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Preliminary communication

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

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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).

$$2\ R_3SnCl \xrightarrow{\text{electrolysis}} ClR_2SnOSnR_2X\ (X = Cl\text{ or }OH) \quad (1)$$

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 cm^2). 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)	$ClBu_2SnOSnBu_2X$ yield (%)	
				X = Cl	X = OH
R' = CH_3	5.6	—	23		51
	4.5	$LiClO_4$	22	40[a]	30[b]
CH_3CH_2	2.5	—	23		64
	5.0	$LiClO_4$	25	95	
$(CH_3)_2CH$	4.0	$LiClO_4$	14	88	
$(CH_3)_3C$	3.3	Bu_4NClO_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) %. ν(OH) 3660 cm^{-1}.

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* 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. (°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) %. ν(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.