^2_2(10)$], as shown in Fig. 4.

Experimental

Methyl (4-bromobenzenesulfonamido)acetate(II) (Arshad *et al.*, 2008b) (1.0 g, 3.247 mmol) was dissolved in an aqueous sodium hydroxide solution (10%, 10 ml). The resulting solution was refluxed for an hour. The reaction mixture was then cooled to room temperature and acidified with 1 N HCl. A white precipitate was obtained. This was filtered off, washed with distilled water and dried. Crystals were obtained by recrystallization from methanol.

Refinement

The H-atoms were included in calculated positions and treated as riding atoms: O—H = 0.82 Å, N—H = 0.86 Å, C—H = 0.93 - 0.97 Å, with $U_{\text{iso}}(\text{H}) = k \times U_{\text{eq}}(\text{parent O-, N- or C-atom})$, where $k = 1.5$ for OH, and 1.2 for N- and C-bound H-atoms.

The maximum and minimum residual electron density peaks of 1.15 and $-0.38 \text{ e}\text{\AA}^{-3}$, respectively, were located at 1.10 Å and 0.78 Å, respectively, from atom Br1.

Figures

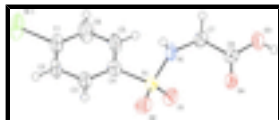


Fig. 1. The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

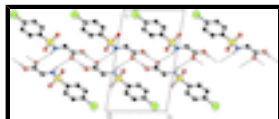


Fig. 2. A view along the *a* axis of the crystal packing of compound (I), with O—H...O and N—H...O hydrogen bonds drawn as dashed lines [see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for clarity].

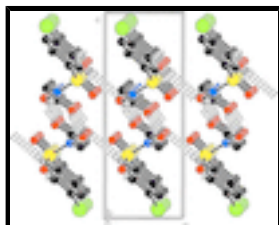


Fig. 3. A view along the *b* axis of the crystal packing of (I), with O—H...O, N—H...O and C—H...O hydrogen bonds drawn as dashed lines [see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for clarity].

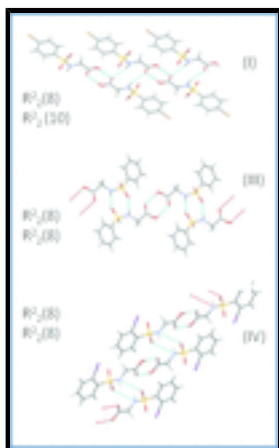


Fig. 4. A view of the hydrogen bonding patterns in the three acid compounds: (I), (III) and (IV). The hydrogen bonds are shown as pale-blue lines.

2-(4-Bromobenzenesulfonamido)acetic acid

Crystal data

$C_8H_8BrNO_4S$

$M_r = 294.12$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 5.0042$ (4) Å

$b = 7.9997$ (6) Å

$c = 13.2289$ (11) Å

$\alpha = 79.691$ (4)°

$\beta = 88.667$ (5)°

$\gamma = 81.404$ (4)°

$V = 515.18$ (7) Å³

$Z = 2$

$F_{000} = 292$

$D_x = 1.896$ Mg m⁻³

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

Cell parameters from 1869 reflections

$\theta = 2.2$ – 21.8 °

$\mu = 4.18$ mm⁻¹

$T = 296$ K

Needle, colorless

$0.28 \times 0.17 \times 0.11$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer	2557 independent reflections
Radiation source: fine-focus sealed tube	1243 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.056$
$T = 296$ K	$\theta_{\text{max}} = 28.3^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.6^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2007)	$h = -6 \rightarrow 6$
$T_{\text{min}} = 0.612$, $T_{\text{max}} = 0.632$	$k = -10 \rightarrow 10$
10359 measured reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.058$	H-atom parameters constrained
$wR(F^2) = 0.168$	$w = 1/[\sigma^2(F_o^2) + (0.0872P)^2 + 0.2177P]$
$S = 0.95$	where $P = (F_o^2 + 2F_c^2)/3$
2557 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
137 parameters	$\Delta\rho_{\text{max}} = 1.15 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.38 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

