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Poly[triaqua(μ_4-5-sulfosalicylato)-potassium(I)]

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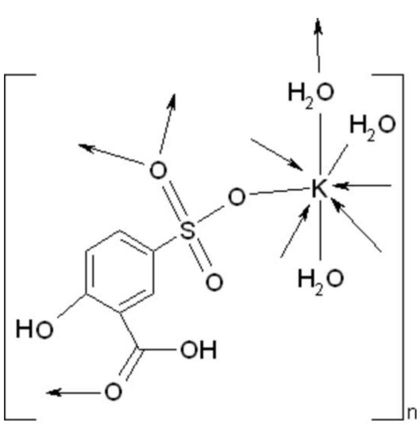
Key indicators: single-crystal X-ray study; T = 223 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.129; data-to-parameter ratio = 11.4.

In the title polymeric complex, $[K(C_7H_5O_6S)(H_2O)_3]_n$, the anions use their sulfonyl and double-bond carbonyl O atoms to link to four K^+ cations in a μ_4-bonding mode. The compound has a three-dimensional framework which consists of dinuclear $[K_2O_{10}]$ fragments linked by sulfonate and carboxylic O atoms from the sulfosalicylate (ssa$^-$) ligand. In the three-dimensional network, there are different types of channels along the a, b and c axes. In the crystal structure, classical intermolecular O—H···O hydrogen bonds also stabilize the three-dimensional structure.

Related literature

For related literature, see: Braga *et al.* (1998); Distler & Sevov (1998); Drumel *et al.* (1995); Gao *et al.* (2005); Henderson *et al.* (2003); Hix *et al.* (2001); Kennedy *et al.* (2004, 2006); Kitagawa *et al.* (2004); Lloret *et al.* (1998); Ma *et al.* (2005); Moulton & Zaworotko (2001); Sheldrick (2000); Yaghi *et al.* (1997, 2003).



Experimental

Crystal data

$[K(C_7H_5O_6S)(H_2O)_3]$
M_r = 310.32
Triclinic, $P\overline{1}$
a = 7.2648 (2) Å
b = 7.2760 (2) Å
c = 11.9225 (4) Å
α = 87.394 (2)°
β = 81.907 (2)°
γ = 72.313 (2)°
V = 594.44 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.66 mm^{-1}
T = 223 (2) K
0.60 × 0.38 × 0.30 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.693, T_{max} = 0.827
5725 measured reflections
2149 independent reflections
2002 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.129
S = 1.01
2149 reflections
188 parameters
11 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

K1—O2^{i}	2.6686 (15)	K1—O9	2.9338 (16)
K1—O5^{ii}	2.7315 (14)	K1—O8^{iii}	2.9394 (18)
K1—O8	2.7345 (16)	K1—O6^{iv}	2.9556 (14)
K1—O6	2.7604 (15)	K1—O7	3.2456 (19)
O2^{i}—K1—O5^{ii}	75.99 (5)	O6—K1—O9	129.16 (4)
O2^{i}—K1—O8	123.24 (5)	O5^{ii}—K1—O8^{iii}	61.99 (5)
O5^{ii}—K1—O8	142.02 (5)	O6—K1—O8^{iii}	142.48 (5)
O5^{ii}—K1—O6	136.33 (5)	O2^{i}—K1—O6^{iv}	125.55 (5)
O8—K1—O6	78.32 (5)	O6—K1—O6^{iv}	72.10 (4)
O2^{i}—K1—O9	69.64 (4)		

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1B···O2	0.840 (10)	1.839 (18)	2.586 (2)	147 (3)
O3—H3A···O9^{i}	0.845 (10)	1.838 (11)	2.674 (2)	170 (2)
O7—H7A···O6^{iv}	0.847 (10)	2.092 (13)	2.917 (2)	164 (3)
O7—H7B···O4^{ii}	0.843 (10)	2.021 (12)	2.852 (2)	168 (2)
O8—H8A···O5^{v}	0.862 (10)	2.55 (3)	2.926 (2)	108 (2)
O8—H8A···O7^{iv}	0.862 (10)	2.50 (2)	2.997 (2)	117.9 (18)
O8—H8B···O4^{vi}	0.870 (10)	2.037 (11)	2.879 (2)	163 (2)
O9—H9A···O3^{i}	0.847 (9)	1.945 (16)	2.674 (2)	144 (2)
O9—H9B···O7^{vi}	0.859 (10)	1.910 (11)	2.763 (2)	172 (2)

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1994); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1994); 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: HJ3040).

