

210. The Preparation and Properties of Some Alkylthio-compounds of Tin.

By E. W. ABEL and D. B. BRADY.

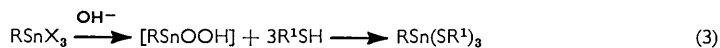
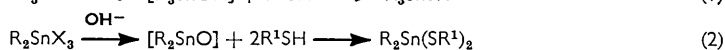
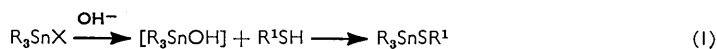
Compounds of the type R_3SnSR^1 , $R_2Sn(SR^1)_2$, and $RSn(SR^1)_3$ have been prepared in aqueous solution from the corresponding organotin halides and thiols. The ring compound $\overline{S \cdot CH_2 \cdot CH_2 \cdot S} \cdot SnMe_2$ and the spiran derivative $\overline{S \cdot CH_2 \cdot CH_2 \cdot S} \cdot Sn \cdot \overline{S \cdot CH_2 \cdot CH_2 \cdot S}$ have also been prepared in aqueous solution, and found to be completely unattacked by water. Action of methyl iodide upon methylthiotrimethyltin gave the stable sulphonium salt $[Me_3Sn \cdot SMe_2]^+I^-$. Bromine causes fission of the tin-sulphur bond to give the organic disulphide and organotin bromide, and phosphorus trichloride produces the organotin chloride and trithiophosphite. The infrared spectra of the compounds are reported.

To further investigations of diamagnetic susceptibility^{1,2} and relative base strengths³ already commenced on the organometallic compounds of Group IV, it was necessary to prepare a variety of previously unreported alkylthio-compounds of tin.

To date, the methods reported for the preparation of these compounds have, in the main, involved the use of carefully maintained anhydrous conditions.⁴ Thus, for example, di(phenylthio)diphenyltin was prepared from the reaction between dibromodiphenyltin and the sodium salt of thiophenol,⁵ di(alkylthio)dialkyltins were made by boiling dialkyltin oxides with the thiol and toluene, and removal of the water formed as an azeotrope.⁶

We find that alkylthio-compounds of tin, in marked contrast to the corresponding silicon derivatives,⁷ are not hydrolysed by water, and may consequently be prepared very conveniently and more cheaply by aqueous methods. Methylthiotrimethyltin has been recovered unchanged after shaking it for one hour with an excess of water.

The respective organotin halides react with a slight excess of thiol according to equations (1), (2), and (3). Sufficient alkali is added to remove completely the acid formed by hydrolysis of the organotin halide.



(X = Cl or Br)

The whole process can be carried out in a totally enclosed system using magnetic stirring to mix the reactants. The product settles out as a heavy oil, which is easily washed, dried, and distilled to give the pure material.

These alkylthio-compounds of tin are colourless, dense, highly refractive and high-boiling liquids, miscible with all common organic solvents.

Previous reports of the heterocyclic and spiro-derivatives of tin formed from ethane-1,2-dithiol and dialkyl-tin dihalides and tin tetrahalides, respectively, have involved

¹ E. W. Abel and R. P. Bush, *Trans. Faraday Soc.*, 1963, **59**, 630.

² E. W. Abel, R. P. Bush, C. R. Jenkins, and T. Zobel, *Trans. Faraday Soc.*, 1964, **60**, 1214.

³ E. W. Abel, D. A. Armitage, and G. R. Willey, *Trans. Faraday Soc.*, 1964, **60**, 1257.

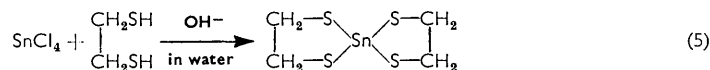
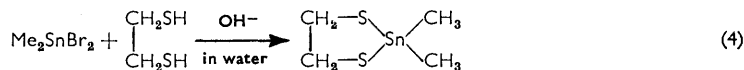
⁴ R. K. Ingham, S. D. Rosenberg, and H. Gilman, *Chem. Rev.*, 1960, **60**, 459.

⁵ H. J. Backer and J. Kramer, *Rec. Trav. Chim.*, 1934, **53**, 1101.