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	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.70371 (17)	0.78972 (8)	0.05167 (5)	0.0774 (4)
S1	0.3354 (2)	0.11701 (17)	0.32101 (10)	0.0365 (4)
O1	0.2650 (7)	0.0106 (5)	0.2528 (3)	0.0486 (10)
O2	0.1387 (7)	0.1738 (5)	0.3930 (3)	0.0492 (10)
O3	0.7775 (7)	-0.4424 (5)	0.4169 (3)	0.0505 (10)

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H3O	0.7174	-0.5221	0.4536	0.076*
O4	0.4156 (7)	-0.2871 (4)	0.4743 (3)	0.0427 (9)
N1	0.5998 (8)	0.0162 (5)	0.3816 (3)	0.0389 (10)
H1	0.6632	0.0630	0.4278	0.047*
C1	0.5927 (12)	0.5917 (7)	0.1309 (4)	0.0456 (14)
C2	0.3808 (12)	0.6089 (7)	0.1979 (5)	0.0495 (15)
H2	0.2914	0.7172	0.2039	0.059*
C3	0.3022 (11)	0.4635 (7)	0.2560 (5)	0.0453 (14)
H3	0.1575	0.4737	0.3010	0.054*
C4	0.4368 (10)	0.3039 (7)	0.2478 (4)	0.0355 (12)
C5	0.6490 (11)	0.2867 (7)	0.1801 (4)	0.0489 (15)
H5	0.7386	0.1784	0.1742	0.059*
C6	0.7271 (13)	0.4320 (8)	0.1211 (5)	0.0584 (17)
H6	0.8695	0.4221	0.0751	0.070*
C7	0.7389 (10)	-0.1470 (6)	0.3652 (4)	0.0410 (13)
H7A	0.9274	-0.1559	0.3839	0.049*
H7B	0.7316	-0.1519	0.2925	0.049*
C8	0.6253 (10)	-0.2978 (7)	0.4249 (4)	0.0368 (12)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1144 (7)	0.0372 (4)	0.0728 (6)	-0.0176 (4)	0.0098 (4)	0.0154 (3)
S1	0.0376 (7)	0.0264 (7)	0.0458 (8)	-0.0085 (5)	0.0029 (5)	-0.0043 (6)
O1	0.053 (2)	0.037 (2)	0.060 (3)	-0.0124 (18)	-0.0073 (18)	-0.0143 (19)
O2	0.045 (2)	0.046 (2)	0.058 (2)	-0.0118 (18)	0.0163 (18)	-0.011 (2)
O3	0.050 (2)	0.027 (2)	0.069 (3)	0.0001 (18)	0.0167 (19)	-0.001 (2)
O4	0.0398 (19)	0.027 (2)	0.059 (2)	-0.0058 (16)	0.0127 (17)	-0.0008 (17)
N1	0.050 (2)	0.019 (2)	0.046 (3)	-0.0062 (19)	-0.002 (2)	-0.001 (2)
C1	0.064 (4)	0.026 (3)	0.044 (3)	-0.009 (3)	-0.004 (3)	0.002 (3)
C2	0.059 (3)	0.017 (3)	0.068 (4)	0.003 (3)	0.000 (3)	-0.006 (3)
C3	0.045 (3)	0.025 (3)	0.065 (4)	-0.002 (2)	0.008 (3)	-0.007 (3)
C4	0.041 (3)	0.027 (3)	0.038 (3)	-0.003 (2)	-0.001 (2)	-0.005 (2)
C5	0.064 (4)	0.022 (3)	0.054 (4)	0.001 (3)	0.013 (3)	0.004 (3)
C6	0.073 (4)	0.040 (4)	0.054 (4)	-0.001 (3)	0.021 (3)	0.006 (3)
C7	0.039 (3)	0.032 (3)	0.049 (3)	-0.010 (2)	0.009 (2)	0.001 (3)
C8	0.035 (3)	0.033 (3)	0.042 (3)	-0.007 (2)	0.000 (2)	-0.003 (2)

Geometric parameters (\AA , $^\circ$)

Br1—C1	1.886 (5)	C2—C3	1.379 (7)
S1—O1	1.429 (4)	C2—H2	0.9300
S1—O2	1.436 (4)	C3—C4	1.374 (7)
S1—N1	1.594 (4)	C3—H3	0.9300
S1—C4	1.765 (5)	C4—C5	1.380 (7)
O3—C8	1.306 (6)	C5—C6	1.382 (7)
O3—H3O	0.8200	C5—H5	0.9300
O4—C8	1.223 (6)	C6—H6	0.9300
N1—C7	1.436 (6)	C7—C8	1.499 (6)

