REACTION OF 5-ARYL-2,3-DIHYDROFURAN-2,3-DIONES WITH KETENE ACETALS

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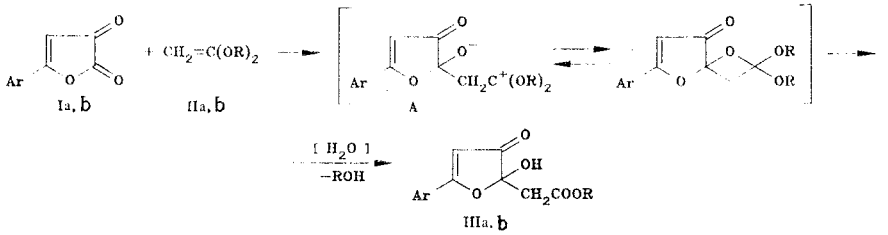
It is known that ketene acetals react with N-substituted isatins to give [2 + 2]-cycloadducts involving the ketone carbonyl group [1]. Other five-membered 2,3-dioxo heterocycles have not been previously investigated in this sort of reaction.

We have established that 5-aryl-2,3-dihydrofuran-2,3-diones Ia, b react with ketene diethylacetal (IIa) and ketene di-n-butylacetal (IIb) to give alkyl 5-aryl-2-hydroxy-3-oxo-2,3-dihydrofuran-2-ylethanoates IIIa, b.



I, III a Ar = C_6H_5, b Ar = p-$CH_3C_6H_4$; a R = C_2H_5, b R = n-C_4H_9

A possible scheme for the formation of esters III includes attack on the lactone carbonyl group of I by ketene acetal II, the formation of a zwitter ion (A) or a spiro compound (B), which is characteristic for aldehydes or ketones [2, 3], and their hydrolysis under the experimental conditions.

Ethyl 2-Hydroxy-3-oxo-5-phenyl-2,3-dihydrofuran-2-ylethanoate (IIIa). A mixture of 1.74 g (0.01 mole) of furandione Ia and 1.74 g (0.015 mole) of acetal IIa was refluxed for 1.5 h in 30 ml of methylene chloride, after which the solvent was evaporated, and the residue was recrystallized from acetonitrile to give 1.70 g (65%) of a product with mp 112-114°C. IR spectrum: 3120 (O—H), 1730 ($COOC_2H_5$), 1683 cm^{-1} [$C_{(3)}$=O]. PMR spectrum ($CDCl_3$): 1.22 (3H, t, CH_3CH_2O); 2.63, 3.05 (2H, dd, J ≈ 16 Hz, CH_2); 4.18 (2H, q, CH_3CH_2O); 5.88 (1H, s, 4-H); 6.28 (1H, broad s, OH); 7.52 ppm (5H, m, C_6H_5). Mass spectrum, m/z (I, %): 262 (18) $[M]^{+\cdot}$, 234 (36) $[M - CO]^{+\cdot}$, 217 (24) $[M - OC_2H_5]^+$, 189 (33) $[M - COOC_2H_5]^+$, 147 (42) $[C_6H_5COCH_2CO]^+$, 120 (3) $[C_6H_5COCH_3]^{+\cdot}$, 115 (50) $[OC—CH_2COOC_2H_5]^+$, 105 (96) $[C_6H_5CO]^+$, 102 (100) $[C_6H_5C{\equiv}CH]^{+\cdot}$, 87 (70) $[CH_2COOC_2H_5]^+$.

n-Butyl 2-Hydroxy-3-oxo-5-(p-tolyl)-2,3-dihydrofuran-2-ylethanoate (IIIb). This compound was similarly obtained in 30 ml of chloroform and had mp 120-122°C. IR spectrum: 3110 (O—H), 1732 ($COOC_4H_9$), 1670 cm^{-1} [$C_{(3)}$=O]. PMR spectrum ($CDCl_3$): 0.85 (3H, t, $CH_3CH_2CH_2CH_2O$); 1.42 (4H, m, $CH_3CH_2CH_2CH_2O$); 2.32 (3H, s, CH_3); 2.55, 2.98 (2H, dd, J ≈ 16 Hz, CH_2); 4.05 (2H, t, $CH_3CH_2CH_2CH_2O$); 5.78 (1H, s, 4-H); 6.22 (1H, broad s, OH); 7.30 ppm (4H, q, C_6H_4). The yield was 60%.

The results of elementary analysis were in agreement with the calculated values.

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