⁶ H. E. Ramsden, W. Buchanan, J. M. Church, and E. W. Johnson, B.P. 719,421.

⁷ "Organosilicon Compounds," C. Eaborn, Butterworths, London, 1960.

carefully controlled anhydrous conditions.⁸⁻¹⁰ We find that both 2,2-dimethyl-2-stanna-1,3-dithiacyclopentane and 1,4,6,9-tetrathia-5-stannaspiro[4,4]nonane are made in virtually quantitative yield in water according to equations (4) and (5).

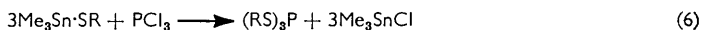


In an investigation of the reaction of ethane-1,2-dithiol with stannous chloride in aqueous solution, the spiran 1,4,6,9-tetrathia-5-stannaspiro[4,4]nonane was isolated in about 15% yield.

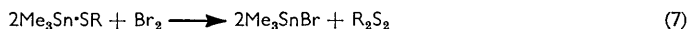
Similarly we find that hexa-alkylditin sulphides and the cyclic dialkyltin sulphides can be prepared in good yield by aqueous methods.

In a preliminary survey of the corresponding method for the preparation of alkylthio-compounds of lead we have found that trimethyl-lead chloride and ethanethiol reacted in aqueous alkali to produce ethylthiotrimethyl-lead in good yield.

Phosphorus trichloride causes fission of the tin-sulphur bond to produce an organothiophosphite and trimethyltin chloride (eqn. 6). This reaction is analogous to the corresponding reactions of alkylthiosilanes.¹¹



The tin-sulphur bond is also cleaved by bromine to give trimethyltin bromide and a disulphide (eqn. 7). The analogous reaction takes place with iodine, and its quantitative nature allows titration of certain organotin-sulphur compounds against standard iodine solutions.



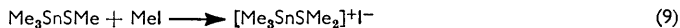
In an effort to produce organotin disulphides, elemental sulphur was heated with, and subsequently dissolved in alkylthiotrimethyltins. Instead of the expected $\text{Me}_3\text{Sn}\cdot\text{S}\cdot\text{SR}$, however, it was found that the product was hexamethylditin sulphide, together with the organic sulphide and disulphide (eqn. 8).



If the organotin disulphide was formed at all, it underwent rapid decomposition.¹²

The thermal decomposition of the pure alkylthiotin compounds is under investigation. A preliminary study of the effect of heat on methylthiotrimethyltin at 270° for 24 hours in an enclosed system shows the formation of tetramethyltin, dimethyl sulphide, some low-boiling hydrocarbons, and an extensive deposit of large, well-formed crystals of stannous sulphide.

Methylthiotrimethyltin and methyl iodide react to produce dimethyl(trimethylstannyl)-sulphonium iodide $[\text{Me}_3\text{SnSMe}_2]^+\text{I}^-$ (eqn. 9). This is a stable crystalline solid (m. p. with decomp. 223—226°), which can be recovered unhydrolysed from aqueous solution.



The corresponding organosilicon sulphonium iodides are very rapidly hydrolysed, and are so thermally unstable that their isolation is extremely difficult.^{13,14}

⁸ M. Wieber and M. Schmidt, *Z. Naturforsch.*, 1963, **18b**, 846.

⁹ R. C. Poller, *Proc. Chem. Soc.*, 1963, 312.

¹⁰ H. J. Backer and W. Drenth, *Rec. Trav. Chim.*, 1951, **70**, 559.

¹¹ E. W. Abel, D. A. Armitage, and R. P. Bush, *J.*, in the press.

¹² M. Schmeisser and M. Muller, *Angew. Chem.*, 1957, **69**, 781.

¹³ E. W. Abel, *J.*, 1960, 4406.

¹⁴ E. W. Abel, D. A. Armitage, and R. P. Bush, *J.*, 1965, in the press.

The infrared spectra of these alkylthio-compounds of tin have been recorded in the range 4000—400 cm^{-1} . Where present, the following modes have been assigned on the basis of previous measurements^{4,15,16} on organotin compounds: (A) symmetric methyl deformation, (B) methyl-tin rocking vibration (C) tin-carbon antisymmetric stretching vibration, (D) tin-carbon symmetric stretching vibration.

