

## NOTE

### A CONVENIENT PREPARATION OF DI-*n*-BUTYLTIN DIPHENOXIDES

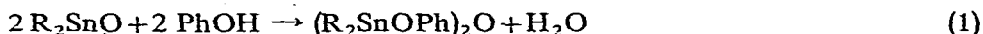
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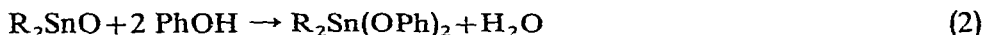
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We have found that di-*n*-butyltin diphenoxides can be conveniently prepared by the direct interaction of di-*n*-butyltin oxide with phenols. Di-*n*-butyltin diphenoxide has previously been prepared from di-*n*-butyltin dichloride with sodium phenoxide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

A previous report<sup>3</sup> has stated that the interaction between a dialkyltin oxide and a phenol, in boiling toluene, does not proceed beyond the tetraalkyl-1,3-diphenoxy-distannoxane stage (1):



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



#### EXPERIMENTAL

Tin was determined as SnO<sub>2</sub><sup>4</sup>. The phenoxy and *p*-methylphenoxy groups were estimated as the bromo derivatives<sup>5</sup> of the corresponding phenols (obtained by hydrolysis of the phenoxides). Chlorine was determined by the oxygen flask method<sup>6</sup>.

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.<sup>1</sup> b.p. 161°/0.35 mm, m.p. 45–48°). (Found: C, 57.0; H, 6.7; Sn, 28.4; C<sub>6</sub>H<sub>5</sub>O on hydrolysis, 44.1. C<sub>20</sub>H<sub>28</sub>O<sub>2</sub>Sn calcd.: C, 57.2; H, 6.7; Sn, 28.4; C<sub>6</sub>H<sub>5</sub>O, 44.3%). The NMR spectrum was consistent with

\* 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<sub>3</sub>C<sub>6</sub>H<sub>4</sub>O. 48.4. C<sub>22</sub>H<sub>32</sub>O<sub>2</sub>Sn calcd.: C, 59.1; H, 7.2; Sn, 26.5; *p*-CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>O. 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<sub>20</sub>H<sub>26</sub>Cl<sub>2</sub>O<sub>2</sub>Sn 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).

#### ACKNOWLEDGEMENT

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