On the Metallo-organic Compounds. XI. Trialkyltin Sulphide, $R_3SnSSnR_3$.

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Recently Kraus and Sessions[1] have shown that the trimethyltin group combines slowly with sulphur in benzene solution to form trimethyltin sulphide. The method, however, is disadvantageous for the preparation because the process is troublesome and gives a poor yield, therefore, the present investigation was carried out.

Trialkyltin sulphide is easily obtained from alcoholic solution by the following reaction.

$$2\,R_3SnX + Na_2S = R_3SnSSnR_3 + 2\,NaX$$

$$R = \text{alkyl group}, \quad X = \text{halogen}$$

Trialkyltin sulphide is a colourless liquid in general and soluble in ordinary organic solvents but insoluble in water. It reacts with a strong mineral acid, such as hydrochloric acid, to form trialkyltin chloride with evolution of hydrogen sulphide according to the equation:

$$R_3SnS\,SnR_3 + 2\,HCl = 2\,R_3SnCl + H_2S$$

Table. Physical Properties of Trialkyltin Sulphides.

	$((CH_3)_3Sn)_2S$	$((C_2H_5)_3Sn)_2S$	$((n\text{-}C_3H_7)_3Sn)_2S$
b.p.	118^{18mm}	$187-188^{20mm}$	$215-219^{18mm}$
m.p.	7	colourless liquid	colourless liquid
Sp. g. at 35°	1.6392	1.4216	1.2110
R. I. at 18°	1.5592	1.5470	1.5290

(1) *J. Am. Chem. Soc.* **47** (1925), 2361.

Experimental. Trimethyltin Sulphide: Trimethyltin iodide was treated with Na_2S in absolute alcohol and the solvent was distilled off. The residue was treated with ether after the addition of water. The ethereal solution was dried with anhydrous Na_2SO_4 and finally it was subjected to vacuum distillation. Trimethyltin sulphide thus prepared is a colourless oily liquid with a strong disagreeable odour; b.p. 118^{18mm}; m.p., 7°C.

It is soluble in ether, petroleum ether, benzene but insoluble in water. It reacts with hydrochloric acid with evolution of hydrogen sulphide and produces the acid derivative, trimethyltin chloride.

Anal. Subs., 0.3095: SnO_2, 0.2597.
Calcd. for $C_6H_{18}Sn_2S$: Sn, 66.02.
Found: Sn, 65.91.

Triethyltin Sulphide: This compound was prepared in the same way as trimethyltin sulphide. It is a colourless liquid (b.p. $187–188^{20mm}$). It is soluble in ordinary organic solvents and insoluble in water.

Anal. Subs., 0.5355: SnO_2, 0.3644 and $BaSO_4$, 0.2727.
Calcd. for $C_{12}H_{30}Sn_2S$, Sn and S, 53.51 and 7.20.
Found: Sn, 53.60 and S, 7.03.

(1) M.W. of the Subs. in Benzene (Cryoscopic Method)

Subs.	Conc (%)	ΔT	C_6H_6(c.c.) at 20°C	Obs. M.W.
0.7566	3.328	0.397	25	425
"	2.107	0.243	40	425
"	1.582	0.178	55	431

(2) M.W. of the Subs. in Naphthalene (Cryoscopic Method)

Subs.	Conc (%)	ΔT	$C_{10}H_8$ (g.)	Obs. M.W.
0.8142	3.809	0.631	20.30	439
"	2.708	0.431	29.25	446
"	1.859	0.296	42.99	442

Calcd. for $((C_2H_5)_3Sn)_2S$. M.W., 443.7.

Tri-*n*-propyltin Sulphide. This compound was prepared in the same way as trimethyltin and triethyltin sulphides. It is a colourless liquid (b.p., $215–219^{18mm}$) and is soluble in organic solvents but insoluble in water.

Anal. Subs., 0.4034: SnO_2, 0.2300; $BaSO_4$, 0.1777.
Cald. for $C_{18}H_{42}Sn_2S$: Sn, 44.98 and S, 6.07.
Found: Sn, 44.90 and S, 6.05.

Summary.

Trialkyltin sulphide has been prepared according to the reaction:

$$2\,R_3SnX + Na_2S = R_3SnSSnR_3 + 2\,NaX$$

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