

Potassium pentaborate

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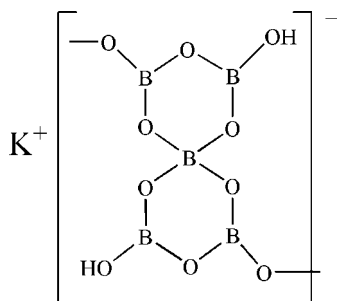
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{B}-\text{O}) = 0.003$ Å; R factor = 0.030; wR factor = 0.072; data-to-parameter ratio = 10.7.

The title compound, $\text{K}[\text{B}_5\text{O}_7(\text{OH})_2]$, was obtained from a hydrothermal reaction. The structure is composed of one K^+ cation and a polyborate $[\text{B}_5\text{O}_7(\text{OH})_2]^-$ anion, which consists of two six-membered rings linked by a common BO_4 tetrahedron. The $[\text{B}_5\text{O}_7(\text{OH})_2]^-$ units are linked together through two exocyclic O atoms to neighbouring units, forming a helical chain structure extending parallel to $[010]$. Adjacent chains are further connected into a three-dimensional structure by $\text{K}-\text{O}$ bonds and weak $\text{O}-\text{H}\cdots\text{O}$ hydrogen-bond interactions.

Related literature

For the nonlinear optical properties of alkali metal borates, see: Mori *et al.* (1995). For syntheses and crystal structures in the $\text{K}_2\text{O}-\text{B}_2\text{O}_3-\text{H}_2\text{O}$ system, see: Marezio (1969); Salentine (1987); Wang *et al.* (2006); Zhang *et al.* (2005).



Experimental

Crystal data

$\text{K}[\text{B}_5\text{O}_7(\text{OH})_2]$
 $M_r = 239.17$
 Monoclinic, $P2_1/c$
 $a = 7.6690$ (3) Å
 $b = 9.0445$ (3) Å
 $c = 12.2304$ (4) Å
 $\beta = 119.132$ (2)°

$V = 741.01$ (5) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.74$ mm⁻¹
 $T = 100$ K
 $0.14 \times 0.09 \times 0.07$ mm

Data collection

Bruker APEXII diffractometer
 Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 2008a)
 $T_{\min} = 0.878$, $T_{\max} = 0.910$

13002 measured reflections
 1452 independent reflections
 1343 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.072$
 $S = 1.00$
 1452 reflections

136 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.77$ e Å⁻³
 $\Delta\rho_{\min} = -0.87$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O}10-\text{H}10A\cdots\text{O}6^i$	0.84	2.36	3.179 (2)	164
$\text{O}12-\text{H}12A\cdots\text{O}11^{ii}$	0.84	2.30	3.0346 (19)	147
$\text{O}12-\text{H}12A\cdots\text{O}4^{ii}$	0.84	2.50	3.170 (2)	138

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008b); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008b); molecular graphics: *SHELXTL* (Sheldrick, 2008b); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FI2113).

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supplementary materials

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Comment

Boron can form a large variety of compounds due to the variability of the coordination environment about B. In the past several decades, much interest has focused on studies of alkali metals borates because some of these compounds show interesting physical properties, such as nonlinear optical behavior for CsLiB₆O₁₀ (Mori *et al.*, 1995). So far, several phases had been obtained in the K₂O–B₂O₃–H₂O system (Marezio, 1969; Salentine, 1987; Zhang *et al.*, 2005; Wang *et al.*, 2006). In this paper, we describe the synthesis and the crystal structure of a new potassium borate, K[B₅O₇(OH)₂].

It features one K⁺ cation and a ¹_∞[B₅O₇(OH)₂][−] polyborate anion (Fig.1), which is closely related to the reported compound of K[B₅O₇(OH)₂]·H₂O (Zhang *et al.*, 2005).

The ¹_∞[B₅O₇(OH)₂][−] ion consists of two six-membered rings linked by a common B atom. Each six-membered ring consists of one BO₃ triangle, one BO₂(OH) triangle and a common BO₄ tetrahedron. The [B₅O₇(OH)₂][−] units are linked via two exocyclic O atoms (O8 and O8A) to neighboring units, forming a 1-D helical chainlike structure (Fig. 2). Adjacent chains are further connected into a 3-D structure by K—O bonds and O—H···O hydrogen bonds interactions, as shown in Fig.3.