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supplementary materials

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Poly[triaqua(μ_4-5-sulfosalicylato)potassium(I)]

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Comment

The rational design and synthesis of metal-organic high-dimensional networks are of great interest because of their intriguing structural diversity and their potential applications as functional solid materials (Moulton *et al.*,2001; Yaghi *et al.*,2003; Kitagawa *et al.*,2004). The study of solid-state metal-organic coordination frameworks has concentrated on transition metal complexes, while little attention being paid to the s-block metals, such as Na and K (Braga *et al.*,1998; Henderson *et al.*, 2003; Sheldrick *et al.*, 2000). Pharmaceuticals, dyes, and pigments typically utilize alkali and alkaline earth cations in preference to transition metals as, in broad terms, the s-block metals have advantages of nontoxicity, cheapness, and often aqueous solubility (Alan *et al.*,2006). So far most of these materials are formed with N-containing ligands, such as 4,4'-bipyridine and pyrazine(Yaghi *et al.*,1997; Lloret *et al.*,1998). The study on ligands containing different coordinating groups is relatively limited(Distler *et al.*,1998; Hix *et al.*,2001; Drumel *et al.*,1995), and we are interested in coordination polymers containing such ligands. It has been demonstrated that the 5-sulfosalicylic acid(Hssa) is a multifunctional ligand(Ma *et al.*, 2005; Gao *et al.*,2005), which has three potential coordinating groups –OH, –CO_2H and –SO_3H. Both –CO_2H and –SO_3H are versatile coordinating groups, that can coordinate to metal in a variety of modes. Until now, the study on the coordination polymers constructed using the Hssa ligand and main group metals with three-dimensional open framework is very rare. In this paper, the first potassium complex with an interesting 3-D framework structure, [K(ssa)(H_2O)$_3$](Hssa=5-sulfosalicylic acid) I, was synthesized in aqueous solution and characterizated by single-crystal diffraction.

There is one crystallographically unique potassium ions in the structure (Fig. 1 and Table 1). The K^+ ion is 8-coordinated by four water molecules (O7, O8, O9 and O8C, symmetry code C: $-x-1, -y, -z+2$),three sulfonate oxygen atoms (O6, O6D and O5E, symmetry code D: $-x, -y, -z+2$; E: $x, y-1, z$) and one double-bond carboxylic oxygen atom (O2B, symmetry code B:-x, $-y$, $-z+1$) from four different ssa^- anions. The K—O(water) distances and potassium sulfonate oxygen distances range from 2.732 (2) to 3.242 (2) and 2.731 (2) to 2.757 (2) Å, respectively, which are similar to reported values (Kennedy *et al.*,2004). The potassium carboxylic oxygen distance is 2.668 (2) Å, while the O—K—O bond angles are in the range of 34.55 (3) to 163.77 (4)°.

In compound (I), –SO_3H group is deprotonated, but the –CO_2H and –OH groups are neutral. The K1 and its symmetry-related ion (K1C, symmetry code C: $-x-1$,-y,-$z+2$) are linked by two μ_2-O from water ligands (O8 and O8C, symmetry code C: $-x-1, -y, -z+2$) to form a dinuclear unit (K_2O_{10}), in which the K1···K1C (symmetry code C: $-x-1$,-y,-$z+2$) distance is 4.3273 (9) Å.

Each –SO_3 group in the ssa^- anion acts as a tridentate ligand linked with three different potassium ions through two sulfonate oxygen atoms, in which one binds to two potassium ions from the dinuclear unit while the other binds to only a single potassium ion. The –CO_2H group monodentately coordinate to one potassium ion from the dinuclear unit. Thus, each ssa^- anion acting as tetradentate ligand binding to four potassium ions.

As shown in Figs. 2 and 3, the network structure of (I) may be described in terms of a three-dimensional open framework which consists of dinuclear $\{K_2O_{10}\}$ fragments linked by sulfonate(carboxylic) oxygen atoms from the ssa$^-$ ligands. The most unusual feature is that in the 3-D network of (I), there exist different types of channels along the *a*, *b* and *c* axes. In fact, there are intercrossing three-dimensional channels in the open framework of compound (I).

