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Pyran derivatives R 0340

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## 51-152

One-Pot Synthesis of 4H-Chromenes by Tandem Benzylation and Cyclization in the Presence of Sodium Bisulfate on Silica Gel. — Application of 1,3-di-tert-butyl-pentan-1,3-dione as the diketone or 2-(hydroxy-tert-butyl-methyl)phenol as the o-hydroxybenzylic alcohol fails to give the desired chromenes. — (AOYAMA\*, T.; YAMAMOTO, T.; MIYOTA, S.; HAYAKAWA, M.; TAKIDO, T.; KODOMARI, M.; Synlett 25 (2014) 11, 1571-1576, http://dx.doi.org/10.1055/s-0033-1339026; Dep. Mater. Appl. Chem., Coll. Sci. Technol., Nihon Univ., Chiyoda, Tokyo 101, Japan; Eng.) — F. Schill

$$\begin{array}{c} \text{Ph} \\ \text{OH} \\ \text{OH} \\ \text{I} \end{array} \begin{array}{c} \text{R}^{1} & \text{OR}^{1} & \text{(II)} \\ \\ \text{R}^{1} & \text{R}^{1} & \text{OR}^{1} & \text{OR}^{1} & \text{-Me} & 94\% \\ \\ \text{R}^{1} & \text{R}^{1} & \text{-ipr} & 63\% \\ \\ \text{R}^{1} & \text{-ipr} & 63\% \\ \\ \text{CR}^{1} & \text{-Ph} & 98\% \\ \end{array}$$

A):  $NaHSO_4-SiO_2$ ,  $CH_2CI_2$ ,  $BO^{\circ}C$ , [5 h]

I 
$$\frac{\text{Me}}{\text{A}}$$
  $\frac{\text{Ph}}{\text{R}^2}$   $\frac{\text{Ph}}{\text{N}}$   $\frac{\text{Ph}}{\text{R}^2}$   $\frac{\text{Ph}}{\text{R}^2}$   $\frac{\text{Ph}}{\text{R}^2}$   $\frac{\text{Ph}}{\text{R}^2}$   $\frac{\text{N}}{\text{R}^2}$   $\frac{\text{R}^2}{\text{R}^2}$   $\frac{\text{Ph}}{\text{R}^2}$   $\frac{\text{N}}{\text{R}^2}$   $\frac{\text{R}^2}{\text{R}^2}$   $\frac{\text{Ph}}{\text{R}^2}$   $\frac{\text{N}}{\text{R}^2}$   $\frac{\text{N}}{\text{N}}$   $\frac{\text{N}}{\text{N}}$ 

d R3: -Ph

84%

$$I \xrightarrow{R^{3} - Et \ (XIV)} A) \xrightarrow{Ph \ O \ R^{3}} I \xrightarrow{Me \ NR^{5} \ (XVI)} A) \xrightarrow{Ph \ O \ Me} XV XVII$$

$$a \ R^{3}: -Me \ 90\% \qquad a \ R^{4}. \ R^{5}: -Me \ 88\%$$

$$b \ R^{3}: -Et \ 78\% \qquad b \ R^{4}. \ R^{5}: -Et \ 82\%$$

$$c \ R^{3}: -iPr \ 51\% \qquad c \ R^{4}: -Ph; \ R^{5}: -H \ 61\%$$

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