N1—H1	0.8600	C7—H7A	0.9700
C1—C2	1.373 (8)	C7—H7B	0.9700
C1—C6	1.379 (8)		
O1—S1—O2	119.3 (2)	C3—C4—C5	120.5 (5)
O1—S1—N1	106.7 (2)	C3—C4—S1	120.6 (4)
O2—S1—N1	109.3 (2)	C5—C4—S1	118.9 (4)
O1—S1—C4	108.9 (2)	C4—C5—C6	119.4 (5)
O2—S1—C4	106.5 (2)	C4—C5—H5	120.3
N1—S1—C4	105.3 (2)	C6—C5—H5	120.3
C8—O3—H3O	109.5	C1—C6—C5	119.6 (5)
C7—N1—S1	124.8 (4)	C1—C6—H6	120.2
C7—N1—H1	117.6	C5—C6—H6	120.2
S1—N1—H1	117.5	N1—C7—C8	113.8 (4)
C2—C1—C6	121.0 (5)	N1—C7—H7A	108.8
C2—C1—Br1	119.6 (4)	C8—C7—H7A	108.8
C6—C1—Br1	119.4 (4)	N1—C7—H7B	108.8
C1—C2—C3	119.2 (5)	C8—C7—H7B	108.8
C1—C2—H2	120.4	H7A—C7—H7B	107.7
C3—C2—H2	120.4	O4—C8—O3	124.3 (5)
C4—C3—C2	120.2 (5)	O4—C8—C7	124.4 (5)
C4—C3—H3	119.9	O3—C8—C7	111.4 (4)
C2—C3—H3	119.9		
O1—S1—N1—C7	2.2 (4)	O1—S1—C4—C5	-57.6 (5)
O2—S1—N1—C7	132.5 (4)	O2—S1—C4—C5	172.6 (4)
C4—S1—N1—C7	-113.5 (4)	N1—S1—C4—C5	56.6 (5)
C6—C1—C2—C3	0.0 (9)	C3—C4—C5—C6	0.6 (9)
Br1—C1—C2—C3	-179.5 (4)	S1—C4—C5—C6	178.9 (5)
C1—C2—C3—C4	0.8 (9)	C2—C1—C6—C5	-0.5 (10)
C2—C3—C4—C5	-1.1 (8)	Br1—C1—C6—C5	179.1 (5)
C2—C3—C4—S1	-179.4 (4)	C4—C5—C6—C1	0.2 (9)
O1—S1—C4—C3	120.7 (5)	S1—N1—C7—C8	-85.5 (5)
O2—S1—C4—C3	-9.1 (5)	N1—C7—C8—O4	8.2 (8)
N1—S1—C4—C3	-125.2 (5)	N1—C7—C8—O3	-172.4 (4)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O3—H3O \cdots O4 ⁱ	0.82	1.85	2.671 (5)	174
N1—H1 \cdots O4 ⁱⁱ	0.86	2.38	3.124 (5)	146
C2—H2 \cdots O1 ⁱⁱⁱ	0.93	2.53	3.384 (7)	153
C3—H3 \cdots O3 ^{iv}	0.93	2.50	3.423 (7)	170
C3—H3 \cdots O2	0.93	2.50	2.884 (7)	105

