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Dipotassium 4,4′-(hexane-3,4-diyl)bis(benzenesulfonate) dihydrate

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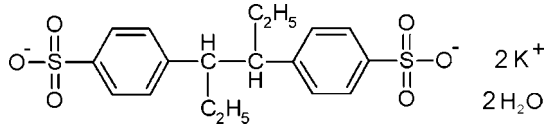
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.052; wR factor = 0.122; data-to-parameter ratio = 14.0.

The anion of the title compound, also called sygethin dihydrate, $2K^+\cdot C_{18}H_{20}O_6S_2^{2-}\cdot 2H_2O$, has crystallographic inversion symmetry. The K^+ cation is surrounded by eight O atoms in a distorted cubic coordination geometry, forming extended K—O—S networks. There are also O—H···O hydrogen bonds.

Related literature

For the synthesis, see: Torf & Khromov-Borisov (1961). For general background, see: Svergun (1979). For a related structure, see: Weeks *et al.* (1973).



Experimental

Crystal data

$2K^+\cdot C_{18}H_{20}O_6S_2^{2-}\cdot 2H_2O$
M_r = 255.36
Triclinic, $P\overline{1}$
a = 5.8741 (5) Å
b = 6.5684 (5) Å
c = 15.2335 (14) Å
α = 84.272 (4)°
β = 83.768 (5)°
γ = 76.522 (6)°
V = 566.51 (8) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.64 mm^{-1}
T = 298 K
0.27 × 0.19 × 0.14 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
4330 measured reflections
2576 independent reflections
1918 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.122
S = 0.89
1918 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Selected bond lengths (Å).

K14—O9^{i}	2.733 (3)	K14—O9	2.934 (3)
K14—O7^{ii}	2.736 (3)	K14—O15	2.937 (3)
K14—O15^{ii}	2.816 (3)	K14—O8^{iii}	2.970 (3)
K14—O7^{iii}	2.834 (3)	K14—O7	3.211 (3)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O15—H5···O8^{iv}	0.84	2.00	2.790 (2)	156

Symmetry code: (iv) $-x, -y+1, -z+1$.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2208).

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Dipotassium 4,4′-(hexane-3,4-diyl)bis(benzenesulfonate) dihydrate

Liana Orola, Mikelis V. Veidis, Sergey Belyakov and Andris Actins

S1. Comment

The synthesis has been described by Torf & Khromov-Borisov (1961). Replacement of the two OH groups of the estrogen hexestrol molecule with two KSO_3 groups results in the formation of the dipotassium salt of 4,4′-(1,2-diethyl-1,2-ethanediyl)bis(benzenesulfonic acid), also known as sygethin. Although the placement of carbon atoms in sygethin is very similar to hexestrol (Weeks *et al.*, 1973) sygethin does not show estrogen-type activity (Svergun, 1979).

The crystal structure of the title compound has been determined. Fig. 1 illustrates the structure. The anion is located on a center of symmetry. The unit cell contains one sygethin anion, two potassium cations and two water molecules.

The packing diagram, Fig. 2, indicates that there are eight oxygen atoms coordinated to the potassium ion in a very distorted cubic geometry: six oxygen atoms are from four sygethin SO_3 ions and two oxygen atoms are from the two water molecules. A hydrogen bond is formed by each water molecule and sygethin.

S2. Experimental

The title compound was supplied by Grindeks Company. To grow crystals suitable for single-crystal diffraction study, the powder form of sygethin dihydrate was dissolved in water at 333 K to obtain a saturated solution. After filtration, the saturated solution was diluted with approximately 50% more water and allowed to crystallize in a petri dish at ambient temperature.

S3. Refinement

The hydrogen atoms were all located in a difference Fourier map. Hydrogen atoms attached to carbon atoms were repositioned goemetrically. During refinement, hydrogen atoms were constrained to the riding mode. U_{iso}(H)=xU_{eq}(C,*O*), where the average values of x are 1.66 for H atoms of the methyl group, 1.2 to 1.29 for H atoms attached to the remaining C atom, and 1.41 for the H atoms of the water molecule.

