Electrochemical Synthesis of 3,5-Diphenyl-2(3H)-furanone Fructuoso Barba*, M. Desamparados Velasco and Antonio Guirado

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From electrochemical reduction of phenacyl bromide, 3,5-diphenyl-2(3H)-furanone was obtained as a side-product and its structure was determined by chemical and spectral techniques.

J. Heterocyclic Chem., 19, 669 (1982).

We have previously reported that the electrochemical reductions of phenacyl bromides leads to 2,4-disubstituted phenylfurans in good yield (1). In this reaction we have found as a side product, a small amount (15%) of a butenelactone that was extremely insoluble in usual solvents. The spectral data are in good agreement with the 3,5-diphenyl-2(3H)-furanone structure. In spite of the low yield, we believe that this method for its preparation is very interesting, since it opens a new route to this otherwise less accessible compound.

The reported preparation of this product is the treatment of 4-oxo-2,4-diphenylbutanoic acid with acetic anhydride to give a good yield of 4,5-diphenyl-2(5H)-furanone with a small amount of 3,5-diphenyl-2(3H)-furanone (2,3). However in some cases only the 4,5-diphenyl-2(5H)-furanone is mentioned (4).

It has been suggested that the 3,5-diphenyl-2(3H)-furanone might be a dimer because of insolubility and because the high melting point 284-286° (lit (2,3) 283-285° and 288°), but now there is evidence from the mass spectrum that confirms the monomeric nature.

It is known that the 4,6-diphenyl-2-pyrone reacts with hydrazine to form 1-amino-4,6-diphenyl-2-pyridone (5-7). The structure of our product is similar to the 4,6-diphenyl-2-pyrone and the fact that we obtained compound II with hydrazine could be considered as a confirmation of the proposed structure.

In spite of the insolubility of the furanone, the reaction was successful and 1-amino-3,5-diphenyl-2-pyrrolone was obtained.

$$Ph \longrightarrow 0 \longrightarrow 0 \longrightarrow N_2H_4 \longrightarrow Ph \longrightarrow NH_2$$

$$I \longrightarrow II$$

EXPERIMENTAL

Electrolysis was carried out in a cell with the compartment separated by a porous glass diaphragm. The temperature was controlled at 20°, and stirring was magnetic. A mercury pool was used as the cathode and a platinum plate as the anode. For prevention of the accumulation of acid in the anode compartment, 3 g of anhydrous sodium carbonate was put on the glass diaphragm. Also, 2 g of solid sodium thiosulphate was added for *in situ* neutralisation of the electrogenerated bromine.

The solvent-supporting electrolyte system was performed with 1.06 g (10⁻² mole) of lithium perchlorate in 50 ml of dry dimethylformamide. A solution of 1.99 g (10⁻² mole) of phenacyl bromide was electrolyzed under a cathode potential of -1.00 V (vs saturated calomel electrode). The coulometric measurements were made with an electronic integrator Amel model 558 coupled to the Amel potentiostat model 557. The electrolysis was continued until the current decreases to less than 1% of the starting value. The electron consumption was 1 F. mole⁻¹.

The cathode solution was worked up by evaporation of the solvent to dryness at 60° under reduced pressure. The residue was extracted with ether and washed with water (2 \times 50 ml) to remove inorganic salts. The solution is stored for one week and a white precipitate, extremely insoluble, was collected by vacuum filtration.

3,5-Diphenyl-2(3H)-furanone (I).

This compound had the following physical properties: ir (potassium bromide): ν max 3080, 3020, 1758, 1490, 1445, 1150, 1095, 995, 962, 788, 730, 690 cm⁻¹; ms: m/e (relative intensity): 236 (M⁺, 6), 235 (14), 192 (2), 179 (5), 178 (13), 131 (8), 105 (100), 102 (50), 102 (50), 77 (88), 76 (38), 51 (79), 50 (46).

Anal. Calcd. for C₁₆H₁₂O₂: C, 81.36; H, 5.08. Found: C, 81.41; H. 5.09. l-Amino-3,5-diphenyl-2-pyrrolone (II).

This compound had the following physical properties: ir (film): ν max 3290, 3050, 3020, 1660, 1600, 1480, 1450, 1040, 760, 690 cm⁻¹; ms: m/e (relative intensity) 250 (M*, 17), 232 (5), 191 (7), 119 (20), 115 (15), 105 (13), 104 (26), 91 (20), 77 (54), 51 (44), 27 (100).

Anal. Calcd. for C₁₆H₁₄N₂O: C, 76.80; N, 11.20; H, 5.60. Found: C, 76.86; N, 11.21; H, 5.61.

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