

O(1)	0.6238 (4)	-0.2211 (5)	0.9172 (2)	0.0286 (9)
O(2)	0.6164 (3)	0.0576 (4)	1.0490 (2)	0.0194 (8)
C(1)	0.7100 (6)	-0.3169 (7)	0.8662 (3)	0.030 (1)
C(2)	0.8524 (6)	-0.3712 (9)	0.9331 (4)	0.043 (2)
C(3)	0.7432 (5)	0.1563 (7)	1.1058 (3)	0.024 (1)
C(4)	0.8710 (5)	0.0337 (8)	1.1409 (4)	0.032 (1)

Table 2. Selected geometric parameters (Å, °)

Sn(1)—Cl(1)	2.3441 (11)	Sn(1)—O(2')	2.084 (3)
Sn(1)—Cl(2)	2.3947 (12)	O(1)—C(1)	1.459 (6)
Sn(1)—Cl(3)	2.3504 (14)	O(2)—C(3)	1.461 (5)
Sn(1)—O(1)	2.164 (3)	C(1)—C(2)	1.491 (7)
Sn(1)—O(2)	2.092 (3)	C(3)—C(4)	1.501 (7)
Cl(1)—Sn(1)—Cl(2)	96.80 (4)	Cl(3)—Sn(1)—O(2')	164.21 (8)
Cl(1)—Sn(1)—Cl(3)	97.10 (4)	O(1)—Sn(1)—O(2)	84.16 (12)
Cl(1)—Sn(1)—O(1)	87.63 (9)	O(1)—Sn(1)—O(2')	82.11 (12)
Cl(1)—Sn(1)—O(2)	165.43 (9)	O(2)—Sn(1)—O(2')	71.50 (12)
Cl(1)—Sn(1)—O(2')	95.49 (8)	Sn(1)—O(1)—C(1)	129.4 (3)
Cl(2)—Sn(1)—Cl(3)	96.22 (5)	Sn(1)—O(2)—Sn(1')	108.50 (12)
Cl(2)—Sn(1)—O(1)	172.73 (9)	Sn(1)—O(2)—C(3)	124.8 (2)
Cl(2)—Sn(1)—O(2)	90.27 (9)	Sn(1')—O(2)—C(3)	125.5 (2)
Cl(2)—Sn(1)—O(2')	91.71 (9)	O(1)—C(1)—C(2)	110.6 (4)
Cl(3)—Sn(1)—O(1)	88.91 (10)	O(2)—C(3)—C(4)	110.7 (4)
Cl(3)—Sn(1)—O(2)	94.76 (9)		

Symmetry code: (i) 1 - x, -y, 2 - z.

Data collection used a Rigaku AFC-7S four-circle diffractometer equipped with an Oxford Cryosystems low-temperature attachment (Cosier & Glazer, 1986) operating at 150 K. Data collection and cell refinement were performed using *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); *TEXSAN* (Molecular Structure Corporation, 1995) was used for data reduction. As there were no identifiable faces, the data were corrected for absorption using ψ scans. The structure was solved by direct methods (Sheldrick, 1990), which located the positions of all non-H atoms for one half molecule in the asymmetric unit. This is related to the other half of the neutral dinuclear species by a crystallographic inversion centre. Refinement was achieved with iterative cycles of least-squares refinement and difference Fourier synthesis (*TEXSAN*). All non-H atoms were refined anisotropically and H atoms [except for H(1)] were included in fixed calculated positions with C—H = 0.96 Å and $U_{\text{iso}}(\text{H}) = U_{\text{eq}}(\text{parent C atom})$. Atom H(1) was located in the difference map and its coordinates fixed.

The authors thank the University of Southampton for support and the EPSRC for a grant to purchase the diffractometer, and acknowledge the use of the Cambridge Structural Database implemented at CCLRC Daresbury Laboratory.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1062). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Trimethyl[3-(triphenylstannyl)propyl]-ammonium Iodide

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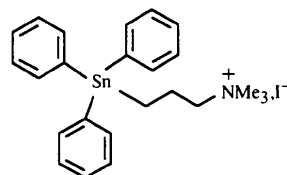
(Received 16 October 1995; accepted 17 January 1996)

Abstract

The title compound, [Sn(C₆H₅)₃(C₆H₁₅N)]I, exists as an ionic species with discrete cations and anions. Both the Sn- and N-atom centres in the cation have tetrahedral geometries.

