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Hydrogen trisodium bis(5-sulfonatosalicylate) monohydrate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.030
wR factor = 0.089
Data-to-parameter ratio = 13.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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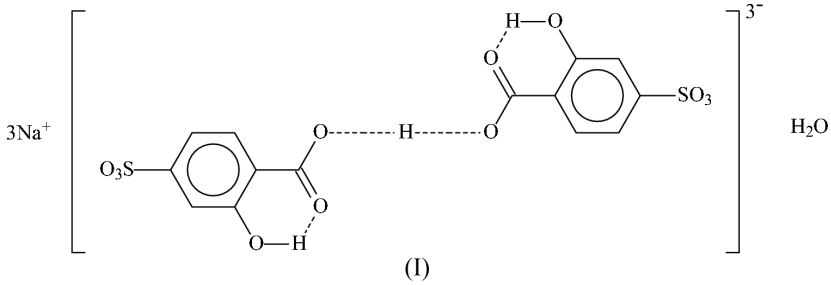
The crystal structure of the title compound, $3Na^{+}\cdot H^{+}\cdot 2C_7H_4O_6S^{2-}\cdot H_2O$, features a trianion in which two 5-sulfonatosalicylate units are linked by an acid H atom through the negatively charged carboxyl O atoms. The trianion lies on a center of inversion, as does one of the sodium ions.

Comment

The sodium salt of 5-sulfosalicylic (3-carboxy-4-hydroxybenzenesulfonic) acid crystallizes as $Na[C_7H_5SO_6]\cdot 2H_2O$ (Aliev & Atovmyan, 2001; Marzotto *et al.*, 2001, 2003), in which only the sulfonic acid group is deprotonated, as well as $Na_2[C_7H_4SO_6]\cdot 3H_2O$ (Cai *et al.*, 1983; Su & Li, 1991), in which both the sulfonic and carboxylic acid groups are deprotonated. The parent acid itself exists in four hydrated forms (Marzotto *et al.*, 2001).



(I)

The title sodium salt, (I), has as the anionic entity two 5-sulfonatosalicylate units that are linked together through an 'acid' H atom; this atom occupies a center of inversion in the crystal structure (Fig. 1) and links the units through their negatively charged carboxyl O atoms. The nature of such acid H atoms has been previously discussed in detail (Gao *et al.*, 2004, 2004*a*,*b*). One of the two independent Na^{+} ions lies on another inversion site; both exist in octahedral environments (Figs. 2 and 3). The monorubidium derivative of 5-sulfosalicylic acid exists as the monohydrate (Hu *et al.*, 2005).

Experimental

The title complex was synthesized by neutralizing 5-sulfosalicylic acid (0.22 g, 1 mmol) with aqueous sodium hydroxide (0.08 g, 2 mmol). The solution was set aside for a week to afford prismatic crystals. Analysis calculated for $C_{14}H_{11}Na_3O_{13}S_2$: C 32.31, H 2.13%; found: C 32.33, H 2.48%.

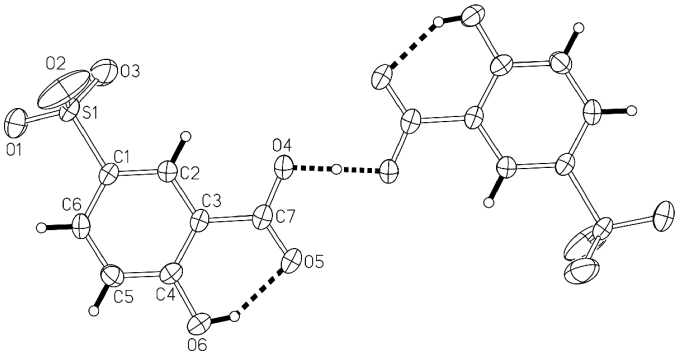


Figure 1
*ORTEP*II (Johnson, 1976) plot of the hydrogen bis(5-sulfonatosalicylate) trianion; displacement ellipsoids are drawn at the 70% probability level and H atoms are shown as spheres of arbitrary radii. The 'acid' H atom lies at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. Dashed lines indicate hydrogen bonds.

