A Novel Synthesis of Eight-Co-ordinate Tin(IV) Compounds

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Interest in the principles that govern the stereochemistry of tin(Iv) compounds has led to the synthesis of four-, five-, and six-co-ordinate compounds by various workers.¹ Although a few examples of eight-co-ordinate tin are reported, including $[Sn(C_2O_4)_4]^{4-2}$ and tin(Iv) phthalocyanin,³ the paucity of such compounds has occasioned our present interest, and we now report a novel method of synthesis of eight-co-ordinate tin(Iv) complexes: tin-phenyl cleavage of organotin-chelate compounds by chelating agents.

As an example of the method, tetrakis-(8-quinolinolato)tin(IV) was prepared by heating a mixture of diphenylbis-(8-quinolinolato)tin(IV) 4 (5 mmoles) and 8-quinolinol (12 mmoles) at 300°. Benzene was distilled off, and the residue was purified by washing with ether, then with boiling toluene to obtain a brick red crystalline product (21%), m.p.>400°. The compound was further purified by vacuum sublimation.

That this is an eight-co-ordinate tin(IV) compound is indicated by the following information: (1) the compound is a non-electrolyte in nitrobenzene $(0.3 \times 10^{-3}\text{M})$ and does not have the

structure $[Sn(Ox)_3]+Ox^-$; (2) the compound is monomeric [Found: M (Rast in acridine), 760. Calc.: M, 696] the insolubility in other solvents precluded molecular-weight determinations by cryoscopic or ebullioscopic methods; (3) polarographic measurements of dimethyl sulphoxide solutions indicate that the two- and four-electron reduction potentials are the same for the complex and for diphenyltin dichloride; (4) that the 8quinolinolate ligands are truly bidentate is indicated by the thermal stability in boiling toluene, by the infrared absorption spectrum of the complex which showed a weak absorption at 418 cm.⁻¹ diagnostic of co-ordination of the ring nitrogen to tin,5 and the failure of methyl iodide to form a quaternary nitrogen (conductometric titration in nitrobenzene).

The use of tin-phenyl cleavage by appropriate chelating agents appears to offer a novel and convenient route to the synthesis of eight-co-ordinate tin compounds.

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⁴ W. H. Nelson and D. F. Martin, J. Inorg. Nuclear Chem., 1965, 27, 89.