In the crystal structure, classical intermolecular O—H···O hydrogen bonds are observed (Table 2),which link –OH, $–CO_2H$ and $–SO_3H$ in the ssa$^-$ ligand and coordinated water molecules and also stabilize the 3-D structure.

Experimental

An aqueous solution (10 cm^3) of 5-sulfosalicylic acid(0.219 g, 1.0 mmol) was added dropwise to a stirred solution(10 cm^3) of KCl(0.091 g, 1.2 mmol) at 70°C for 20 min. Then the solution was adjusted to pH=2.8 by the addition of dilute HCl solution. The resulting colerless solution was allowed to stand in air at room temperature for a week, yielding block colorless crystals(65%). Elemental analysis found(calculated): C 24.01(23.99),H 3.46(3.50), S 3.39(3.36).

Refinement

The water and hydroxyl H atoms were found in a difference Fourier map and were refined with distance restraints O—H=0.85 (1)Å and H···H=1.39 (1) Å. H atoms bonded to C atoms were treated as riding, with U_{iso}(H) values equal to 1.2U_{eq}(C) and C—H distances of 0.93 Å.

Figures

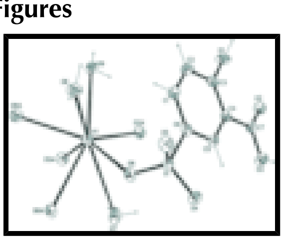

Fig. 1. View of the asymmetric unit of (I) with the atom labeling scheme. Displacement ellipsoids are drawn at the 35% probability level and H atoms are drawn as spheres of arbitrary radii·The symmetry codes are those used in Table 1.

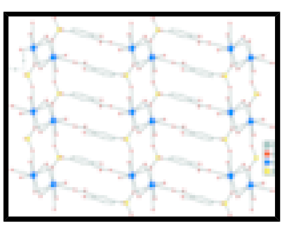

Fig. 2. Ball-stick views of compound (I) showing different channels along the *a* axis. The H atoms of the coordinated water are omitted for clarity.

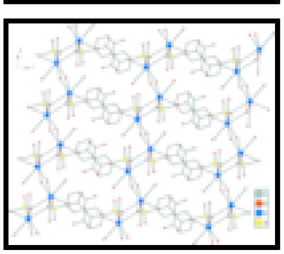

Fig. 3. Ball-stick views of compound (I) showing different channels along the *b* axis. The H atoms of the coordinated water are omitted for clarity.

poly[triaqua(μ_4-5-sulfosalicylato)potassium(I)]

Crystal data

$[K(C_7H_5O_6S)(H_2O)_3]$	V = 594.44 (3) Å^3
M_r = 310.32	Z = 2
Triclinic, $P\overline{1}$	F_{000} = 320
Hall symbol: -P 1	D_x = 1.734 Mg m^{-3}
a = 7.2648 (2) Å	Mo $K\alpha$ radiation λ = 0.71070 Å
b = 7.2760 (2) Å	μ = 0.66 mm^{-1}
c = 11.9225 (4) Å	T = 223 (2) K
α = 87.394 (2)º	Block, colourless
β = 81.907 (2)º	0.60 × 0.38 × 0.30 mm
γ = 72.313 (2)º	

