

Sodium 1-Naphthylamine-6-sulfonate–Water (1/4)

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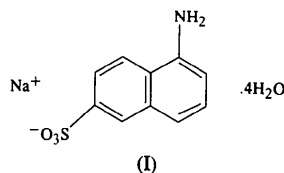
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Abstract

A feature of the title compound, sodium 5-amino-2-naphthalenesulfonate–water (1/4), Na⁺.C₁₀H₈NO₃S⁻.4H₂O, is that the Na⁺ ions are coordinated by distorted edge-sharing octahedra of water O atoms. The coordination distances around Na⁺ vary from 2.217 (8) to 2.80 (1) Å.

Comment

Brown & Corbridge (1966) were first to determine the crystal data for naphthylaminesulfonic acid. We have reported previously the crystal structures of 1-naphthylamine-6-sulfonic acid and its dihydrate (Miao, Wang & Li, 1993) and magnesium(II) 1-naphthylamine-6-sulfonate hexahydrate (Wang, Li & Miao, 1993). We report here the crystal structure of the title compound, (I), as the third part of this study.



The structure of (I) is composed of [Na(H₂O)₄]⁺_∞ cation chains and C₁₀H₈NO₃S⁻ anions. The Na⁺ ions are surrounded by distorted edge-sharing octahedra of water O atoms, as is also found in both Na⁺.C₆H₉O₇⁻.H₂O (Bouwman, Peters, van Bekkum & Gorter, 1991) and Na⁺.C₅H₇O₄⁻.2H₂O (Macdonald, Morrison, Murray & Freer, 1991). The Na···O distances vary from 2.217 (8) to 2.80 (1) Å and the nonlinear O···Na···O angles vary from 73.4 (1) to 102.9 (3)°, values which are close to those reported previously (Minemoto, Sonoda & Miki, 1992; Fitzgerald, Gallucci & Gerkin, 1991; Blackburn, Gallucci, Gerkin & Repart, 1992). Bond lengths and angles in the anion are within the expected ranges. There are several hydrogen bonds associated with the crystal packing. One hydrogen bond connects the Na(H₂O)₄⁺ ions with the amine N atom of the anion [O(6)—H(61)···N(1)], while the others connect the water molecules with the sulfonate O atoms of the anion (Table 3).

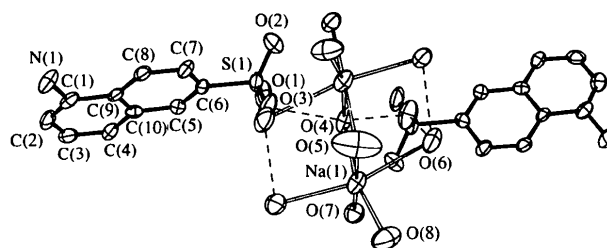


Fig. 1. The molecular structure of (I) with the atomic numbering and 50% probability displacement ellipsoids. A twofold axis passes through the O(4) and O(5) atoms.

Experimental

The title compound was prepared by reaction of Na₂CO₃ with 1-naphthylamine-6-sulfonic acid in aqueous solution and was isolated as a colourless salt.

Crystal data

Na⁺.C₁₀H₈NO₃S⁻.4H₂O

M_r = 317.30

Monoclinic

C2/c

a = 28.787 (4) Å

b = 8.619 (3) Å

c = 11.623 (5) Å

β = 95.64 (2)°

V = 2869.9 (7) Å³

Z = 8

D_x = 1.468 Mg m⁻³

D_m = 1.50 Mg m⁻³

D_m measured by pycnometry

Mo Kα radiation

λ = 0.71073 Å

Cell parameters from 25

reflections

θ = 5.69–10.96°

μ = 0.2712 mm⁻¹

T = 298 K

Prism

0.25 × 0.20 × 0.20 mm

Colourless

Data collection

Enraf–Nonius diffractometer

ω/2θ scans

Absorption correction:

empirical *via* ψ scans

(Walker & Stuart, 1983)

