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POLYFURYL(ARYL)ALKANES AND THEIR DERIVATIVES.

4.* SYNTHESIS OF FURYLDIARYLMETHANE DERIVATIVES

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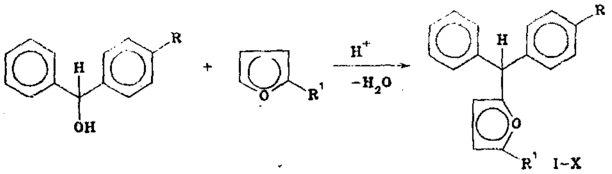
UDC 547.727.07:542.953

A number of substituted furyldiarylmethanes were obtained by the reaction of furan derivatives with secondary aromatic alcohols.

Very little information on furyldiarylmethane derivatives is available. Only the preparation for furylbis(N,N-dimethylaminophenyl)methane by the reaction of furfural with dimethylaniline has been described [2]. An alternative method for their synthesis is the reaction of furans with substituted benzhydrols.

We have established that alkylfurans, as well as ethyl furoate, react with benzhydrol derivatives in benzene in the presence of catalytic amounts of perchloric acid to give the corresponding furyldiarylmethanes (I-X) in up to 96% yields (Table 1):



I–V R=H; I $R^1=CH_3$; II $R^1=C_4H_9$; III $R^1=C_5H_{11}$; IV $R^1=C_6H_{13}$; V $R^1=C_7H_{15}$; VI, VII $R^1=CH_3$; VI R=Br; VII $R=OCH_3$; VIII–X $R^1=COOC_2H_5$; VIII R=Cl; IX R=Br; X $R=OCH_3$

The product of the reaction of unsubstituted benzhydrol with ethyl furoate is not the expected (5-carbethoxy-2-furyl)diphenylmethane but rather 1,1,1',1'-tetraphenyldimethyl ether, which is obtained as a result of self-etherification of benzhydrol. The formation of this product in the reaction of benzhydrol with phenol was previously reported [3].

The presence of several groups of resonance peaks is characteristic for the PMR spectra of furyldiarylmethanes I-X: signals of aryl protons at 6.87-7.1 ppm, double doublets of furan β protons of the compounds that contain an alkyl group in the 5 position at 5.7-5.8 (3H) and 5.5-5.6 ppm (4H), and double doublets of furan β protons of the compounds that contain an ester group in the 5 position at 5.8-5.9 (3H) and 6.9-7.0 ppm (4H).

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*See [1] for Communication 3.

Krasnodar Polytechnic Institute, Krasnodar 350700. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 3, pp. 307-308, March, 1984. Original article submitted May 23, 1983.

0009-3122/84/2003-0244$08.50

TABLE 1. Furyldiarylmethanes I-X

Compound	bp, °C (mm)	n_D^{20}	d_4^{20}	Found, %			Empirical formula	Calc., %			Yield, %
				C	H	halogen		C	H	halogen	
I	44*	—	—	87,4	6,1	—	$C_{18}H_{16}O$	87,1	6,5	—	96
II	160—165 (1)	1,5588	1,0610	86,3	7,2	—	$C_{21}H_{22}O$	86,8	7,7	—	94
III	175—180 (1)	1,5538	1,0500	86,5	8,0	—	$C_{22}H_{24}O$	86,8	8,0	—	94
IV	185—190 (1)	1,5536	1,0371	86,2	8,0	—	$C_{23}H_{26}O$	86,7	8,3	—	90
V	198—203 (1)	1,5484	1,0172	87,0	8,9	—	$C_{24}H_{28}O$	86,7	8,5	—	91
VI	63—64*	—	—	66,7	5,0	24,9	$C_{18}H_{15}BrO$	66,1	4,6	24,4	73
VII	196—200 (4)	1,5842	—	82,1	7,0	—	$C_{19}H_{18}O_2$	82,0	6,0	—	79
VIII	230—231 (1)	—	—	71,1	5,2	10,3	$C_{20}H_{17}ClO_3$	70,5	5,0	10,4	81
IX	242—245 (1)	1,6012	—	62,8	4,5	20.5	$C_{20}H_{17}BrO_3$	62,4	4,5	20,7	84
X	242—244 (3)	1,5872	—	75,3	5,9	—	$C_{21}H_{24}O_4$	75,0	6,0	—	72

*Melting point.

EXPERIMENTAL

The UV spectra of solutions of the compounds in ethanol were recorded with a Specord UV-vis spectrophotometer. The IR spectra of thin layers or mineral oil suspensions of the compounds were recorded with a UR-20 spectrometer. The PMR spectra of solutions of CCl_4 were recorded with a Tesla BS-487 spectrometer (80 MHz) with hexamethyldisiloxane as the internal standard.

(5-Methyl-2-furyl)diphenylmethane (I). A solution of 18.4 g (0.1 mole) of benzhydrol in 100 ml of benzene was placed in a two-necked round-bottom flask equipped with a Dean—Stark trap and heated to the boiling point, 0.2 ml of 1 N $HClO_4$ in CH_3COOH was added, and 10 ml (0.11 mole) of α-methylfuran was added dropwise. After 1.8 ml of water had collected in the Dean—Stark trap, the reaction mixture was cooled to room temperature, washed successively with a 5% solution of sodium carbonate (100 ml) and water (100 ml), and dried with anhydrous Na_2SO_4. The solvent was removed, and the residue was recrystallized from ethanol.

Compounds II-X were similarly obtained. In the preparation of II-V and VIII-X the furan component was added all at once. In the isolation of furyldiarylmethanes II-V and VII-X, after removal of the solvent, the residue was fractionated in vacuo. The products were obtained in 72-94% yields.

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