EXPERIMENTAL

Trialkyltin halides and dialkyltin dihalides were prepared by proportionation reactions between tetra-alkyltins and the tin tetrahalide in appropriate quantities.^{4,17} Ethyltribromotin was prepared as a colourless liquid, b. p. 68—69°/1 mm.; n_D^{20} 1.6223 (Found: Br, 61.3. Calc. for $\text{C}_2\text{H}_5\text{Br}_3\text{Sn}$: Br, 61.9%), by the method of Luijten and Van der Kerk.¹⁷ Methyltribromotin was prepared by heating dibromodimethyltin with bromine for 8 hr. The product was recrystallised from light petroleum (40—60°) to give colourless crystals, m. p. 50—52° (Found: Br, 63.5. Calc. for $\text{CH}_3\text{Br}_3\text{Sn}$: Br, 63.2%). Where required, methanethiol was used as a gas directly from a cylinder, and was passed into the enclosed reaction system under cylinder pressure (about 2 atm. at room temperature). Infrared spectra were recorded for thin liquid films between sodium chloride and potassium bromide plates using Perkin-Elmer 237 and Unicam S.P. 100 spectrophotometers.

Preparation of Alkyl- and Aryl-thiotrialkyltins.—Each reaction was carried out on ca. 0.2 molar scale. The trialkyltin halide was dissolved in water (about 200 c.c.), and to this was added the requisite thiol in about 5% excess. To this mixture was slowly added, with vigorous stirring, an aqueous solution containing sufficient sodium hydroxide to neutralise the acid formed from hydrolysis of the organotin halide. After 3 hr. the heavy oil formed was allowed to settle, and separated, washed with distilled water, dried (MgSO_4), filtered, and distilled. The yields of pure distilled product (Table 1) were about 60—70%, based upon organotin halide starting material.

TABLE I.
Alkyl- and aryl-thiotrialkyltins.

Product	B. p./mm.	d_4^{20}	n_D^{20}	Found (%)			Reqd. (%)		
				C	H	S	C	H	S
Me_3SnSMe	163°	1.453	1.5303	22.5	6.8	14.7	22.8	5.7	15.2
Me_3SnSEt	177	1.394	1.5205	26.3	5.7	14.3	26.7	6.3	14.3
$\text{Me}_3\text{SnSPr}^a$	54/3	1.352	1.5178	29.9	7.1	13.3	30.1	6.8	13.4
$\text{Me}_3\text{SnSPr}^i$	182	1.318	1.5123	29.8	7.5	13.4	30.1	6.8	13.4
$\text{Me}_3\text{SnSBu}^a$	44/0.05	1.281	1.5098	33.2	6.9	12.7	33.2	7.2	12.7
$\text{Me}_3\text{SnSBu}^i$	42/0.1	1.267	1.5083	33.4	7.2		33.2	7.2	
$\text{Me}_3\text{SnSOct}^a$	94/0.1	1.175	1.5000	42.5	8.7	10.1	42.7	8.5	10.4
Me_3SnSPh	69/0.01	1.418	1.5934	39.9	5.2	11.6	39.6	5.2	11.7
$\text{Et}_3\text{SnSMe}^*$	94/2	1.375	1.5274	32.7	7.7	12.1	33.2	7.2	12.7
$\text{Et}_3\text{SnSEt}^*$	68/0.7	1.359	1.5153						
$\text{Et}_3\text{SnSBu}^t^*$	47/0.02	1.240	1.5130						
$\text{Et}_3\text{SnSPh}^*$	150/1.7	1.314	1.5794	44.7	6.3	10.2	45.7	6.4	10.2

* Compound previously known; refs. 18—20.

Preparation of Di(alkylthio)dialkyltins.—These preparations were carried out on a similar scale, and in a similar way to that described above for the alkylthiotrialkyltins. Care must be taken, however, to add the requisite quantity of aqueous alkali very slowly and with vigorous stirring, in order to avoid the precipitation of the rather insoluble organotin oxides. Yields of pure distilled product (Table 2) were 65—70%.