Data collection

Siemens SMART CCD area-detector diffractometer	2149 independent reflections
Radiation source: fine-focus sealed tube	2002 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.017
T = 223(2) K	θ_{max} = 25.4º
φ and ω scans	θ_{min} = 3.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -8 \rightarrow 8$
T_{min} = 0.693, T_{max} = 0.827	$k = -8 \rightarrow 8$
5725 measured reflections	$l = -13 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.035	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.129	$w = 1/[\sigma^2(F_o^2) + (0.112P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.014
2149 reflections	$\Delta\rho_{max}$ = 0.57 e Å^{-3}
188 parameters	$\Delta\rho_{min}$ = −0.66 e Å^{-3}
11 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
K1	−0.21738 (6)	−0.07091 (6)	0.89768 (4)	0.0259 (2)
S1	−0.00136 (7)	0.35575 (6)	0.84164 (4)	0.0204 (2)
O1	−0.2733 (2)	0.2594 (3)	0.41097 (13)	0.0375 (4)
H1B	−0.178 (3)	0.218 (4)	0.3606 (17)	0.045*
O2	0.0876 (2)	0.1545 (2)	0.32035 (12)	0.0336 (4)
O3	0.3188 (2)	0.1565 (2)	0.42516 (13)	0.0344 (4)
H3A	0.388 (3)	0.135 (3)	0.3612 (12)	0.041*
O4	0.2022 (2)	0.3544 (2)	0.81377 (12)	0.0277 (4)
O5	−0.1288 (2)	0.5369 (2)	0.88906 (12)	0.0303 (4)
O6	−0.0158 (2)	0.1913 (2)	0.91284 (12)	0.0286 (4)
O7	0.2490 (3)	−0.2851 (2)	0.86574 (14)	0.0408 (4)
H7B	0.244 (5)	−0.390 (3)	0.8412 (19)	0.061*
H7A	0.201 (4)	−0.271 (4)	0.9349 (11)	0.061*
O8	−0.5126 (2)	0.2531 (2)	0.96902 (14)	0.0377 (4)
H8A	−0.519 (4)	0.341 (3)	1.0167 (18)	0.045*
H8B	−0.613 (3)	0.301 (3)	0.933 (2)	0.045*
O9	−0.5442 (2)	−0.0411 (2)	0.77160 (13)	0.0309 (4)
H9B	−0.614 (3)	−0.108 (3)	0.8055 (16)	0.037*
H9A	−0.523 (4)	−0.071 (3)	0.7020 (9)	0.037*
C1	−0.2767 (3)	0.3642 (3)	0.70429 (18)	0.0281 (5)
H1A	−0.3683	0.4060	0.7690	0.034*
C2	−0.3391 (3)	0.3402 (3)	0.60464 (18)	0.0311 (5)
H2A	−0.4729	0.3647	0.6013	0.037*
C3	−0.2040 (3)	0.2791 (3)	0.50731 (17)	0.0264 (5)
C4	−0.0053 (3)	0.2436 (3)	0.51325 (16)	0.0222 (4)
C5	0.0560 (3)	0.2686 (3)	0.61575 (16)	0.0209 (4)
H5A	0.1894	0.2453	0.6200	0.025*
C6	−0.0788 (3)	0.3274 (3)	0.71112 (16)	0.0210 (4)
C7	0.1370 (3)	0.1813 (3)	0.41096 (17)	0.0240 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0292 (3)	0.0247 (3)	0.0241 (3)	−0.0091 (2)	−0.0019 (2)	−0.0032 (2)
S1	0.0232 (3)	0.0198 (3)	0.0183 (3)	−0.0071 (2)	−0.0006 (2)	−0.0029 (2)
O1	0.0273 (9)	0.0596 (11)	0.0249 (8)	−0.0089 (8)	−0.0084 (6)	−0.0059 (7)
O2	0.0330 (9)	0.0487 (9)	0.0183 (8)	−0.0118 (7)	−0.0009 (6)	−0.0043 (7)
O3	0.0260 (9)	0.0536 (10)	0.0241 (8)	−0.0148 (8)	0.0033 (6)	−0.0075 (7)
O4	0.0246 (8)	0.0335 (8)	0.0270 (8)	−0.0116 (6)	−0.0023 (6)	−0.0065 (6)
O5	0.0364 (9)	0.0249 (7)	0.0257 (8)	−0.0039 (6)	−0.0005 (6)	−0.0083 (6)
O6	0.0372 (9)	0.0274 (7)	0.0241 (8)	−0.0137 (6)	−0.0057 (6)	0.0012 (6)
O7	0.0563 (11)	0.0404 (9)	0.0337 (9)	−0.0304 (8)	0.0058 (8)	−0.0100 (7)
O8	0.0310 (9)	0.0449 (9)	0.0326 (9)	−0.0036 (7)	−0.0041 (7)	−0.0067 (7)
O9	0.0297 (8)	0.0392 (9)	0.0249 (8)	−0.0140 (7)	0.0028 (6)	−0.0047 (7)
C1	0.0237 (11)	0.0316 (11)	0.0245 (11)	−0.0045 (9)	0.0032 (8)	−0.0017 (9)
C2	0.0195 (10)	0.0409 (12)	0.0297 (11)	−0.0052 (9)	−0.0017 (8)	0.0001 (9)
C3	0.0290 (11)	0.0275 (10)	0.0224 (10)	−0.0069 (9)	−0.0066 (8)	0.0010 (8)
C4	0.0245 (10)	0.0212 (9)	0.0190 (10)	−0.0054 (8)	0.0008 (8)	−0.0008 (8)
C5	0.0190 (10)	0.0221 (9)	0.0218 (10)	−0.0070 (8)	−0.0018 (7)	−0.0007 (7)
C6	0.0258 (10)	0.0182 (9)	0.0189 (10)	−0.0065 (8)	−0.0026 (8)	−0.0027 (7)
C7	0.0262 (10)	0.0244 (10)	0.0205 (10)	−0.0077 (8)	0.0001 (8)	−0.0001 (8)

