EXPERIMENTAL

To 5.0 g. (0.0113 mol.) of triphenylbenzyltin in 150 ml. of ether cooled to −35° (by a Dry Ice–acetone bath) was added 0.012 mol. of phenyllithium in 10 ml. of ether. The solution turned bright yellow immediately and then tetraphenyltin precipitated from the solution a few minutes later. After 1 hr. of stirring the mixture was allowed to stand a few minutes while the solid settled. The supernatant solution was decanted onto a Dry Ice–ether slurry with vigorous stirring, and the yellow color of benzyllithium was discharged. On working up the mixture by conventional procedures involving alkaline liquid–liquid extraction, acid liquid–liquid extraction, and crystallization there was obtained 0.6 g. (39%) of phenylacetic acid melting at 73–74° (mixed melting point). From this experiment, the yield of tetraphenyltin was 4.1 g. (88%). In two other experiments the yields of tetraphenyltin were 91.8% and 89%, respectively.

It might be mentioned that, under corresponding conditions, from reaction between triphenylethyltin and phenyllithium there was obtained a 9.4% yield of tetraphenyltin in addition to a 52.6% recovery of triphenylethyltin.

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Studies on Synthetic Estrogens. I

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Following the observation that oxygen heterocyclic compounds such as coumarins,[1] isoflavens, isoflavones,[2] etc. have estrogenic activity, Buu-Hoï and co-workers[3] synthesized a number of benzofuran derivatives and some of these compounds showed estrogenic activity. In view of the physiological properties of the benzofuran derivatives[4] it was of considerable interest to synthesize additional derivatives.

2-*p*-Anisoylbenzofurans were prepared by refluxing an alcoholic solution of the potassium salt of *o*-hydroxyketones with *p*-methoxyphenacylbromide[5] according to Buu-Hoï.[3b] They have been characterized through their oximes.

The details of the *in vivo* biological activity of these compounds will be reported later.

EXPERIMENTAL[6]

o-Hydroxyketones were prepared by the method of Fries.[7]

2-p-Anisoyl-3,5-dimethylbenzofuran. A solution of 5-methyl-2-hydroxyacetophenone (0.1 mole) dissolved in caustic potash (0.125 mole) was added to *p*-methoxyphenacylbromide (0.1 mole) in ethanol. It was refluxed for 2 hr. on a water bath. The 2-*p*-anisoyl-3,5-dimethylbenzofuran formed was isolated and recrystallized from an acetic acid ethanol mixture. Similarly other benzofurans were prepared. The data concerning the new compounds are listed in Table I.

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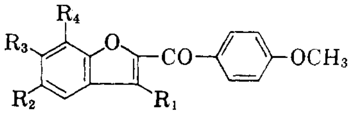
TABLE I

2-*p*-ANISOYLBENZOFURANS



											Oxime			
						Solvent for	M.P.,		Analysis		M.P.,		Analysis	
Sl. No.	R_1	R_2	R_3	R_4	Formula	Recrystn.	°C.		Calcd.	Found	°C.		Calcd.	Found
1.	CH_3	CH_3	H	H	$C_{18}H_{16}O_3$	AcOH-EtOH	118	C:	77.14	76.7	169	C:	73.23	72.9
								H:	5.71	5.55		H:	5.76	5.61
2.	CH_3	Cl	H	H	$C_{17}H_{13}O_3Cl$	AcOH-EtOH	165	Cl:	11.81	11.6	110	Cl:	11.26	10.8
3.	CH_3	Br	H	H	$C_{17}H_{13}O_3Br$	AcOH-EtOH	154	Br:	23.19	22.6	189	Br:	22.22	22.1
4.	CH_3	CH_3	H	Br	$C_{18}H_{15}O_3Br$	AcOH-EtOH	190	Br:	22.28	21.9				
5.	CH_3	Cl	Cl	H	$C_{17}H_{12}O_3Cl_2$	AcOH-EtOH	155	Cl:	21.19	20.8				
6.	C_2H_5	CH_3	H	H	$C_{19}H_{18}O_3$	EtOH	148	C:	77.56	77.1	213	C:	73.77	73.4
								H:	6.12	5.71		H:	6.15	5.92
7.	C_2H_5	Cl	H	H	$C_{18}H_{15}O_3Cl$	EtOH	103	Cl:	11.29	11.1	144	Cl:	10.77	10.5
8.	C_2H_5	Br	H	H	$C_{18}H_{15}O_3Br$	EtOH	93	Br:	22.28	21.8	215	Br:	21.39	20.9

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