Melting points were obtained both from a Fisher–Johns instrument and on a DuPont 900 Differential Thermal Analyzer.

Infrared spectra were recorded as Nujol mulls on a Baird infrared recording spectrophotometer Model 4-55 and on a Perkin–Elmer 337 grating infrared spectrophotometer, calibrated with polystyrene film.

Elemental analyses were carried out by Dr. CAROL K. FITZ, Needham Heights, Massachusetts and Galbraith Laboratories, Inc., Knoxville, Tennessee.

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1 F. A. COTTON AND R. FRANCIS, *J. Am. Chem. Soc.*, 82 (1960) 2986.
2 N. A. MATWIYOFF AND R. S. DRAGO, *Inorg. Chem.*, 3 (1964) 337.
3 K. HILLS AND M. C. HENRY, *J. Organometal. Chem.*, 3 (1965) 159.
4 F. A. COTTON, R. FRANCIS AND W. D. HORROCKS, JR., *J. Phys. Chem.*, 64 (1960) 1534.
5 A. L. ALLRED AND E. G. ROCHOW, *J. Inorg. Nucl. Chem.*, 5 (1958) 269.
6 D. COOK, *Can. J. Chem.*, 41 (1963) 522.
7 H. GILMAN, *Organic Chemistry*, Vol. I, Wiley, New York, 2nd ed., 1943.
8 N. A. MATWIYOFF AND R. S. DRAGO, *J. Organometal. Chem.*, 3 (1965) 393.

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Preparation of dialkyltin dinitrates

Trialkyltin nitrates[1] have been prepared by the interaction of the appropriate trialkyltin halide with alcoholic silver nitrate solution. The preparation of dimethyltin dinitrate from tetramethyltin and liquid dinitrogen tetroxide[2], and also from dimethyltin oxide and nitric acid[3] has recently been reported. The preparation of diethyltin dinitrate[4] is claimed in the early literature.

We now report the preparations of dimethyltin dinitrate and the previously unreported di-*n*-propyltin and di-*n*-butyltin dinitrates by the silver nitrate method.

$$R_2SnCl_2 + 2\ AgNO_3 \longrightarrow R_2Sn(NO_3)_2 + 2\ AgCl$$

The 1,10-phenanthroline complexes of the di-*n*-propyltin and di-*n*-butyltin dinitrates have been prepared.

The dialkyltin dinitrates are very deliquescent compounds which are soluble in polar organic solvents. The nitrates undergo decomposition with the liberation of oxides of nitrogen at room temperature even when stored in a sealed tube, but they can be stored at low temperature (—18°) without appreciable decomposition.

The 1,10-phenanthroline complexes, on the other hand, are perfectly stable at room temperature and are non-deliquescent.

The NMR and IR spectra of these compounds are being studied.

Experimental

*Di-n-propyltin dinitrate**. Silver nitrate (36.90 g, 2.0 mol.) in a Soxhlet thimble was extracted for several hours by dry methanol (200 ml) in which di-*n*-propyltin dichloride (30.0 g, 1.0 mol.) was dissolved. The precipitated silver chloride (31.4 g, ~ 100%) was removed by filtration. Evaporation of the filtrate at 20°/0.1 mm, fol-

lowed by recrystallisation from chloroform yielded di-*n*-propyltin dinitrate, m.p. 137–138° (22.67 g, 63.4 %). (Found: Sn, 36.2. $C_6H_{14}N_2O_6Sn$ calcd.: Sn, 36.0 %.)

Dimethyltin dinitrate (12.00 g, 92.0 %), (Found: Sn, 43.8. $C_2H_6N_2O_6Sn$ calcd.: Sn, 43.5 %.) was similarly obtained from dimethyltin dichloride (10.51 g, 1.0 mol.) and silver nitrate (16.27 g, 2.0 mol.).

*Di-n-butyltin dinitrate** (65.7 %), m.p. 103.5–104.5°, (Found: Sn, 33.4. $C_8H_{18}N_2O_6Sn$ calcd.: Sn, 33.2 %.) was also prepared by the same method.

*Di-n-propyltin dinitrate–1,10-phenanthroline**. 1,10-Phenanthroline hydrate (2.0 g, 1.0 mol.) in absolute ethanol (25 ml) was added to di-*n*-propyltin dinitrate (3.3 g, 1.0 mol.) in the same solvent (25 ml) with shaking. The white precipitate, which formed immediately, was recrystallised from absolute ethanol to yield the di-*n*-propyltin dinitrate 1,10-phenanthroline complex (4.8 g, 93.7 %), m.p. 205–206° (dec.). (Found: C, 42.0; H, 4.4; N, 11.6; Sn 23.3. $C_{18}H_{22}N_4O_6Sn$ calcd.: C, 42.5; H, 4.4; N, 11.0; Sn, 23.3 %.)

*Di-n-butyltin dinitrate–1,10-phenanthroline** (5.2 g, 96.0 %), m.p. 209–212° (dec.), (Found: C, 44.5; H, 4.8; N, 10.1; Sn, 22.0. $C_{20}H_{26}N_4O_6Sn$ calcd.: C, 44.7; H, 4.9; N, 10.4; Sn, 22.0 %.) was similarly obtained from di-*n*-butyltin dinitrate (3.6 g, 1.0 mol.) and 1,10-phenanthroline hydrate (2.0 g, 1.0 mol.).

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1 H. C. CLARK AND R. J. O'BRIEN, *Inorg. Chem.*, 2 (1963) 740.
2 C. C. ADDISON, W. B. SIMPSON AND A. WALKER, *J. Chem. Soc.*, (1964) 2360.
3 M. M. MCGRADY AND R. S. TOBIAS, *Inorg. Chem.*, 3 (1964) 1157.
4 A. CAHOURS, *Ann. Chem.*, 114 (1860) 354; C. LÖWIG, *Ann. Chem.*, 84 (1852) 308.

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* New compound.

Explosion hazard: benzene-π-cyclopentadienyliron (II) perchlorate

We wish to report that benzene-π-cyclopentadienyliron(II) perchlorate, $[C_6H_6(\pi\text{-}C_5H_5)Fe]ClO_4$, is potentially very dangerous. It crystallizes from alcoholic solution as yellow-brown prisms. It detonated violently when touched with a spatula after recrystallization. The worker involved severely injured one hand and lost his thumb. Two months after the accident he died from extensive liver damage. The cause of this damage is, as yet, unknown but toxication from the original explosion has not been ruled out.

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