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

Fig. 1

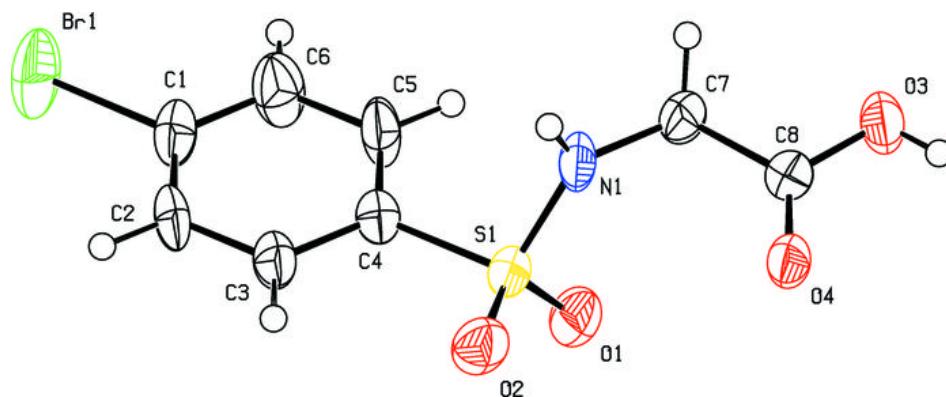


Fig. 2

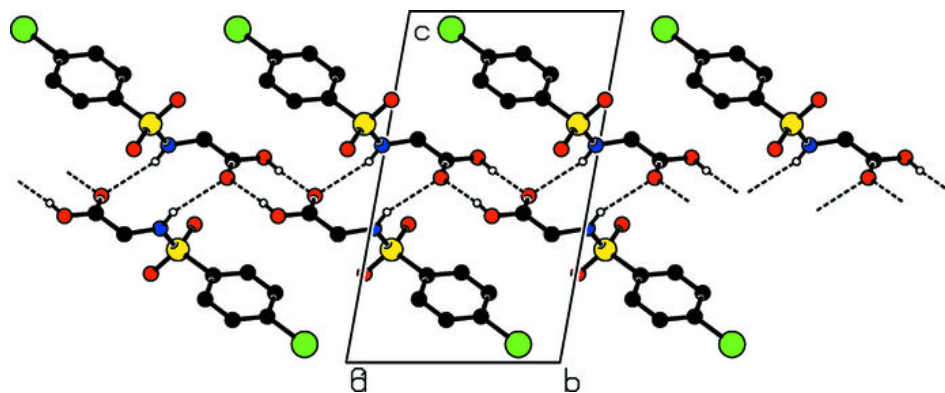


Fig. 3

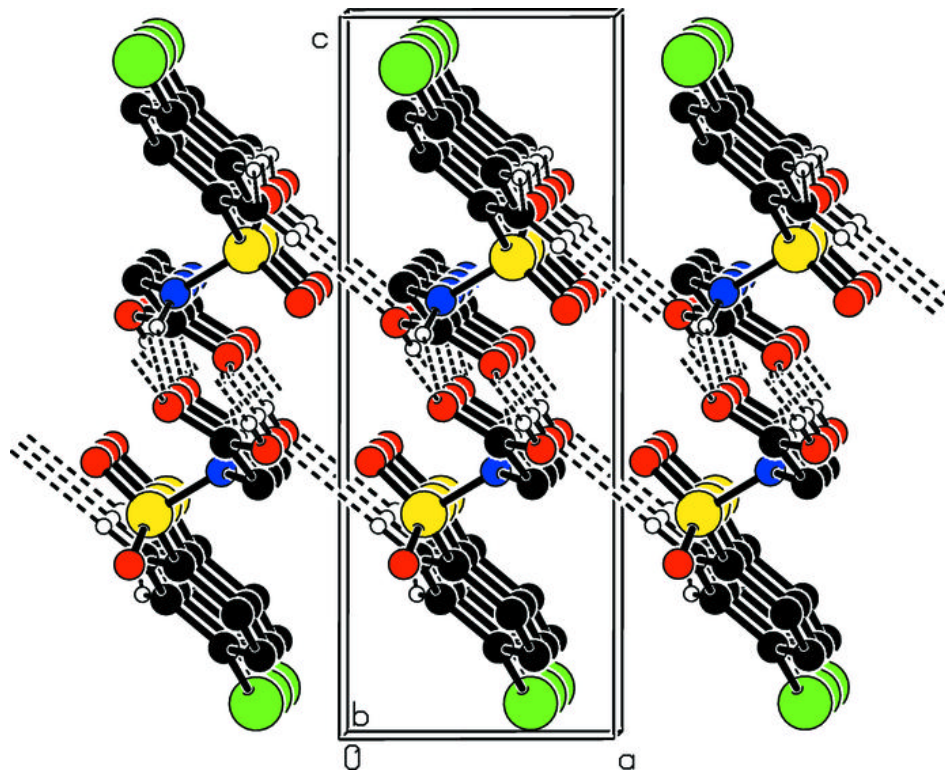


Fig. 4