Experimental

All reagents used in the synthesis were analytic grade and were used without further purification. A mixture of GaO(OH) (0.06 g), H₃BO₃ (0.47 g), KNO₃ (0.15 g) and distilled water (0.1 ml) was sealed in a Teflon-lined bomb and heated at 483 K for 3 d and then cooled to room temperature. The resulting colorless crystals were washed with hot deionized water and dried in a vacuum dryer to a constant mass at room temperature.

Refinement

H atoms bonded to O10 and O12 atoms were positioned geometrically, and were refined riding with O—H = 0.84 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Figures

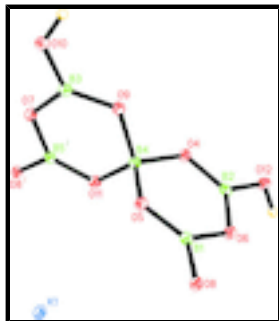


Fig. 1. The asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes: (i) $-x, -1/2 + y, -1/2 - z$.

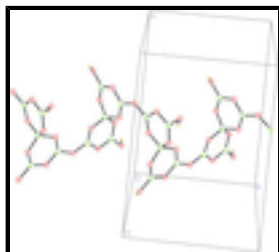


Fig. 2. The one-dimensional chain structure constructed by $[\text{B}_5\text{O}_7(\text{OH})_2]^-$ units. B, O and H atoms are shown as green, red and yellow, respectively.

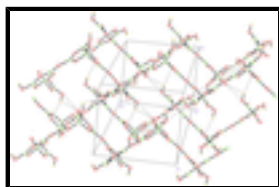


Fig. 3. Packing view along the c axis of title compound, showing three-dimensional structure constructed by $\text{O}—\text{H}\cdots\text{O}$ hydrogen bonds, where all potassium cations are omitted for clarity. B, O and H atoms are shown as green, red and yellow, respectively. H bonds are drawn as dashed lines.

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Crystal data

$\text{K}[\text{B}_5\text{O}_7(\text{OH})_2]$

$M_r = 239.17$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 7.6690$ (3) Å

$b = 9.0445$ (3) Å

$c = 12.2304$ (4) Å

$\beta = 119.132$ (2)°

$V = 741.01$ (5) Å³

$Z = 4$

$F(000) = 472$

$D_x = 2.144$ Mg m⁻³

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

Cell parameters from 6851 reflections

$\theta = 3.0$ – 30.5 °

$\mu = 0.74$ mm⁻¹

$T = 100$ K

Rod, colourless

$0.14 \times 0.09 \times 0.07$ mm

Data collection

Bruker APEXII
diffractometer

Radiation source: fine-focus sealed tube
graphite

Detector resolution: 83.33 pixels mm⁻¹

1452 independent reflections

1343 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\text{max}} = 26.0$ °, $\theta_{\text{min}} = 3.0$ °

φ and ω scans $h = -9 \rightarrow 8$
 Absorption correction: multi-scan $k = -11 \rightarrow 11$
 (*SADABS*; Sheldrick, 2008a) $l = -15 \rightarrow 15$
 $T_{\min} = 0.878$, $T_{\max} = 0.910$
 13002 measured reflections

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.030$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.072$	H-atom parameters constrained
$S = 1.00$	$w = 1/[\sigma^2(F_o^2) + (0.026P)^2 + 1.7263P]$
1452 reflections	where $P = (F_o^2 + 2F_c^2)/3$
136 parameters	$(\Delta/\sigma)_{\max} < 0.001$
0 restraints	$\Delta\rho_{\max} = 0.77 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.87 \text{ e } \text{\AA}^{-3}$

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	$U_{\text{iso}}^*/U_{\text{eq}}$
O4	-0.2494 (2)	-0.13046 (15)	-0.14778 (13)	0.0093 (3)
O5	-0.0757 (2)	-0.32468 (15)	-0.19460 (13)	0.0095 (3)
O6	0.0327 (2)	-0.25360 (15)	0.01760 (12)	0.0096 (3)
O7	-0.4011 (2)	-0.14669 (15)	-0.50951 (12)	0.0097 (3)
O8	0.1858 (2)	-0.45303 (15)	-0.01975 (13)	0.0091 (3)
O9	-0.4141 (2)	-0.26660 (15)	-0.33902 (12)	0.0095 (3)
O10	-0.6325 (2)	-0.33377 (16)	-0.55281 (13)	0.0111 (3)
H10A	-0.7360	-0.3223	-0.5465	0.017*
O11	-0.1489 (2)	-0.08947 (15)	-0.30142 (13)	0.0101 (3)
O12	-0.1416 (2)	-0.06260 (16)	0.05911 (13)	0.0108 (3)
H12A	-0.0286	-0.0380	0.1172	0.016*
B1	0.0458 (3)	-0.3419 (2)	-0.0712 (2)	0.0088 (4)
B2	-0.1192 (3)	-0.1489 (2)	-0.0252 (2)	0.0094 (4)