Preparation of Tri(alkylthio)alkyltins.—These preparations were carried out on a 0.1 molar scale in the manner described above. The lower yield (30—50%) reported for these com-

¹⁵ R. Okawara, D. E. Webster, and E. C. Rochow, *J. Amer. Chem. Soc.*, 1960, **82**, 3287.

¹⁶ P. Taimsalu and J. L. Wood, *Trans. Faraday Soc.*, 1963, **59**, 1754.

¹⁷ J. G. A. Luijten and G. J. M. Van der Kerk, "Investigations in the Field of Organotin Chemistry," Tin Research Institute, Greenford, Middlesex, England, 1955.

¹⁸ G. S. Sasin, *J. Org. Chem.*, 1953, **18**, 1142.

¹⁹ R. Sasin and G. S. Sasin, *J. Org. Chem.*, 1955, **20**, 770.

²⁰ H. H. Anderson and J. A. Vasta, *J. Org. Chem.*, 1954, **19**, 1300.

TABLE 2.
 Di(alkylthio)dialkyltins.

Product	B. p./mm.	d_4^{20}	n_D^{20}	Found (%)			Reqd. (%)		
				C	H	S	C	H	S
Me ₂ Sn(SMe) ₂	44°/0.05	1.547	1.6003	19.8	4.9		19.8	5.0	
Me ₂ Sn(SEt) ₂	58/0.07	1.440	1.5713	26.6	6.6	23.1	26.6	6.0	23.7
Me ₂ Sn(SPr ⁿ) ₂	74/0.1	1.323	1.5498	32.6	7.2	21.5	32.1	6.7	21.4
Me ₂ Sn(SBu ⁿ) ₂	81/0.1	1.280	1.5400	36.4	7.2	19.2	36.7	7.4	19.6
Me ₂ Sn(SOct ⁿ) ₂ * ...	166/0.2	1.092	1.5129	47.8	8.7	15.2	49.2	9.2	14.6
Et ₂ Sn(SMe) ₂	61/0.1	1.440	1.5793	26.9	6.0		26.6	6.0	
Et ₂ Sn(SEt) ₂	94/0.05	1.319	1.5572	32.2	6.9		32.1	6.7	

* This compound foamed persistently during distillation, and was found to be very difficult to obtain perfectly pure.

 TABLE 3.
 Tri(alkylthio)alkyltins.

Product	B. p./mm.	d_4^{20}	n_D^{20}	Found (%)			Reqd. (%)		
				C	H	S	C	H	S
MeSn(SEt) ₃	90°/0.05	1.469	1.5972	26.2	5.8	30.6	26.5	5.7	30.3
MeSn(SPr ⁿ) ₃	95/0.001	1.337	1.5684	33.9	7.5	26.9	33.4	6.7	26.8
EtSn(SMe) ₃	66/0.001	1.548	1.6232	21.5	5.8		20.8	4.9	

pounds (Table 3) appear to be due to the formation of quantities of polymeric organotin oxygen compounds during the reaction which are insoluble and have to be removed by filtration.

Preparation of 2,2-Dimethyl-2-stanna-1,3-dithiacyclopentane.—Ethane-1,2-dithiol (20.0 g., 0.21 mole) was added to dimethyldichlorotin (46.4 g., 0.21 mole), dissolved in water (200 c.c.). Aqueous sodium hydroxide solution was run in slowly with vigorous stirring until the reaction mixture was just alkaline. After 4 hours' stirring, the oil initially formed crystallized, and was filtered off and washed with water (500 c.c.). Recrystallization from methanol yielded long needles (26.0 g., 52%), m. p. 82° (lit.,⁸ 82—83°) (Found: C, 19.7; H, 4.8. Calc. for C₄H₁₀S₂Sn: C, 19.9; H, 4.2%).

Preparation of 1,4,6,9-tetrathia-5-stannaspiro[4,4]nonane.—Ethane-1,2-dithiol (6.9 g., 0.07 mole) was added to stannic chloride pentahydrate (11.9 g., 0.03 mole) in water (250 c.c.). Immediate precipitation of fine white crystals took place, which after being washed with water and recrystallized from methanol gave the product (3.0 g., 30%), m. p. 179—180° (lit.,¹⁰ 181°) (Found: C, 15.3; H, 3.0. Calc. for C₄H₈S₄Sn: C, 15.8; H, 2.7%).