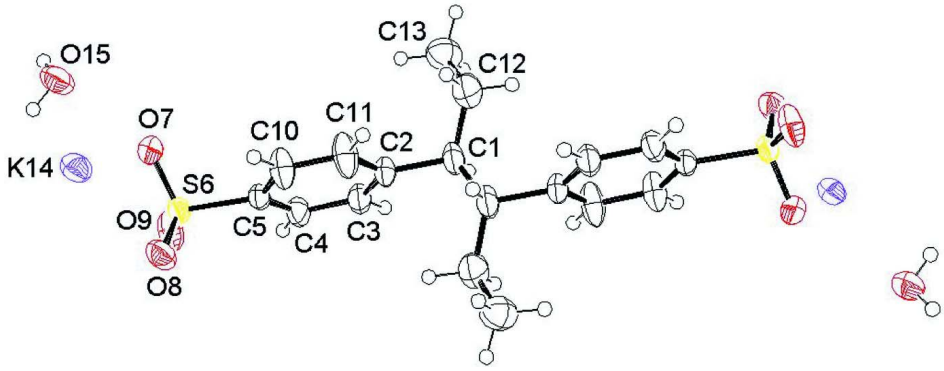

Figure 1

The structure of the title compound, with displacement ellipsoids at the 50% probability level. [Symmetry code for unlabeled atoms: 1-x, -y, -z.]

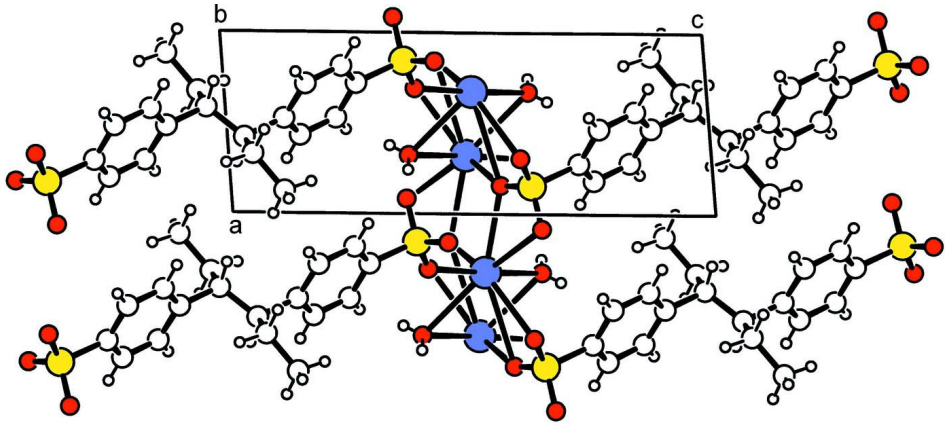

Figure 2

Packing diagram of the title compound.

Dipotassium 4,4′-(hexane-3,4-diyl)bis(benzenesulfonate) dihydrate

Crystal data

$2K^{+}\cdot C_{18}H_{20}O_6S_2^{2-}\cdot 2H_2O$
$M_r = 255.36$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.8741\ (5)$ Å
$b = 6.5684\ (5)$ Å
$c = 15.2335\ (14)$ Å
$\alpha = 84.272\ (4)°$
$\beta = 83.768\ (5)°$
$\gamma = 76.522\ (6)°$
$V = 566.51\ (8)$ Å^3
$Z = 1$
$F(000) = 266$
$D_x = 1.497$ Mg m^{-3}
$D_m =$ Mg m^{-3}
D_m measured by ?
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2576 reflections
$\theta = 1.4–27.4°$
$\mu = 0.64$ mm^{-1}
$T = 298$ K
Prism, colourless
0.27 × 0.19 × 0.14 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
φ and ω scans
4330 measured reflections