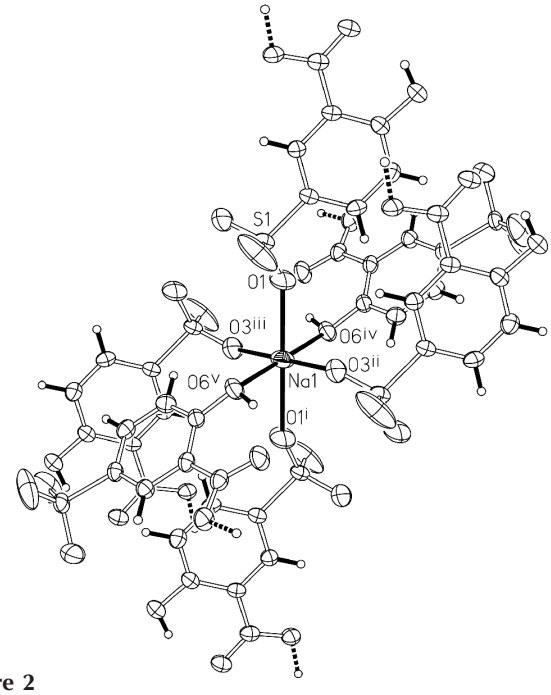


Figure 2
*ORTEP*II (Johnson, 1976) plot illustrating the geometry around the Na1 ion. Symmetry codes are as given in Table 1.

Crystal data

$3Na^+ \cdot H^+ \cdot 2C_7H_4O_6S^{2-} \cdot H_2O$	$D_x = 1.936$ Mg m^{-3}
$M_r = 520.32$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 7762 reflections
$a = 22.563$ (5) Å	$\theta = 3.6$–$27.5°$
$b = 5.464$ (1) Å	$\mu = 0.45$ mm^{-1}
$c = 17.901$ (4) Å	$T = 295$ (2) K
$\beta = 126.02$ (3)°	Block, colorless
$V = 1785.1$ (6) Å^3	0.38 × 0.29 × 0.22 mm
$Z = 4$	

Data collection

Rigaku R-AXIS RAPID diffractometer	2034 independent reflections
	1889 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.014$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$\theta_{max} = 27.5°$
	$h = -28 \rightarrow 28$
$T_{min} = 0.788$, $T_{max} = 0.908$	$k = -7 \rightarrow 7$
8215 measured reflections	$l = -23 \rightarrow 23$