Reaction between Stannous Chloride and Ethane-1,2-dithiol.—Ethane-1,2-dithiol (4.15 g., 0.04 mole) was added to stannous chloride dehydrate (10.0 g., 0.04 mole) in ethanol (25 c.c.). A bright yellow precipitate was formed, which rapidly became white. Crystallization of this from benzene produced 1,4,6,9-tetrathia-5-stannaspiro[4,4]nonane (1.0 g., 15%), m. p. 180°. The infrared spectrum was identical to that of an authentic sample.

Preparation of Ethylthiotrimethyl-lead.—Trimethylchlorolead (17.8 g., 0.06 mole), dissolved in aqueous sodium hydroxide solution was added to ethanethiol (4.4 g., 0.07 mole), and after vigorous shaking (1 hr.) a heavy yellow oil was formed, which was separated and dried (MgSO₄). Subsequent distillation gave ethylthiotrimethyl-lead (10.2 g., 53%), b. p. 36°/0.05 mm., n_D^{20} 1.5918 (Found: C, 18.7; H, 4.4. C₅H₁₄PbS requires C, 19.2; H, 4.5%).

Interaction of Phenylthiotrimethyltin and Phosphorus Trichloride.—Phosphorus trichloride (4.4 g., 0.03 mole) was added slowly to phenylthiotrimethyltin (26.2 g., 0.10 mole) at 0°. Upon warming to 20° an exothermic reaction occurred, and the whole mixture set solid upon cooling. Chlorotrimethyltin (11.3 g.), b. p. 152°, m. p. 37°, was removed by distillation, and the remaining residue was washed with distilled water and dried, to give crude triphenyltrithiophosphate (10.8 g.; 95%), m. p. 71°. This was recrystallized from light petroleum (40—60°) to give the pure triester (7.7 g., 67%), m. p. 76° (lit.,²¹ 77°) (Found: C, 58.6; H, 4.7. Calc. for C₁₈H₁₅PS₃: C, 60.3; H, 4.2%).

Interaction of Phenylthiotrimethyltin and Bromine.—A solution of bromine (4.8 g., 0.03 mole) in carbon tetrachloride (20 c.c.) was added slowly to a solution of phenylthiotrimethyltin (16.4 g., 0.06 mole) in carbon tetrachloride. Distillation of the resulting solution gave initially

²¹ A. Michaelis and G. L. Linke, *Chem. Ber.*, 1907, **40**, 3419.

carbon tetrachloride, and then bromotrimethyltin (6.7 g.), b. p. 159°, m. p. 26°. The residue was washed with water to remove the remaining bromotrimethyltin, and after drying and recrystallization from methanol gave diphenyl disulphide (6.6 g.; 76%), m. p. 59° (lit.²² 60—62°), as fine white needles, with infrared spectrum (4000—650 cm.⁻¹) identical to that of an authentic sample.

Interaction of Methylthiotrimethyltin and Iodomethane.—Iodomethane (9.8 g., 0.069 mole) was added to methylthiotrimethyltin (14.5 g., 0.069 mole). No reaction occurred at room temperature but on warming white crystals were formed. These were recrystallized from methanol to give *dimethyl(trimethylstannyl)sulphonium iodide* $[\text{Me}_2\text{S}\cdot\text{SMe}_2]^+\text{I}^-$ (15.0 g., 62%) m. p. 223—226° (decomp.) (Found: C, 17.5; H, 5.2. $\text{C}_5\text{H}_{15}\text{ISSn}$ requires C, 17.0; H, 4.3%).

{A conductivity measurement in water at 25° gave $\Lambda = 61.6 \text{ ohm}^{-1} \text{ cm.}^2$ (0.0117N-solution) for $[\text{Me}_3\text{Sn}\cdot\text{SMe}_2]^+\text{I}^-$.

Interaction of Methylthiotrimethyltin and Sulphur.—Methylthiotrimethyltin (10.4 g., 0.05 mole) and sulphur (1.6 g., 0.05 mole) were heated for 24 hr. at 120°; during this time a clear yellow oil was formed. Distillation yielded hexamethylditin sulphide (4.7 g.; 53%), b. p. 50°/0.05 mm., n_D^{20} 1.5600 (Found: C, 19.8; H, 5.0. Calc. for $\text{C}_6\text{H}_{18}\text{SSn}_2$: C, 20.0; H, 5.0%). Infrared and proton magnetic resonance spectra of this sample were identical to those of an authentic specimen.