Comment

The title compound, (I), is an ionic species with discrete cations and anions. All I···C distances are > 3.83 Å, I···N > 4.41 Å, and I···I > 5.09 Å. The geometries about both the Sn and N atoms in the title compound are essentially tetrahedral with C—Sn—C and C—N—C valence angles ranging from 106.2 (3) to 113.3 (3)°, and from 106.7 (5) to 111.6 (5)°, respectively. The



(I)

Sn—C bond lengths fall in a narrow band, between 2.135 (8) and 2.145 (7) Å, and are unexceptional. The aliphatic chain has an extended all-*anti* conformation with torsion angles Sn—C1—C2—C3 = 162.9 (5) and C1—C2—C3—N = 179.0 (6)°. The structure of only

one other stannyl-substituted ammonium halide has been reported previously: $[\text{Me}_3\text{SnCH}_2\text{NMe}_3]^+\text{I}^-$ also contains essentially tetrahedral Sn- and N-atom centres (Cox & Wardell, 1995).

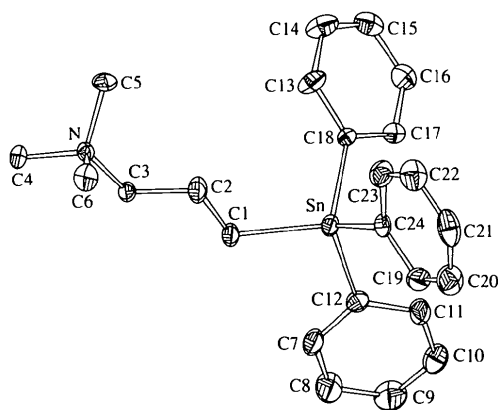


Fig. 1. The cation $[\text{Sn}(\text{C}_6\text{H}_5)_3(\text{C}_6\text{H}_{15}\text{N})]^+$ showing the atom-numbering scheme. Ellipsoids are shown at the 20% probability level and H atoms have been omitted for clarity.

Experimental

The title compound was prepared by the reaction of $\text{Ph}_3\text{SnCH}_2\text{CH}_2\text{CH}_2\text{I}$ with NMe_3 ; it was recrystallized from EtOH and had m.p. 478–481 K.

Crystal data

$[\text{Sn}(\text{C}_6\text{H}_5)_3(\text{C}_6\text{H}_{15}\text{N})]\text{I}$

$M_r = 578.11$

Monoclinic

$P2_1/a$

$a = 18.678(9) \text{ \AA}$

$b = 7.627(3) \text{ \AA}$

$c = 19.099(10) \text{ \AA}$

$\beta = 116.60(4)^\circ$

$V = 2433(2) \text{ \AA}^3$

$Z = 4$

$D_x = 1.578 \text{ Mg m}^{-3}$

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 14 reflections

$\theta = 8.3\text{--}10.8^\circ$

$\mu = 2.32 \text{ mm}^{-1}$

$T = 198 \text{ K}$

Plate

$0.7 \times 0.44 \times 0.08 \text{ mm}$

Colourless

Data collection

Nicolet P3 diffractometer

$\theta/2\theta$ scans

Absorption correction:

ψ scans (North, Phillips & Mathews, 1968)

$T_{\min} = 0.17$, $T_{\max} = 0.42$

4799 measured reflections

4308 independent reflections

3045 observed reflections

$[F > 4\sigma(F)]$

$R_{\text{int}} = 0.024$

$\theta_{\max} = 25^\circ$

$h = -22 \rightarrow 22$

$k = -0 \rightarrow 9$

$l = 0 \rightarrow 22$

2 standard reflections monitored every 50 reflections

intensity decay: 2%

Refinement

Refinement on F

$R = 0.043$

$wR = 0.043$

$(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 1.23 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.58 \text{ e \AA}^{-3}$