supplementary materials

B3	-0.4815 (3)	-0.2516 (2)	-0.4635 (2)	0.0095 (4)
B4	-0.2217 (3)	-0.2019 (2)	-0.2457 (2)	0.0093 (4)
B5	0.2421 (3)	-0.5629 (2)	-0.0753 (2)	0.0091 (4)
K1	0.34799 (7)	-0.07665 (5)	-0.26481 (4)	0.01448 (14)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O4	0.0097 (7)	0.0089 (7)	0.0088 (7)	0.0014 (5)	0.0040 (6)	0.0006 (5)
O5	0.0104 (7)	0.0097 (7)	0.0085 (7)	0.0015 (5)	0.0047 (6)	0.0002 (5)
O6	0.0104 (7)	0.0098 (7)	0.0080 (7)	0.0016 (5)	0.0041 (6)	-0.0002 (5)
O7	0.0109 (7)	0.0097 (7)	0.0080 (6)	-0.0017 (6)	0.0040 (6)	-0.0001 (5)
O8	0.0102 (7)	0.0092 (7)	0.0077 (6)	0.0014 (5)	0.0043 (6)	0.0001 (5)
O9	0.0093 (7)	0.0100 (7)	0.0089 (7)	-0.0010 (5)	0.0041 (6)	0.0008 (5)
O10	0.0096 (7)	0.0126 (7)	0.0112 (7)	-0.0027 (6)	0.0051 (6)	-0.0009 (6)
O11	0.0092 (7)	0.0112 (7)	0.0085 (7)	-0.0021 (5)	0.0032 (6)	0.0009 (5)
O12	0.0098 (7)	0.0115 (7)	0.0095 (7)	0.0018 (5)	0.0035 (6)	-0.0015 (5)
B1	0.0095 (10)	0.0070 (10)	0.0105 (10)	-0.0015 (8)	0.0052 (9)	-0.0004 (8)
B2	0.0101 (10)	0.0073 (10)	0.0124 (11)	-0.0014 (8)	0.0067 (9)	0.0003 (8)
B3	0.0097 (10)	0.0074 (10)	0.0118 (11)	0.0016 (8)	0.0054 (9)	-0.0003 (8)
B4	0.0098 (10)	0.0083 (10)	0.0092 (10)	0.0000 (8)	0.0043 (9)	0.0005 (8)
B5	0.0100 (10)	0.0082 (10)	0.0111 (10)	-0.0005 (8)	0.0067 (9)	0.0000 (8)
K1	0.0143 (2)	0.0188 (3)	0.0091 (2)	0.00475 (18)	0.00474 (18)	0.00124 (17)

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

O4—B2	1.347 (3)	O9—B3	1.356 (3)
O4—B4	1.464 (3)	O9—B4	1.477 (3)
O5—B1	1.342 (3)	O10—B3	1.362 (3)
O5—B4	1.482 (3)	O10—H10A	0.8400
O6—B1	1.391 (3)	O11—B5 ⁱ	1.339 (3)
O6—B2	1.391 (3)	O11—B4	1.477 (3)
O7—B5 ⁱ	1.381 (3)	O12—B2	1.369 (3)
O7—B3	1.391 (3)	O12—H12A	0.8400
O8—B1	1.379 (3)	B5—O11 ⁱⁱ	1.339 (3)
O8—B5	1.386 (3)	B5—O7 ⁱⁱ	1.381 (3)
B2—O4—B4	122.10 (16)	O12—B2—O6	119.56 (18)
B1—O5—B4	121.96 (16)	O9—B3—O10	123.79 (19)
B1—O6—B2	117.54 (16)	O9—B3—O7	121.38 (18)
B5 ⁱ —O7—B3	118.31 (16)	O10—B3—O7	114.82 (17)
B1—O8—B5	131.13 (17)	O4—B4—O11	108.14 (16)
B3—O9—B4	121.29 (16)	O4—B4—O9	108.59 (16)
B3—O10—H10A	109.4	O11—B4—O9	112.07 (16)
B5 ⁱ —O11—B4	121.94 (16)	O4—B4—O5	111.51 (16)
B2—O12—H12A	109.4	O11—B4—O5	109.41 (16)
O5—B1—O8	123.92 (19)	O9—B4—O5	107.14 (16)
O5—B1—O6	122.50 (18)	O11 ⁱⁱ —B5—O7 ⁱⁱ	122.64 (18)