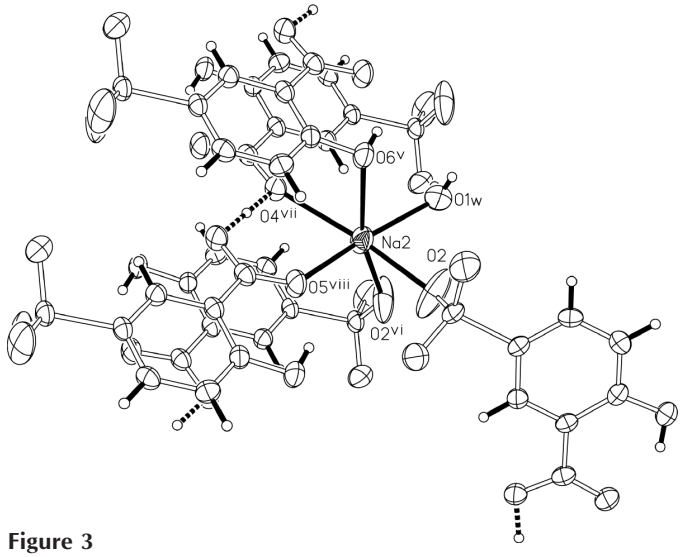


Figure 3
*ORTEP*II (Johnson, 19976) plot illustrating the geometry around the Na2 ion. Symmetry codes are as given in Table 1.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 2.0813P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.030$	
$wR(F^2) = 0.089$	
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
2034 reflections	$\Delta\rho_{max} = 0.45$ e Å^{-3}
156 parameters	$\Delta\rho_{min} = -0.37$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.430 (1)	Na2—O5^{viii}	2.330 (1)
S1—O2	1.453 (1)	Na2—O6^{v}	2.715 (2)
S1—O3	1.442 (1)	Na2—O1*w*	2.412 (2)
S1—C1	1.764 (2)	O4—C7	1.286 (2)
Na1—O1	2.280 (1)	O5—C7	1.243 (2)
Na1—O1^{i}	2.280 (1)	O6—C4	1.362 (2)
Na1—O3^{ii}	2.533 (2)	C1—C2	1.385 (2)
Na1—O3^{iii}	2.533 (2)	C1—C6	1.392 (2)
Na1—O6^{iv}	2.561 (1)	C2—C3	1.393 (2)
Na1—O6^{v}	2.561 (1)	C3—C4	1.405 (2)
Na2—O2	2.347 (2)	C3—C7	1.489 (2)
Na2—O2^{vi}	2.456 (2)	C4—C5	1.392 (2)
Na2—O4^{vii}	2.589 (1)	C5—C6	1.382 (2)
O1—S1—O2	113.3 (1)	C2—C1—S1	118.5 (1)
O1—S1—O3	113.9 (1)	C6—C1—S1	120.8 (1)
O1—S1—C1	106.8 (1)	C2—C1—C6	120.5 (1)
O2—S1—O3	111.2 (1)	C1—C2—C3	120.4 (1)
O2—S1—C1	104.2 (1)	C2—C3—C4	118.8 (1)
O3—S1—C1	106.6 (1)	C2—C3—C7	119.9 (1)
O1—Na1—O1^{i}	180	C4—C3—C7	121.3 (1)
O1—Na1—O3^{ii}	89.5 (1)	O6—C4—C5	117.3 (1)
O1—Na1—O3^{iii}	90.5 (1)	O6—C4—C3	122.3 (1)
O1—Na1—O6^{iv}	79.3 (1)	C3—C4—C5	120.4 (1)
O1—Na1—O6^{v}	100.7 (1)	C4—C5—C5	120.2 (1)
O3^{ii}—Na1—O3^{iii}	180	C1—C6—C5	119.7 (1)
O3^{ii}—Na1—O6^{v}	67.8 (1)	O5—C7—O4	123.4 (1)
O3^{ii}—Na1—O6^{iv}	112.2 (1)	O5—C7—C3	119.7 (1)
O6^{iv}—Na1—O6^{v}	180	O4—C7—C3	116.9 (1)

Symmetry codes: (i) $\frac{1}{2}-x, \frac{7}{2}-y, -z$; (ii) $x, 1+y, z$; (iii) $\frac{1}{2}-x, \frac{5}{2}-y, -z$; (iv) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (v) $x, 3-y, z-\frac{1}{2}$; (vi) $1-x, y, \frac{1}{2}-z$; (vii) $1-x, 1+y, \frac{1}{2}-z$; (viii) $x, 2-y, z-\frac{1}{2}$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4o···O4^{ix}	1.238	1.238	2.475 (2)	180
O6—H6o···O5	0.85 (1)	1.87 (2)	2.615 (2)	147 (2)
O1w—H1w···O2^{vii}	0.85 (1)	2.51 (3)	3.200 (3)	139 (4)
O1w—H1w···O3^{ii}	0.85 (1)	2.29 (2)	3.107 (2)	162 (5)

Symmetry codes: (vii) $1-x, 1+y, \frac{1}{2}-z$; (ix) $1-x, 1-y, 1-z$.

The aromatic H atoms were placed at calculated positions [C—H = 0.93 Å and U_{iso}(H) 1.2U_{eq}(C)] and were included in the refinement in the riding-model approximation. The water and hydroxyl H atoms were located in a difference Fourier map and were refined with a distance restraint of 0.85 (1) Å. Since the 'acid' H atom is located on a center of inversion, only its U_{iso} value was refined.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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