$S = 1.430$

3045 reflections

256 parameters

H atoms: see below

$w = 1/[\sigma^2(F) + 0.000641F^2]$

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)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	U_{eq}
Sn	0.20840 (3)	0.16258 (6)	0.19310 (3)	0.0408 (2)
I	0.41694 (3)	-0.86743 (6)	0.56221 (3)	0.0550 (2)
C1	0.2073 (5)	0.0142 (10)	0.2880 (5)	0.056 (2)
C2	0.2594 (5)	-0.1523 (9)	0.3147 (4)	0.053 (2)
C3	0.2684 (4)	-0.1995 (9)	0.3950 (4)	0.037 (2)
N	0.3165 (3)	-0.3621 (6)	0.4326 (3)	0.039 (1)
C4	0.3225 (4)	-0.3730 (10)	0.5135 (4)	0.049 (2)
C5	0.3991 (4)	-0.3510 (12)	0.4396 (6)	0.066 (3)
C6	0.2747 (5)	-0.5219 (10)	0.3886 (5)	0.060 (3)
C7	0.0809 (5)	0.3843 (11)	0.2119 (5)	0.066 (3)
C8	0.0290 (6)	0.5216 (16)	0.2003 (6)	0.100 (5)
C9	0.0121 (7)	0.6336 (13)	0.1401 (7)	0.095 (4)
C10	0.0482 (6)	0.6144 (12)	0.0931 (7)	0.089 (4)
C11	0.1021 (5)	0.4796 (11)	0.1068 (5)	0.065 (3)
C12	0.1195 (4)	0.3624 (9)	0.1661 (4)	0.043 (2)
C13	0.3922 (5)	0.2160 (11)	0.2868 (5)	0.071 (3)
C14	0.4643 (5)	0.3044 (13)	0.3142 (6)	0.084 (3)
C15	0.4687 (5)	0.4671 (13)	0.2896 (6)	0.070 (3)
C16	0.3993 (5)	0.5446 (12)	0.2367 (5)	0.069 (3)
C17	0.3270 (5)	0.4571 (11)	0.2092 (5)	0.061 (3)
C18	0.3222 (4)	0.2910 (9)	0.2343 (4)	0.043 (2)
C19	0.1135 (5)	0.0118 (11)	0.0250 (5)	0.057 (2)
C20	0.0998 (6)	-0.0910 (13)	-0.0378 (5)	0.079 (3)
C21	0.1587 (8)	-0.2016 (14)	-0.0362 (6)	0.088 (4)
C22	0.2305 (7)	-0.2102 (13)	0.0286 (7)	0.086 (4)
C23	0.2446 (5)	-0.1058 (11)	0.0921 (5)	0.069 (3)
C24	0.1868 (4)	0.0066 (9)	0.0928 (4)	0.047 (2)

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

C1—Sn	2.145 (7)	C12—Sn	2.140 (6)
C18—Sn	2.145 (7)	C24—Sn	2.135 (8)
C2—C1	1.541 (9)	C3—C2	1.510 (9)
N—C3	1.511 (8)	C4—N	1.502 (8)
C5—N	1.490 (8)	C6—N	1.487 (9)
C12—Sn—C1	106.2 (3)	C18—Sn—C1	108.3 (3)
C18—Sn—C12	107.4 (3)	C24—Sn—C1	113.3 (3)
C24—Sn—C12	111.5 (3)	C24—Sn—C18	110.0 (3)
C2—C1—Sn	118.1 (5)	C3—C2—C1	107.4 (6)
N—C3—C2	116.7 (5)	C4—N—C3	106.7 (5)
C5—N—C3	111.6 (5)	C5—N—C4	108.3 (6)
C6—N—C3	110.7 (5)	C6—N—C4	108.3 (6)
C6—N—C5	111.0 (6)		

Data collection used 2θ scan rates of $5.33 (I_p < 150)$ to $58.6^\circ \text{ min}^{-1} (I_p > 2500)$, where I_p is the pre-scan intensity. Scan widths were 2.4 to 2.7° in 2θ . All non-H atoms were refined anisotropically. H atoms were placed in calculated positions and refined riding upon the C atoms to which they were attached with common group U_{iso} for methylene, methyl and phenyl H atoms. Methyl groups were treated as rigid bodies. The final difference map contained four peaks of 1.00 and 1.23 e \AA^{-3} , two of them $ca 1 \text{ \AA}$ from Sn and two $ca 1 \text{ \AA}$ from I. All computations were performed on the SUN SPARCserver of the University of Aberdeen.

