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NOTE

A CONVENIENT PREPARATION OF DI-n-BUTYLTIN DIPHENOXIDES

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We have found that di-n-butyltin diphenoxides can be conveniently prepared by the direct interaction of di-n-butyltin oxide with phenols. Di-n-butyltin diphenoxide has previously been prepared from di-n-butyltin dichloride with sodium phenoxide[1], and by trans-esterification of the corresponding dimethoxide with phenol[2].

A previous report[3] has stated that the interaction between a dialkyltin oxide and a phenol, in boiling toluene, does not proceed beyond the tetraalkyl-1,3-diphenoxydistannoxane stage (1):

$$2\,R_2SnO + 2\,PhOH \rightarrow (R_2SnOPh)_2O + H_2O \qquad (1)$$

We find that, at a higher temperature (boiling tetralin), di-n-butyltin diphenoxides may be obtained according to scheme (2):

$$R_2SnO + 2\,PhOH \rightarrow R_2Sn(OPh)_2 + H_2O \qquad (2)$$

EXPERIMENTAL

Tin was determined as SnO_2[4]. The phenoxy and *p*-methylphenoxy groups were estimated as the bromo derivatives[5] of the corresponding phenols (obtained by hydrolysis of the phenoxides). Chlorine was determined by the oxygen flask method[6].

The NMR spectra were obtained using a Perkin–Elmer R-10 spectrometer.

Di-n-butyltin diphenoxide

Di-n-butyltin oxide (31.0 g, 1.0 mol.) was suspended in tetralin (50 ml) and phenol (58.0 g, 4.95 mol.) added. The reaction mixture was heated under reflux at 225–240° (bath temperature) with stirring, until the theoretical amount of water (2.2 g, 1.0 mol.) was evolved (3½ h). A Dean and Stark apparatus with xylene* in the side arm was used. The solvent was removed, and the di-n-butyltin diphenoxide (43.8 g, 84%) was obtained on distillation as a pale yellow oil, which crystallised on cooling, b.p. 146–148°/0.1 mm, m.p. 51–53° (lit.[1] b.p. 161°/0.35 mm, m.p. 45–48°). (Found: C, 57.0; H, 6.7; Sn, 28.4; C_6H_5O on hydrolysis, 44.1. $C_{20}H_{28}O_2Sn$ calcd.: C, 57.2; H, 6.7; Sn, 28.4; C_6H_5O, 44.3%.) The NMR spectrum was consistent with

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* Xylene was used because the organic phase of the distillate was denser than water.

the structure and indicated that the ratio aliphatic/aromatic protons was 1.8 (theoretical 1.8).

Di-n-butyltin bis(p-methylphenoxide) (new compound) (23.7 g, 73%) was similarly obtained from di-n-butyltin oxide (18.2 g, 1.0 mol.) and *p*-cresol (28.5 g, 3.6 mol.), b.p. 166.5–168.5°/0.05 mm, m.p. 65–71°. (Found: C, 57.8; H, 7.2; Sn, 26.1; *p*-$CH_3C_6H_4O$. 48.4. $C_{22}H_{32}O_2Sn$ calcd.: C. 59.1; H. 7.2; Sn. 26.5; p-$CH_3C_6H_4O$. 47.9%.) The ratio aliphatic/aromatic protons by NMR was 3.0 (theoretical, 3.0).

Di-n-butyltin bis(p-chlorophenoxide) (new compound) (75%), b.p. 178–180°/ 0.07 mm, m.p. 80–85°. (Found: C, 47.9; H, 5.5; Cl, 14.3; Sn, 23.5. $C_{20}H_{26}Cl_2O_2Sn$ calcd.: C, 49.3; H, 5.4; Cl, 14.5; Sn, 24.3%.) The ratio aliphatic/aromatic protons by NMR was 2.3 (theoretical, 2.3).

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