Geometric parameters (Å, °)

K1—O2^{i}	2.6686 (15)	O3—H3A	0.845 (10)
K1—O5^{ii}	2.7315 (14)	O5—K1^{v}	2.7315 (14)
K1—O8	2.7345 (16)	O6—K1^{iv}	2.9556 (14)
K1—O6	2.7604 (15)	O7—H7B	0.843 (10)
K1—O9	2.9338 (16)	O7—H7A	0.847 (10)
K1—O8^{iii}	2.9394 (18)	O8—K1^{iii}	2.9394 (18)
K1—O6^{iv}	2.9556 (14)	O8—H8A	0.862 (10)
K1—O7	3.2456 (19)	O8—H8B	0.870 (10)
K1—K1^{iii}	4.3258 (9)	O9—H9B	0.859 (10)
K1—K1^{iv}	4.6230 (9)	O9—H9A	0.847 (9)
K1—H7A	3.03 (3)	C1—C2	1.366 (3)
S1—O5	1.4465 (14)	C1—C6	1.394 (3)
S1—O6	1.4569 (14)	C1—H1A	0.9400
S1—O4	1.4662 (15)	C2—C3	1.403 (3)
S1—C6	1.7683 (19)	C2—H2A	0.9400
O1—C3	1.347 (3)	C3—C4	1.398 (3)
O1—H1B	0.840 (10)	C4—C5	1.395 (3)
O2—C7	1.227 (3)	C4—C7	1.474 (3)
O2—K1^{i}	2.6686 (15)	C5—C6	1.382 (3)
O3—C7	1.311 (3)	C5—H5A	0.9400
O2^{i}—K1—O5^{ii}	75.99 (5)	O6^{iv}—K1—H7A	40.9 (2)
O2^{i}—K1—O8	123.24 (5)	O7—K1—H7A	15.0 (2)

