Chinese Chemical Letters Vol. 10, No. 5, pp. 345–346, 1999

Condensation Products from β-Keto-δ-valerolactones

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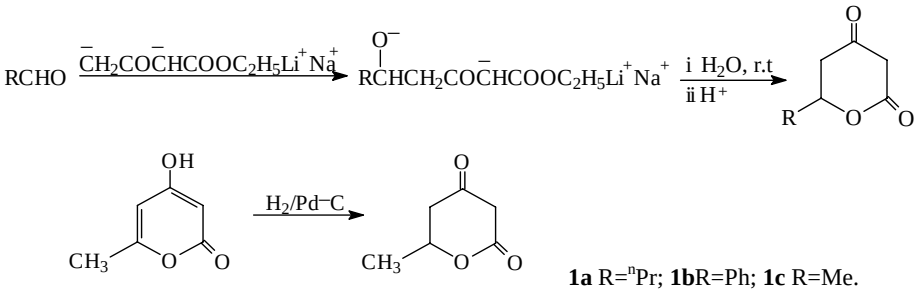
Abstract: Unexpected condensation products from β-keto-δ-valerolactones were obtained. Their structures were confirmed by ^{1}HNMR spectrum and elemental analysis.

Keywords: Condensation, condensation reaction, β-keto-δ-valerolactone.

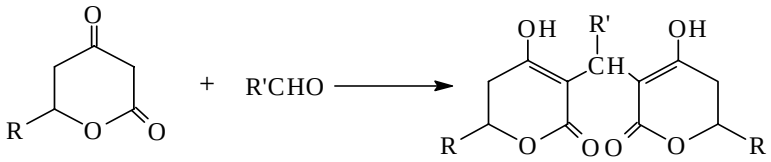
Previously we reported a new condensation reaction of β-keto-δ-valerolactones[1-2] and substituted anilines in the presence of ethyl orthoformate, by which a new carbon-carbon double bond in 3 position of β-keto-δ-valerolactones was formed[3]. We attempted to build a new carbon-carbon double bond in 3 position of β-keto-δ-valerolactones by Mannich reaction[4-5], but failed to gain the expected results. Thus we reacted β-keto-δ-valerolactone **1**b, formaldehyde with propylamine in the presence of ethanol, the product was very complex and no desired compound was separated. We used 2,6-dichloroaniline instead of propylamine and obtained the unexpected product **3**d.



1b **3d**

In order to prove this condensation reaction, we reacted β-keto-δ-valerolactones with different kinds of aldehydes in the presence of ethanol and received similiar results.

Its reaction conditions were also investigated, and the result indicated that the condensation temperature and time were different because of the different reactivity of various kinds of aldehydes. By means of this method, a series of bis (6-alkyl (aryl)-5,6-dihydro-4-hydroxy-2H-pyran-3yl) alkyl (aryl) methanes **3** were synthesized.



1 **2** **3**

In a general procedure, β-keto-δ-valerolactones **1** reacted with aldehydes **2** in the preesence of ethanol at different reaction conditions and the products **3** were obtained. The products **3** were purified by silica gel column or recrystallization and confirmed by ^{1}HNMR spectrum and elemental analysis.

Table 1. Prepared Data of Bis (6-alkyl (aryl)-5,6-dihydro-4-hydroxy-2H-pyran-3yl) alkyl (aryl) methanes **3**

compound **3**	R	R'	Reaction temperature	Reaction time	Yield(%)	m.p.(°C)
a	Me	H	r.t	8	80.6	193-195
b	Me	^{n}Pr	reflux	20	84.4	134-135
c	Me	Ph	reflux	12	81.4	182-183
d	^{n}Pr	H	r.t	8	82.8	163-164
e	Ph	H	r.t	20	80.3	179-180
f	Ph	^{n}Pr	reflux	24	80.5	132-133
g	Ph	Ph	reflux	16	85.1	176-177

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Received 14 December 1998