T_{min} = 0.974, *T_{max}* =

0.999

3079 measured reflections

2928 independent reflections

1163 observed reflections

[|*F_o*| > 3σ(*F_o*)]

R_{int} = 0.025

θ_{max} = 23°

h = 0 → 34

k = 0 → 9

l = -13 → 13

3 standard reflections

monitored every 400

reflections

intensity decay: 3.98%

Refinement

Refinement on *F*

R = 0.050

wR = 0.058

S = 0.815

1163 reflections

231 parameters

H-atom coordinates refined

with *B_{iso}* = 4.0 Å²

w = 0 if *F_o* ≤ 3σ(*F_o*),

w = 1.0 if *F_o* < 48 and

w = (48/*F_o*)² if *F_o* ≥ 48

(Δ/σ)_{max} = 0.02

Δρ_{max} = 0.329 e Å⁻³

Δρ_{min} = -0.297 e Å⁻³

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Na(1)	0.4734 (1)	0.3787 (4)	0.1169 (2)	0.041 (1)
S(1)	0.10053 (7)	0.3722 (3)	0.2621 (2)	0.036 (1)
O(1)	0.0818 (2)	0.5065 (7)	0.2113 (6)	0.064 (8)
O(2)	0.0962 (2)	0.3753 (8)	0.3854 (4)	0.062 (1)
O(3)	0.0822 (2)	0.2324 (7)	0.2152 (6)	0.063 (2)
O(4)	1/2	0.5673 (8)	1/4	0.040 (2)
O(5)	1/2	0.1900 (8)	1/4	0.046 (2)
O(6)	0.4055 (2)	0.4072 (7)	0.2721 (5)	0.054 (1)
O(7)	0.4491 (2)	0.6008 (6)	-0.0094 (4)	0.048 (1)
O(8)	0.4350 (2)	0.1853 (7)	0.0245 (5)	0.067 (2)
N(1)	0.3334 (2)	0.2394 (8)	0.1508 (6)	0.051 (2)
C(1)	0.3037 (2)	0.3347 (8)	0.0953 (5)	0.034 (2)
C(2)	0.3203 (3)	0.4307 (9)	0.0088 (6)	0.038 (2)
C(3)	0.2915 (3)	0.5348 (9)	-0.0431 (6)	0.042 (2)
C(4)	0.2449 (3)	0.5431 (8)	-0.0058 (6)	0.033 (1)
C(5)	0.1782 (3)	0.4572 (8)	0.1257 (6)	0.028 (2)
C(6)	0.1604 (2)	0.3674 (8)	0.2136 (5)	0.025 (2)
C(7)	0.1882 (2)	0.2590 (8)	0.2674 (6)	0.032 (2)
C(8)	0.2347 (3)	0.2495 (8)	0.2298 (6)	0.029 (2)
C(9)	0.2548 (2)	0.3429 (7)	0.1388 (5)	0.025 (2)
C(10)	0.2264 (3)	0.4468 (7)	0.0841 (5)	0.025 (2)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Na(1)—O(4)	2.323 (7)	C(1)—C(2)	1.42 (1)
Na(1)—O(5)	2.321 (8)	C(1)—C(9)	1.54 (1)
Na(1)—O(6)	2.80 (1)	C(2)—C(3)	1.33 (1)
Na(1)—O(7)	2.473 (7)	C(3)—C(4)	1.45 (1)
Na(1)—O(7')	2.666 (8)	C(4)—C(10)	1.47 (1)
Na(1)—O(8)	2.217 (8)	C(5)—C(6)	1.42 (1)
S(1)—O(1)	1.385 (7)	C(6)—C(7)	1.34 (1)
S(1)—O(2)	1.451 (6)	C(7)—C(8)	1.45 (1)
S(1)—O(3)	1.405 (7)	C(8)—C(9)	1.49 (1)
N(1)—C(1)	1.31 (1)	C(9)—C(10)	1.33 (1)
O(1)—S(1)—O(2)	109.7 (5)	C(6)—C(5)—C(10)	127.1 (8)
O(1)—S(1)—O(3)	115.8 (4)	S(1)—C(6)—C(5)	128.0 (6)
O(1)—S(1)—C(6)	102.9 (4)	S(1)—C(6)—C(7)	113.6 (6)
O(2)—S(1)—O(3)	109.5 (5)	C(5)—C(6)—C(7)	118.4 (8)
O(2)—S(1)—C(6)	118.1 (3)	C(6)—C(7)—C(8)	115.0 (8)
O(3)—S(1)—C(6)	100.7 (4)	C(7)—C(8)—C(9)	128.1 (8)
N(1)—C(1)—C(2)	117.7 (9)	C(1)—C(9)—C(8)	129.6 (7)
N(1)—C(1)—C(9)	116.1 (8)	C(1)—C(9)—C(10)	114.4 (7)
C(2)—C(1)—C(9)	125.9 (7)	C(8)—C(9)—C(10)	116.0 (8)
C(1)—C(2)—C(3)	118.4 (9)	C(4)—C(10)—C(5)	126.8 (7)
C(2)—C(3)—C(4)	116.9 (9)	C(4)—C(10)—C(9)	117.9 (8)
C(3)—C(4)—C(10)	126.4 (8)	C(5)—C(10)—C(9)	115.3 (7)