Infrared absorption maxima.

$\text{Me}_3\text{Sn}\cdot\text{SMe}$: 2990s, 2920s, 2860m, 2260w, 1730w, 1698w, 1440s, 1320s, 1190m(A), 960m, 772vs(B), 720s, 700s, 532vs(C), 508s(D).

$\text{Me}_3\text{Sn}\cdot\text{SEt}$: 2990s, 2920s, 2880m, 2370w, 1730w, 1700w, 1456m, 1380m, 1263s, 1194m(A), 1057w, 972m, 778vs(B), 720s, 657m, 533vs(C), 510s(D).

$\text{Me}_3\text{Sn}\cdot\text{SPr}^m$: 2930vs, 2890vs, 2840m, 2320w, 1720w, 1690w, 1452m, 1370m, 1334w, 1325w, 1290m, 1236m, 1184m(A), 1087w, 1043w, 890w, 770vs(B), 720s, 643w, 529vs(C), 508s(D).

$\text{Me}_3\text{Sn}\cdot\text{SPr}^i$: 2940s, 2890w, 2820m, 2320vw, 1720vw, 1690vw, 1458m, 1448m, 1380m, 1360m, 1248m, 1236m, 1184m(A), 1150m, 920vw, 880w, 770vs(B), 712m, 621m, 529vs(C), 506s(D), 430s.

$\text{Me}_3\text{Sn}\cdot\text{SBu}^n$: 2980vs, 2950vs, 2880s, 2360w, 1724w, 1698w, 1467s, 1438m, 1381m, 1352w, 1340w, 1300m, 1286m, 1228m, 1190m(A), 1100w, 914w, 872w, 772vs(B), 728s, 655m, 650w, 532vs(C), 508s(D).

$\text{Me}_3\text{Sn}\cdot\text{SBu}^t$: 2950vs, 2910vs, 2370w, 1970vw, 1726w, 1698w, 1472s, 1452s, 1395m, 1366s, 1218m, 1191m(A), 1172s, 1158s, 1028m, 930vw, 820s, 780vs(B), 718s, 582m, 530vs(C), 508s(D), 423m.

$\text{Me}_3\text{Sn}\cdot\text{SOct}^n$: 2920s, 2840s, 1470m, 1440w, 1382w, 1302vw, 1270w, 1241w, 1192w(A), 776s(B), 722m, 532s(C), 508m(D).

$\text{Me}_3\text{Sn}\cdot\text{SPh}$: 3070s, 2990s, 2910s, 2360w, 1950w, 1880w, 1806w, 1732w, 1700w, 1584vs, 1480vs, 1441s, 1399m, 1271vw, 1195m(A), 1162vw, 1122m, 1090s, 1072m, 1050w, 1029s, 970vw, 910w, 780vs(B), 747vs, 698vs, 534vs(C), 510s(D), 482s, 420s.

$\text{Et}_3\text{Sn}\cdot\text{SMe}$: 2980s, 2750vw, 2160vw, 1467s, 1425s, 1382s, 1320m, 1238m, 1192m, 1010s, 960m, 672vs, 515vs(C), 487s(D).

$\text{Et}_3\text{Sn}\cdot\text{SEt}$: 2960vs, 2880s, 2750w, 2160vw, 1460s, 1422m, 1380m, 1260m, 1238m, 1190m, 1008s, 960m, 760w, 678vs, 515s(C), 488m(D).

$\text{Et}_3\text{Sn}\cdot\text{SBu}^t$: 3010s, 2940s, 1476m, 1430w, 1385w, 1368m, 1240vw, 1220vw, 1190w, 1172w, 1160m, 1020m, 965w, 676m, 646m, 587w, 512m(C), 487w(D).