O8—B1—O6	113.46 (17)	O11 ⁱⁱ —B5—O8	123.92 (19)
O4—B2—O12	118.11 (18)	O7 ⁱⁱ —B5—O8	113.41 (18)
O4—B2—O6	122.32 (18)		
B4—O5—B1—O8	-178.99 (18)	B2—O4—B4—O11	-103.7 (2)
B4—O5—B1—O6	5.3 (3)	B2—O4—B4—O9	134.42 (18)
B5—O8—B1—O5	3.4 (3)	B2—O4—B4—O5	16.6 (3)
B5—O8—B1—O6	179.45 (18)	B5 ⁱ —O11—B4—O4	-125.34 (19)
B2—O6—B1—O5	3.0 (3)	B5 ⁱ —O11—B4—O9	-5.7 (3)
B2—O6—B1—O8	-173.13 (17)	B5 ⁱ —O11—B4—O5	113.01 (19)
B4—O4—B2—O12	171.24 (17)	B3—O9—B4—O4	135.83 (18)
B4—O4—B2—O6	-10.0 (3)	B3—O9—B4—O11	16.4 (3)
B1—O6—B2—O4	-0.7 (3)	B3—O9—B4—O5	-103.6 (2)
B1—O6—B2—O12	178.03 (17)	B1—O5—B4—O4	-14.3 (3)
B4—O9—B3—O10	165.13 (18)	B1—O5—B4—O11	105.3 (2)
B4—O9—B3—O7	-16.1 (3)	B1—O5—B4—O9	-132.97 (18)
B5 ⁱ —O7—B3—O9	3.6 (3)	B1—O8—B5—O11 ⁱⁱ	-6.9 (3)
B5 ⁱ —O7—B3—O10	-177.49 (17)	B1—O8—B5—O7 ⁱⁱ	175.04 (18)

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

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

<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>
O10—H10A \cdots O6 ⁱⁱⁱ	0.84	2.36	3.179 (2)	164
O12—H12A \cdots O11 ^{iv}	0.84	2.30	3.0346 (19)	147
O12—H12A \cdots O4 ^{iv}	0.84	2.50	3.170 (2)	138