Data collection and cell refinement: Nicolet P3 software (Nicolet XRD Corporation, 1980). Data reduction: RDNIC (Howie, 1980). Structure solution: SHELXS86 (Sheldrick,

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1055). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Sodium 1,12-Dodecylene Disulfate Hydrate

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Abstract

There are two independent dodecylene disulfate molecules in the crystal of $2\text{Na}^+ \cdot \text{C}_{12}\text{H}_{24}\text{O}_8\text{S}_2^{2-} \cdot 2/3\text{H}_2\text{O}$, one with a centrosymmetric conformation, its center lying on a crystallographic center of symmetry, the second with an asymmetric conformation, which is partially disordered. The molecules form a layer structure parallel to the *ab* plane, the polar parts of neighbouring molecular layers connecting *via* coordination to the Na^+ ion. Additionally, there are hydrogen bonds between the water molecule and two sulfate groups.

Comment

The crystal structures of sodium dodecyl sulfate hydrate with varying water content have been reported by sev-

eral authors (Sundell, 1977; Coiro, Mazza & Pochetti, 1986; Coiro, Manigrasso, Mazza & Pochetti, 1987). The role of head groups in the structural arrangement can be demonstrated by a comparison of the crystal structures of sodium dodecyl sulfate and sodium dodecylene disulfate. The interactions within the layer planes as a function of the number of sulfate groups of both amphiphilic compounds are of interest with respect to the characterization of adsorption layers at the interfaces. This is the first example of a crystal structure determination of sodium 1,12-dodecylene disulfate hydrate, (I).

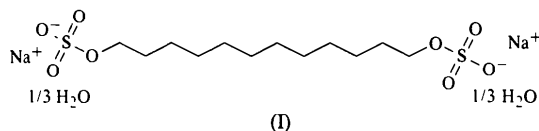


Fig. 1 shows the asymmetric unit together with the atomic numbering scheme. One disulfate molecule is centrosymmetric with the center of the molecule situated at a crystallographic inversion center. The second molecule, which has an asymmetric conformation, is in a general position and has disorder of every second C atom (*i.e.* atoms with numbers ending in 2, 4, 6...12) of its hydrocarbon chain and of three O atoms of a sulfate group. The apparently short C—C distances and the wide C—C—C angles of this molecule are probably artefacts caused by the difficulties refining pairs of disordered atoms.

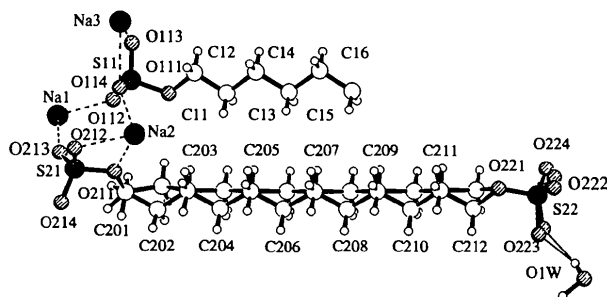


Fig. 1. Drawing of the asymmetric unit. Shaded circles are O atoms, filled circles are S atoms and Na^+ cations, dotted lines are $\text{Na}^+ \cdots \text{O}$ contacts, and thin lines are hydrogen bridges. For the disordered pairs only the labels of one atom are shown.

The hydrocarbon chain in both molecules is planar with the exception of C201 of the disordered molecule. C201 deviates from the least squares plane by 0.102 (5) Å (plane defined by the atoms C201–C212, first-chain orientation) and by 0.257 (5) Å (plane defined by C201, C302, C203–C312, second-chain orientation). The torsion angles O—C—C—C are antiperiplanar for both the ordered molecule and for the C212/C312 side of the disordered molecule and synclinal on the C201 side of the same molecule (this holds for both parts of the disordered pair). This kind of torsion angle is normally antiperiplanar in dodecyl sulfates, but there is