

Preliminary communication

ELECTROCHEMICAL CONVERSION OF R_3SnCl TO $ClR_2SnOSnR_2X$ ($X = Cl, OH$)*

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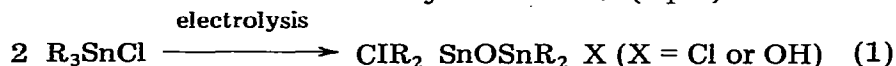
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Summary

Tetraalkyldistannoxane derivatives ($ClR_2SnOSnR_2Cl$ or $ClR_2SnOSnR_2OH$) were prepared from trialkyltin chlorides by electrolysis.

Electrochemical reactions to prepare a variety of organotin compounds involving either tin or cadmium as the sacrificial anode have been reported [1]. In this communication we describe a new synthesis [2] of the tetraalkyldistannoxane derivatives from trialkyltin chlorides (eq. 1).



The chloride (ca. 2 g) dissolved in an alcohol (5–10 ml) was electrolyzed in an undivided open cell, with stirring, at a constant current (0.01–0.2 A) using two platinum electrodes (3 cm²). Electrolysis in methanol and ethanol can be carried out without a supporting electrolyte. However, addition of a small amount (1–5 wt%) of supporting electrolyte was necessary for the electrolysis

TABLE 1

ELECTROLYSIS OF $n\text{-Bu}_3SnCl$ TO $ClBu_2SnOSnBu_2X$ ($X = Cl$ or OH)

| Solvent R'OH | Electricity (F/mol) | Supporting electrolyte | Reaction temp. (°C) | ClBu ₂ SnOSnBu ₂ X yield (%) | |
|------------------------------------|------------------------|-----------------------------------|------------------------|---|-----------------|
| | | | | X = Cl | X = OH |
| R' = CH ₃ | 5.6 | — | 23 | | 51 |
| | 4.5 | LiClO ₄ | 22 | 40 ^a | 30 ^b |
| CH ₃ CH ₂ | 2.5 | — | 23 | | 64 |
| | 5.0 | LiClO ₄ | 25 | 95 | |
| (CH ₃) ₂ CH | 4.0 | LiClO ₄ | 14 | 88 | |
| (CH ₃) ₃ C | 3.3 | Bu ₄ NClO ₄ | 30 | 83 | |

^a Anal. Found (calcd.): C, 34.69 (34.77); H, 6.75 (6.57); Sn, 43.11 (42.95)%. ^b Anal. Found (calcd.): C, 35.68 (35.96); H, 7.06 (6.98); Sn, 44.55 (44.43)%. $\nu(OH)$ 3660 cm⁻¹.

* Dedicated to Professor E.G. Rochow on the occasion of his 70th birthday.

TABLE 2

ELECTROLYSIS OF R_3SnCl TO $ClR_2SnOSnR_2X$ ($X = Cl$ or OH)

| R_3SnCl | Solvent $R'OH$ | Electricity (F/mol) | Supporting electrolyte | Reaction temp. ($^{\circ}C$) | $ClR_2SnOSnR_2X$ yield (%) |
|-------------------|-------------------|------------------------|---------------------------|-----------------------------------|-------------------------------|
| $R = n-C_8H_{17}$ | $R' = CH_3$ | 5.0 | $LiClO_4$ | 15 | $X = Cl^a$ 80 |
| | $(CH_3)_3C$ | 5.0 | $LiClO_4$ | 30 | Cl 76 |
| C_6H_{11} | $(CH_3)_2CH$ | 5.0 | $LiClO_4$ | 30 | Cl 65 |
| C_6H_5 | CH_3 | 12 | — | 16 | OH^b 23 |

^a Anal. Found (calcd.): C, 49.40 (49.45); H, 8.87 (8.82); Sn, 30.49 (30.54) %. ^b Anal. Found (calcd.): C, 46.77 (46.93); H, 3.50 (3.45) %. $\nu(OH)$ 3620 cm^{-1} .

in other alcohols. The reaction conditions and the results of the electrolysis of tri-*n*-butyltin chloride are summarized in Table 1.

Similarly, tri-*n*-octyltin, tricyclohexyltin, and triphenyltin chlorides were electrolyzed as shown in Table 2.

From the results, it is clear that one of the three Sn—C bonds in R_3SnCl is cleaved to give tetraalkyldistannoxane derivatives by anodic oxidation, probably via a cationic intermediate, R_2XSn^+ .

References

- 1 J.J. Habeeb and D.G. Tuck, *J. Organometal. Chem.*, 134 (1977) 363, and references cited therein.
- 2 See A.K. Sawyer (ed.), *Organotin Compounds*, Vol. I—III, Marcel Dekker, New York, 1971.