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

Fig. 1

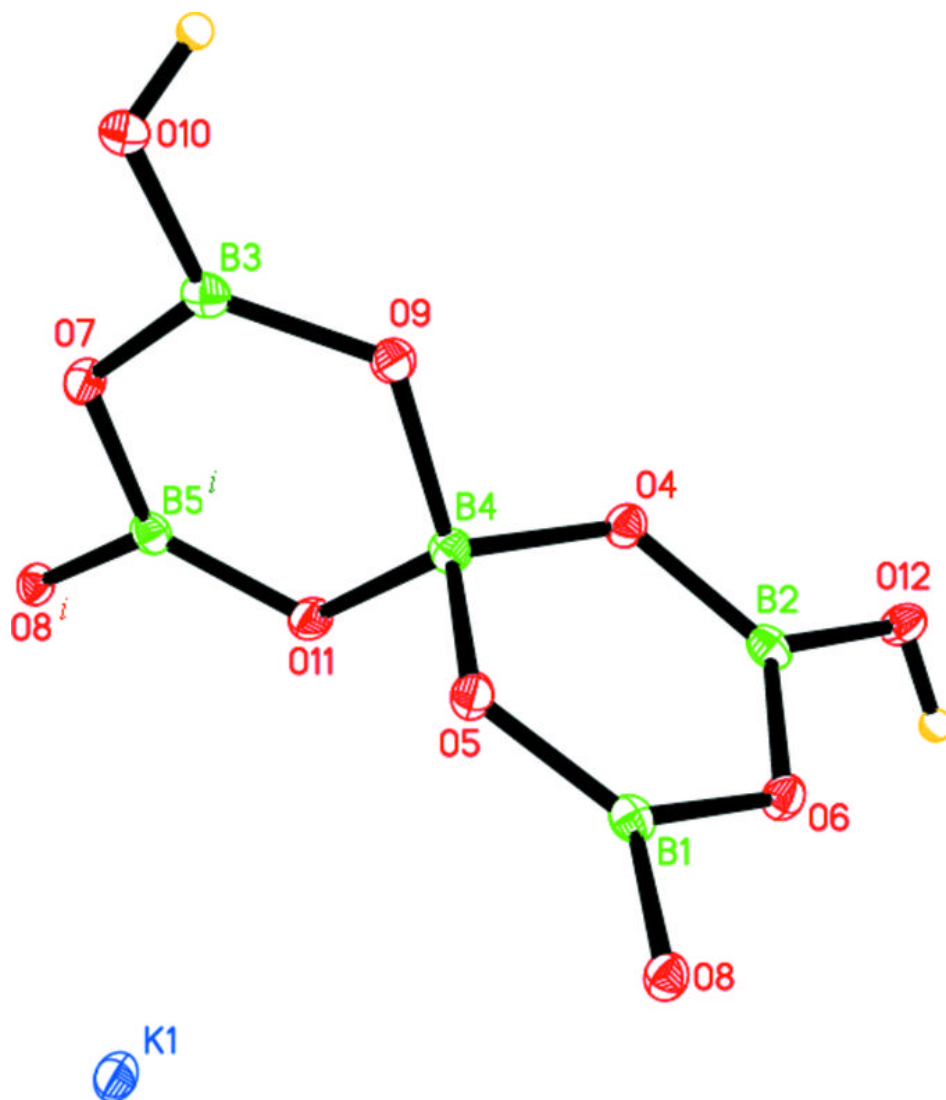


Fig. 2

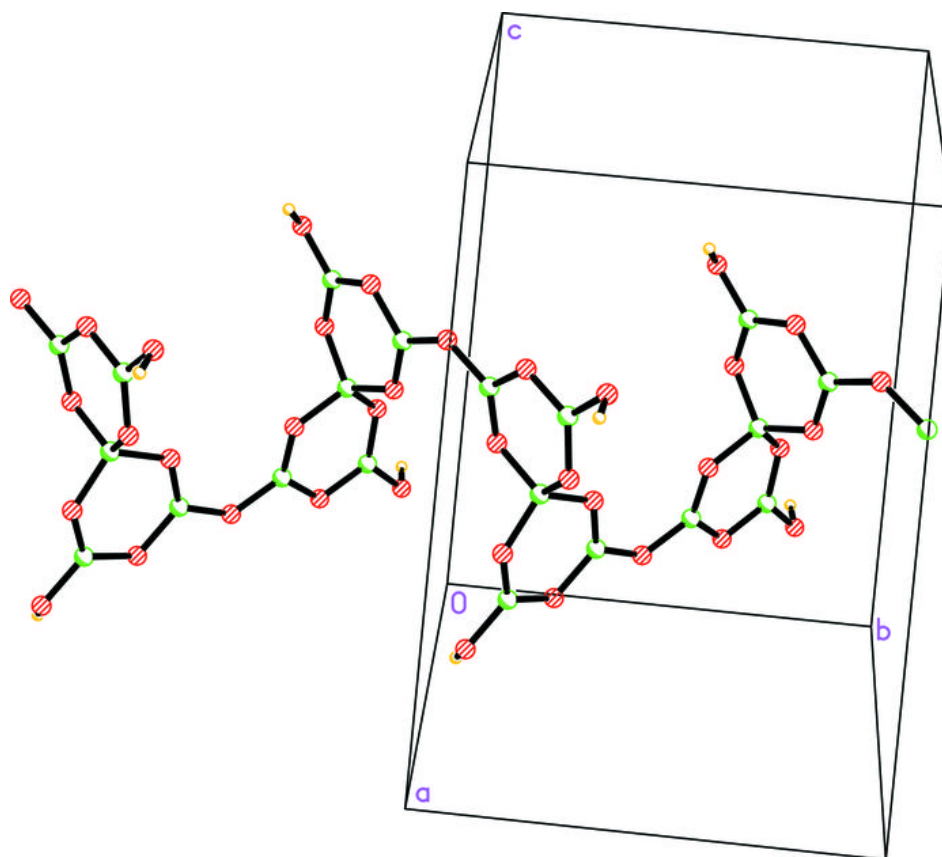


Fig. 3