$O5^{ii}$—K1—O8	142.02 (5)	$K1^{iii}$—K1—H7A	136.3 (2)
$O2^{i}$—K1—O6	95.58 (5)	$K1^{iv}$—K1—H7A	45.4 (4)
$O5^{ii}$—K1—O6	136.33 (5)	O5—S1—O6	112.44 (9)
O8—K1—O6	78.32 (5)	O5—S1—O4	113.18 (9)
$O2^{i}$—K1—O9	69.64 (4)	O6—S1—O4	110.93 (9)
$O5^{ii}$—K1—O9	88.56 (5)	O5—S1—C6	106.78 (9)
O8—K1—O9	71.83 (5)	O6—S1—C6	107.43 (8)
O6—K1—O9	129.16 (4)	O4—S1—C6	105.60 (8)
$O2^{i}$—K1—$O8^{iii}$	121.94 (5)	C3—O1—H1B	108.0 (19)
$O5^{ii}$—K1—$O8^{iii}$	61.99 (5)	C7—O2—$K1^{i}$	143.75 (14)
O8—K1—$O8^{iii}$	80.71 (5)	C7—O3—H3A	108.3 (18)
O6—K1—$O8^{iii}$	142.48 (5)	S1—O5—$K1^{v}$	148.50 (9)
O9—K1—$O8^{iii}$	71.07 (4)	S1—O6—K1	131.03 (8)
$O2^{i}$—K1—$O6^{iv}$	125.55 (5)	S1—O6—$K1^{iv}$	120.92 (8)
$O5^{ii}$—K1—$O6^{iv}$	78.51 (4)	K1—O6—$K1^{iv}$	107.90 (4)
O8—K1—$O6^{iv}$	106.27 (5)	K1—O7—H7B	97 (2)
O6—K1—$O6^{iv}$	72.10 (4)	K1—O7—H7A	68 (2)
O9—K1—$O6^{iv}$	155.59 (5)	H7B—O7—H7A	110.3 (16)
$O8^{iii}$—K1—$O6^{iv}$	84.56 (4)	K1—O8—$K1^{iii}$	99.29 (5)
$O2^{i}$—K1—O7	69.99 (4)	K1—O8—H8A	131.3 (16)
$O5^{ii}$—K1—O7	67.47 (4)	$K1^{iii}$—O8—H8A	98 (2)
O8—K1—O7	146.52 (5)	K1—O8—H8B	122.5 (15)
O6—K1—O7	69.40 (4)	$K1^{iii}$—O8—H8B	81.3 (19)
O9—K1—O7	136.88 (4)	H8A—O8—H8B	104.8 (14)
$O8^{iii}$—K1—O7	120.54 (4)	K1—O9—H9B	109.1 (16)
$O6^{iv}$—K1—O7	55.87 (4)	K1—O9—H9A	120.1 (17)
$O2^{i}$—K1—$K1^{iii}$	134.91 (4)	H9B—O9—H9A	107.3 (14)
$O5^{ii}$—K1—$K1^{iii}$	100.32 (4)	C2—C1—C6	120.72 (19)
O8—K1—$K1^{iii}$	42.11 (4)	C2—C1—H1A	119.6
O6—K1—$K1^{iii}$	114.26 (4)	C6—C1—H1A	119.6
O9—K1—$K1^{iii}$	65.32 (3)	C1—C2—C3	120.2 (2)
$O8^{iii}$—K1—$K1^{iii}$	38.60 (3)	C1—C2—H2A	119.9
$O6^{iv}$—K1—$K1^{iii}$	96.46 (3)	C3—C2—H2A	119.9
O7—K1—$K1^{iii}$	150.75 (3)	O1—C3—C4	122.78 (18)
$O2^{i}$—K1—$K1^{iv}$	115.45 (4)	O1—C3—C2	117.81 (18)
$O5^{ii}$—K1—$K1^{iv}$	107.73 (4)	C4—C3—C2	119.40 (18)
O8—K1—$K1^{iv}$	93.33 (4)	C5—C4—C3	119.68 (17)
O6—K1—$K1^{iv}$	37.47 (3)	C5—C4—C7	120.71 (17)
O9—K1—$K1^{iv}$	163.58 (3)	C3—C4—C7	119.62 (17)
$O8^{iii}$—K1—$K1^{iv}$	114.39 (4)	C6—C5—C4	120.24 (18)
$O6^{iv}$—K1—$K1^{iv}$	34.62 (3)	C6—C5—H5A	119.9

O7—K1—K1^{iv}	55.33 (3)	C4—C5—H5A	119.9
K1^{iii}—K1—K1^{iv}	108.502 (17)	C5—C6—C1	119.76 (18)
O2^{i}—K1—H7A	84.9 (2)	C5—C6—S1	120.37 (15)
O5^{ii}—K1—H7A	68.1 (5)	C1—C6—S1	119.87 (15)
O8—K1—H7A	138.7 (4)	O2—C7—O3	123.26 (19)
O6—K1—H7A	68.5 (5)	O2—C7—C4	122.08 (18)
O9—K1—H7A	149.2 (3)	O3—C7—C4	114.66 (17)
O8^{iii}—K1—H7A	111.5 (4)		