Symmetry code: (i) $1 - x, 1 - y, -z$.Table 3. Hydrogen-bonding geometry (\AA , $^\circ$)

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
O(6)—H(61)···N(1)	0.75	2.11 (6)	2.79 (1)	137.8 (6)
O(4)—H(41)···O(3')	0.75	2.10 (6)	2.82 (1)	161.5 (6)
O(5)—H(51)···O(1 ⁱⁱ)	0.68	2.23 (6)	2.91 (1)	172.2 (6)
O(6)—H(62)···O(3 ⁱⁱⁱ)	0.72	2.26 (7)	2.83 (1)	136.5 (7)
O(7)—H(72)···O(2 ⁱⁱⁱ)	0.96	2.59 (6)	3.12 (1)	170.2 (6)
O(8)—H(82)···O(3 ⁱⁱⁱ)	0.81	2.11 (6)	2.87 (1)	157.4 (5)
O(8)—H(81)···O(2 ⁱⁱⁱ)	0.78	2.27 (6)	3.04 (1)	168.9 (6)

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

The title structure was solved by direct methods. Full-matrix least squares were used for refinement on *F*, with anisotropic displacement parameters for non-H atoms and isotropic displacement parameters for H atoms. Calculations were performed on a VAX 3100 computer using the *MolEN* (Fair, 1990) program package for structure solution and refinement.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCR (Reference: AB1205). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Chlorotris(*p*-methylsulfonylphenyl)tin(IV)

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Abstract

The title compound, $[\text{SnCl}(\text{C}_7\text{H}_7\text{O}_2\text{S})_3]$, crystallizes in an unusual space group for Ar_3SnCl compounds, namely, *Pna*₂. The structure has $[-\text{O}=\text{S}(\text{CH}_3)(\text{O})-\text{C}_6\text{H}_4(\text{Ar})_2\text{Sn}(\text{Cl})-]_n$ polymer chains zigzagging along the screw axis parallel to *c*, which are then closely packed to create a dense structure, unlike the open lattice found for the *tert*-butyl analogue. The molecular unit has distorted *C*_s symmetry, with one aryl group forming the pseudo-mirror plane. The environment around the Sn atom is quasi-five-coordinate, compared with that found in either four-coordinate monomeric Ph_3SnCl or in five-coordinate $\text{Ph}_3\text{SnCl}\cdot\text{L}$.