2576 independent reflections
1918 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.4°$, $\theta_{min} = 1.4°$

$h = -7 \rightarrow 7$
$k = -8 \rightarrow 8$
$l = -18 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.122$
$S = 0.89$
1918 reflections
137 parameters
0 restraints

H-atom parameters constrained
$w = [1-(F_o-F_c)^2/36\sigma^2(F)]^2/[53.8T_0(x) + 84.3T_1(x) + 51.6T_2(x) + 20.0T_3(x) + 5.48T_4(x)]$ where T_i are Chebychev polynomials and x = F_c/F_{max}
$(\Delta/\sigma)_{max} = 0.000280$
$\Delta\rho_{max} = 1.49$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5804 (7)	−0.0364 (6)	0.0370 (2)	0.0454
C2	0.4756 (6)	0.0667 (5)	0.1221 (2)	0.0398
C3	0.5685 (7)	0.2183 (5)	0.1527 (2)	0.0455
C4	0.4714 (7)	0.3172 (5)	0.2292 (2)	0.0431
C5	0.2797 (5)	0.2595 (5)	0.27676 (19)	0.0324
S6	0.15644 (14)	0.37197 (12)	0.37713 (5)	0.0329
O7	0.1593 (5)	0.1938 (4)	0.44237 (15)	0.0503
O8	−0.0830 (4)	0.4813 (4)	0.36269 (17)	0.0476
O9	0.3068 (5)	0.5041 (5)	0.39615 (18)	0.0554
C10	0.1865 (7)	0.1054 (7)	0.2483 (2)	0.0490
C11	0.2858 (7)	0.0092 (8)	0.1721 (3)	0.0564
C12	0.6723 (9)	−0.2695 (7)	0.0533 (3)	0.0589
C13	0.8749 (8)	−0.3309 (7)	0.1140 (3)	0.0603
K14	0.67561 (14)	0.20792 (11)	0.48854 (5)	0.0427
O15	0.3244 (5)	0.1011 (4)	0.62693 (19)	0.0594
H11	0.7195	0.0235	0.0172	0.0592*
H31	0.7003	0.2542	0.1201	0.0587*
H41	0.5361	0.4240	0.2493	0.0543*
H101	0.0552	0.0653	0.2817	0.0628*
H111	0.2199	−0.0957	0.1524	0.0748*
H121	0.7280	−0.3309	−0.0033	0.0684*
H122	0.5410	−0.3272	0.0825	0.0689*
H131	0.9207	−0.4839	0.1192	0.0894*
H132	1.0067	−0.2743	0.0867	0.0891*
H133	0.8213	−0.2757	0.1713	0.0894*
H5	0.2249	0.2110	0.6406	0.0828*
H13	0.4165	0.0717	0.6665	0.0829*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.053 (2)	0.050 (2)	0.0306 (16)	−0.0163 (14)	0.0018 (14)	−0.0036 (16)

C2	0.0498 (19)	0.0424 (18)	0.0251 (14)	−0.0110 (13)	−0.0015 (12)	−0.0038 (15)
C3	0.066 (2)	0.0375 (17)	0.0340 (16)	−0.0099 (13)	0.0122 (15)	−0.0186 (17)
C4	0.062 (2)	0.0350 (16)	0.0350 (16)	−0.0118 (13)	0.0071 (15)	−0.0172 (16)
C5	0.0360 (15)	0.0342 (15)	0.0247 (13)	−0.0073 (11)	−0.0044 (11)	−0.0006 (13)
S6	0.0364 (4)	0.0328 (4)	0.0272 (4)	−0.0097 (3)	−0.0024 (3)	0.0000 (3)
O7	0.0666 (17)	0.0453 (14)	0.0291 (12)	−0.0015 (10)	0.0008 (11)	0.0045 (13)
O8	0.0413 (14)	0.0446 (14)	0.0498 (14)	−0.0112 (11)	−0.0066 (11)	0.0089 (11)
O9	0.0584 (17)	0.0668 (18)	0.0492 (15)	−0.0335 (13)	0.0058 (12)	−0.0235 (14)
C10	0.0441 (19)	0.068 (2)	0.0424 (18)	−0.0273 (17)	0.0078 (15)	−0.0226 (18)
C11	0.053 (2)	0.080 (3)	0.049 (2)	−0.039 (2)	0.0069 (16)	−0.032 (2)
C12	0.073 (3)	0.055 (2)	0.049 (2)	−0.0125 (18)	0.0002 (19)	−0.014 (2)
C13	0.062 (3)	0.054 (2)	0.066 (3)	−0.005 (2)	−0.016 (2)	−0.010 (2)
K14	0.0488 (5)	0.0328 (4)	0.0463 (4)	−0.0084 (3)	−0.0041 (3)	−0.0066 (3)
O15	0.0713 (19)	0.0425 (14)	0.0553 (16)	−0.0111 (12)	−0.0175 (14)	0.0138 (13)