O6—S1—O5—K1^{v}	159.19 (15)	O8^{iii}—K1—O8—K1^{iii}	0.0
O4—S1—O5—K1^{v}	32.5 (2)	O6^{iv}—K1—O8—K1^{iii}	−81.50 (5)
C6—S1—O5—K1^{v}	−83.23 (18)	O7—K1—O8—K1^{iii}	−133.31 (7)
O5—S1—O6—K1	99.55 (11)	K1^{iv}—K1—O8—K1^{iii}	−114.18 (4)
O4—S1—O6—K1	−132.59 (10)	C6—C1—C2—C3	−0.4 (3)
C6—S1—O6—K1	−17.64 (13)	C1—C2—C3—O1	−179.40 (19)
O5—S1—O6—K1^{iv}	−85.42 (11)	C1—C2—C3—C4	−0.2 (3)
O4—S1—O6—K1^{iv}	42.44 (11)	O1—C3—C4—C5	179.45 (17)
C6—S1—O6—K1^{iv}	157.38 (8)	C2—C3—C4—C5	0.3 (3)
O2^{i}—K1—O6—S1	49.90 (12)	O1—C3—C4—C7	−0.2 (3)
O5^{ii}—K1—O6—S1	125.43 (11)	C2—C3—C4—C7	−179.40 (19)
O8—K1—O6—S1	−72.93 (12)	C3—C4—C5—C6	0.2 (3)
O9—K1—O6—S1	−18.31 (14)	C7—C4—C5—C6	179.90 (17)
O8^{iii}—K1—O6—S1	−130.24 (10)	C4—C5—C6—C1	−0.8 (3)
O6^{iv}—K1—O6—S1	175.52 (15)	C4—C5—C6—S1	179.07 (13)
O7—K1—O6—S1	116.07 (12)	C2—C1—C6—C5	0.9 (3)
K1^{iii}—K1—O6—S1	−95.39 (11)	C2—C1—C6—S1	−178.97 (16)
K1^{iv}—K1—O6—S1	175.52 (15)	O5—S1—C6—C5	134.71 (15)
O2^{i}—K1—O6—K1^{iv}	−125.61 (5)	O6—S1—C6—C5	−104.47 (16)
O5^{ii}—K1—O6—K1^{iv}	−50.09 (8)	O4—S1—C6—C5	13.98 (18)
O8—K1—O6—K1^{iv}	111.55 (6)	O5—S1—C6—C1	−45.39 (18)
O9—K1—O6—K1^{iv}	166.17 (5)	O6—S1—C6—C1	75.43 (17)
O8^{iii}—K1—O6—K1^{iv}	54.24 (9)	O4—S1—C6—C1	−166.12 (15)
O6^{iv}—K1—O6—K1^{iv}	0.0	K1^{i}—O2—C7—O3	−5.6 (4)
O7—K1—O6—K1^{iv}	−59.44 (5)	K1^{i}—O2—C7—C4	174.65 (14)
K1^{iii}—K1—O6—K1^{iv}	89.09 (5)	C5—C4—C7—O2	178.52 (18)
O2^{i}—K1—O8—K1^{iii}	122.25 (5)	C3—C4—C7—O2	−1.8 (3)
O5^{ii}—K1—O8—K1^{iii}	10.60 (10)	C5—C4—C7—O3	−1.2 (3)
O6—K1—O8—K1^{iii}	−148.71 (5)	C3—C4—C7—O3	178.45 (17)
O9—K1—O8—K1^{iii}	73.01 (5)		

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (ii) *x*, *y*−1, *z*; (iii) −*x*−1, −*y*, −*z*+2; (iv) −*x*, −*y*, −*z*+2; (v) *x*, *y*+1, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

O1—H1B···O2	0.840 (10)	1.839 (18)	2.586 (2)	147 (3)
O3—H3A···O9^{i}	0.845 (10)	1.838 (11)	2.674 (2)	170 (2)
O7—H7A···O6^{iv}	0.847 (10)	2.092 (13)	2.917 (2)	164 (3)
O7—H7B···O4^{ii}	0.843 (10)	2.021 (12)	2.852 (2)	168 (2)
O8—H8A···O5^{vi}	0.862 (10)	2.55 (3)	2.926 (2)	108 (2)
O8—H8A···O7^{iv}	0.862 (10)	2.50 (2)	2.997 (2)	117.9 (18)
O8—H8B···O4^{vii}	0.870 (10)	2.037 (11)	2.879 (2)	163 (2)
O9—H9A···O3^{i}	0.847 (9)	1.945 (16)	2.674 (2)	144 (2)
O9—H9B···O7^{vii}	0.859 (10)	1.910 (11)	2.763 (2)	172 (2)

