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REACTION OF β-KETOENOLS WITH DIPHENYLBORINIC ESTERS

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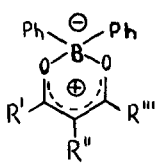
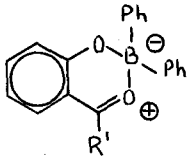
In previous papers the formation of crystalline coordinated compounds was described, from 2-n-butoxy-1,3,2-benzodioxaborole and tropolones,[1,2,3] 1,3-diketones[4] or other bidentate ligands.[5] The recent apparition of two notes by Umland and Schleyerbach[6,7] prompts us to report the preparation of similar compounds on replacing the benzodioxaborole group by the diphenylborinic residue.

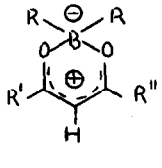
β - Aminoethyl diphenylborinate (Flavognost)[8] and β -ketoenols (1,3-diketones or o-acylphenols) afford on refluxing for 5-10 hrs in benzene crystalline yellow products I and II, respectively, either by cooling or by addition of petroleum ether, in yields of 50-70 %.



I



II



III

These compounds are stable to the atmosphere and may be recrystallized from benzene, acetonitrile or ethanol. They give satisfactory elementary analyses.

Absorption maxima in acetonitrile and melting points are given in the table. Absorption maxima below 270 mμ are not included (flavognost has a band at 247 mμ with shoulders at 259 and 269 mμ). The longest-wavelength band is characteristic of the conjugated system ; two bands at 310-314 and 269-284 mμ are common to all compounds reported in the table.

	R'	R"	R'"	M.p.	Maxima (mμ) in acetonitrile				
I	Ph	H	Ph	227	391,		309,	300,	279 s
	Ph	H	An	231 dec.	400,	334,	309,	300,	284
	An	H	An	283	420,	404,	314,		269
	Me	H	An	209	365,	320,	310,		270
	Me	Me	Me	135	338,		310,		278 s
II	H	-	-	149[a]	375,		309		280
	Me	-	-	115 dec.	364,		309,		275

a Lit.[6] m.p. 151.°

Infrared spectra in potassium bromide pellets present in the range 1600-2000 cm^{-1} one band at 1595-1620 cm^{-1} due to the aryl groups. Bands due to B-O stretching vibrations appear at 1300-1370 $cm.^{-1}$

The products I have the same skeleton III as compounds described previously with R' = R" = F (literature cited in [4a,9,10]), R' = R" = Alk,[11] and R', R" = 1,2-benzodioxa.[4]

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