Geometric parameters (Å, º)

C1—C1^{i}	1.518 (7)	C11—H111	0.952
C1—C2	1.525 (4)	C12—C13	1.542 (6)
C1—C12	1.505 (6)	C12—H121	0.980
C1—H11	0.993	C12—H122	0.982
C2—C3	1.382 (5)	C13—H131	0.975
C2—C11	1.386 (5)	C13—H132	0.972
C3—C4	1.393 (4)	C13—H133	0.971
C3—H31	0.938	O15—H5	0.844
C4—C5	1.380 (5)	O15—H13	0.832
C4—H41	0.961	K14—O9^{ii}	2.733 (3)
C5—S6	1.773 (3)	K14—O7^{iii}	2.736 (3)
C5—C10	1.382 (5)	K14—O15^{iii}	2.816 (3)
S6—O7	1.456 (3)	K14—O7^{iv}	2.834 (3)
S6—O8	1.452 (2)	K14—O9	2.934 (3)
S6—O9	1.442 (3)	K14—O15	2.937 (3)
C10—C11	1.383 (5)	K14—O8^{iv}	2.970 (3)
C10—H101	0.952	K14—O7	3.211 (3)
C1^{i}—C1—C2	111.4 (4)	O7—S6—O9	112.47 (18)
C1^{i}—C1—C12	116.9 (4)	O8—S6—O9	114.94 (17)
C2—C1—C12	112.1 (3)	C5—C10—C11	119.9 (3)
C1^{i}—C1—H11	104.1	C5—C10—H101	119.6
C2—C1—H11	104.9	C11—C10—H101	120.5
C12—C1—H11	106.2	C2—C11—C10	121.4 (3)
C1—C2—C3	121.3 (3)	C2—C11—H111	119.0
C1—C2—C11	121.1 (3)	C10—C11—H111	119.6
C3—C2—C11	117.6 (3)	C1—C12—C13	114.2 (4)
C2—C3—C4	121.9 (3)	C1—C12—H121	109.5
C2—C3—H31	117.9	C13—C12—H121	108.1
C4—C3—H31	120.2	C1—C12—H122	106.9
C3—C4—C5	119.1 (3)	C13—C12—H122	107.6

C3—C4—H41	121.1	H121—C12—H122	110.5
C5—C4—H41	119.8	C12—C13—H131	106.9
C4—C5—S6	121.1 (2)	C12—C13—H132	109.2
C4—C5—C10	120.0 (3)	H131—C13—H132	109.6
S6—C5—C10	118.9 (3)	C12—C13—H133	109.4
C5—S6—O7	104.86 (14)	H131—C13—H133	111.4
C5—S6—O8	106.39 (14)	H132—C13—H133	110.4
O7—S6—O8	110.82 (17)	H5—O15—H13	106.5
C5—S6—O9	106.55 (15)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O15—H5···O8^{v}	0.84	2.00	2.790 (2)	156

Symmetry code: (v) −*x*, −*y*+1, −*z*+1.