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

Fig. 1

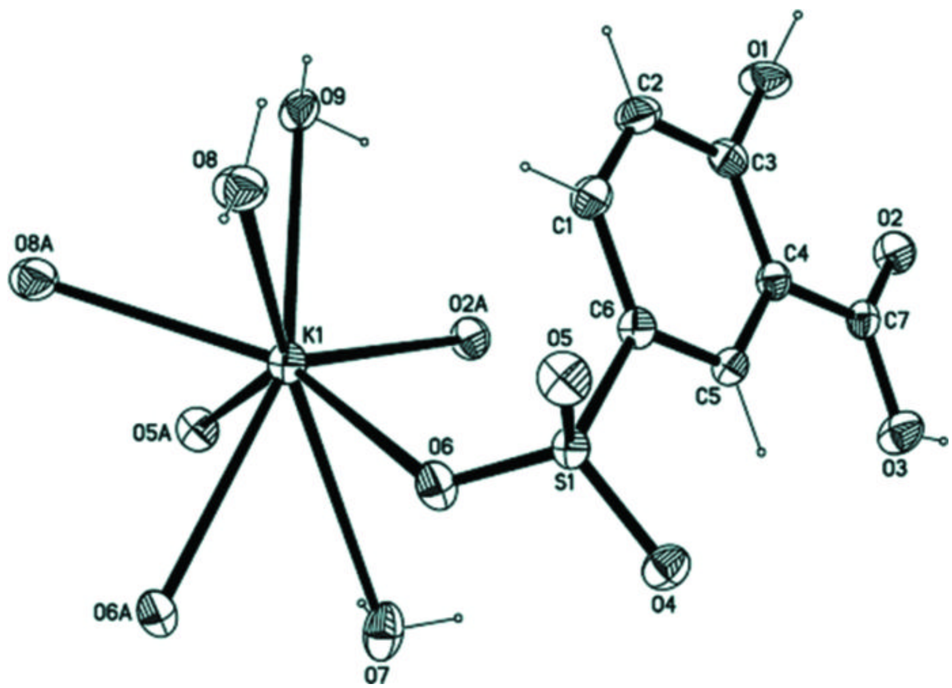

Fig. 2

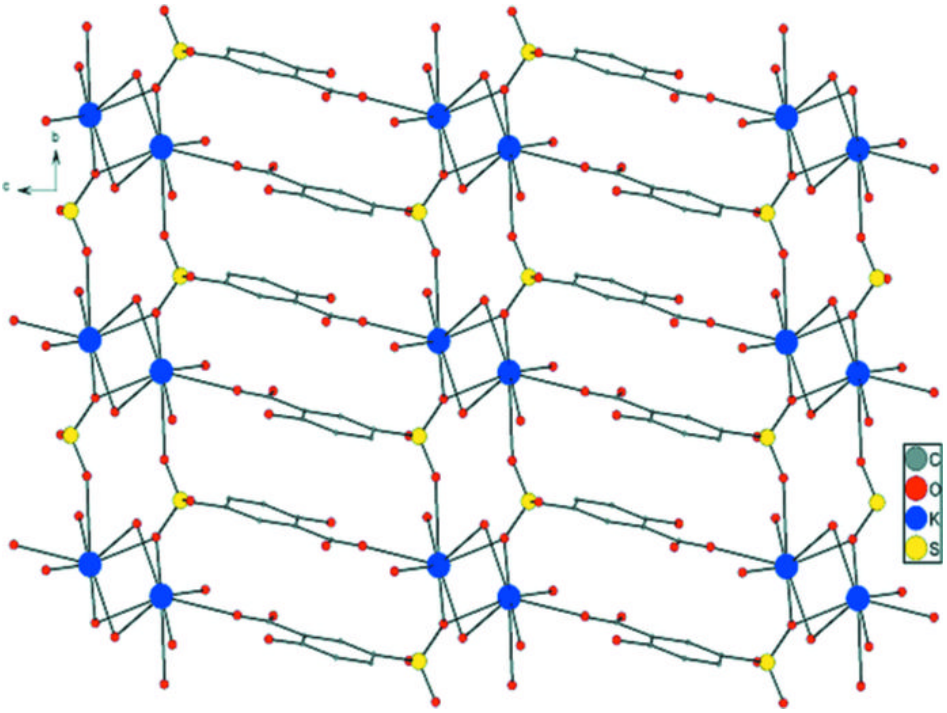

Fig. 3