$\text{Et}_3\text{Sn}\cdot\text{SPh}$: 3070m, 2970vs, 2880vs, 2730w, 1940w, 1879w, 1801w, 1584s, 1479vs, 1470vs, 1442s, 1525s, 1385s, 1305w, 1238m, 1192m, 1127w, 1090s, 1070m, 1030s, 1021s, 964m, 910w, 744vs, 698vs, 676vs, 512vs(C), 482vs(D), 420s.

$\text{Me}_2\text{Sn}(\text{SMe})_2$: 300s, 2920vs, 2860m, 2360w, 1440vs, 1408m, 130vs, 1192m(A), 964s, 764vs(B), 698s, 540vs(C), 518vs(D).

$\text{Me}_2\text{Sn}(\text{SEt})_2$: 2990s, 2930s, 2880m, 1456m, 1380m, 1264vs, 1192m(A), 1049m, 1022w, 973m, 760vs(B), 650m, 540s(C), 518s(D).

²² H. Lecker and M. Wittwer, *Chem. Ber.*, 1922, **55**, 1474.

$\text{Me}_2\text{Sn}(\text{SPr}^n)_2$: 2980vs, 2930vs, 2880s, 1460s, 1438m, 1380m, 1340w, 1298s, 1240s, 1192w(A), 1096w, 1055w, 898w, 838w, 782vs(B), 765vs(B), 720s, 642m, 539s(C), 518s(D).

$\text{Me}_2\text{Sn}(\text{SBu}^n)_2$: 2970vs, 2930vs, 2880s, 1468s, 1440m, 1380m, 1302m, 1277m, 1230m, 1192w(A), 1102w, 918w, 896vw, 875w, 778vs, 762vs(B), 748vs, 728s, 648m, 540s(C), 518s(D).

$\text{Me}_2\text{Sn}(\text{SOct}^n)_2$: 2960vs, 2880vs, 2750w, 2690w, 2380w, 1468vs, 1440vw, 1380s, 1352m, 1301s, 1268w, 1242s, 1205m, 1192m(A), 1170w, 1036w, 918w, 892vw, 762vs(B), 722vs, 646m, 539s(C), 518s(D).

$\text{Et}_2\text{Sn}(\text{SMe})_2$: 3000s, 2980s, 2910m, 1442m, 1385m, 1321m, 1238w, 1192m, 1020m, 962m, 672s, 610w, 520m(C), 495m(D).

$\text{Et}_2\text{Sn}(\text{SEt})_2$: 3000s, 2960s, 2900m, 1458s, 1422sh, 1378m, 1295w, 1262m, 1240w, 1190m, 1052vw, 1018m, 970m, 672s, 518m(C), 495m(D).

$\text{MeSn}(\text{SEt})_3$: 2990s, 2950s, 2890m, 1710vw, 1450s, 1378m, 1262vs, 1190vw(A), 1058m, 1035w, 972m, 758s(B), 626m, 508m(D).

$\text{MeSn}(\text{SPr}^n)_3$: 3010vs, 3000vs, 2940s, 1722vw, 1464s, 1442sh, 1384m, 1342w, 1298s, 1242vs, 1212w, 1192w(A), 1092w, 1082sh, 1056w, 898m, 890m, 840w, 782s, 755s(B), 722sh, 616sh, 642m, 528s(D).

$\text{EtSn}(\text{SMe})_3$: 2990m, 2950s, 2890m, 2870m, 1438s, 1380m, 1310s, 1231w, 1186m, 961s, 682sh, 671s, 510m(D).

$(\text{Me}_3\text{Sn})_2\text{S}$: 2990m, 2910m, 2360vw, 1390w, 1190m(A), 775vs(B), 718sh, 532vs(C), 508s(D).

2,2-Dimethyl-2-stanna-1,3-dithiocyclopentane: 3000vs, 2990vs, 2910sh, 1690vw, 1460s, 1429m, 1383m, 1290m, 1260m, 1198w(A), 1169w, 1122w, 925m, 842m, 770vs(B), 668m, 638m, 545s(C), 511s(D), 442m, 437m.

1,4,6,9-Tetrathia-5-stannaspiro[5,5]nonane: 3010vs, 1468s, 1412s, 1385m, 1292s, 1246s, 1160m, 1125m, 1000w, 932m, 922s, 842s, 722w, 664m, 658m, 628m, 442vs, 390s.

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