

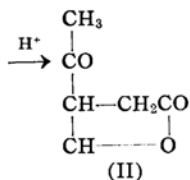
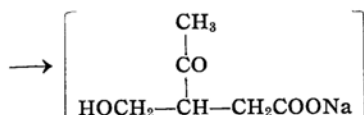
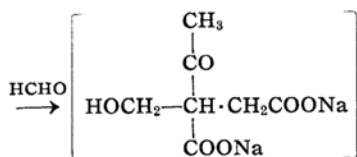
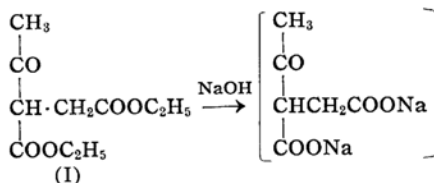
*The New Synthetic Method of  
β-Acetyl-butylolactone*

By Yoshihiko NISHIZAWA

(Received October 3, 1959)

B. R. Baker<sup>1)</sup> has prepared β-acetyl-butylolactone (II) from itaconic acid half ester or dimethyl methoxy-succinate. This method, however, gave a very poor yield and β-acetyl-butylolactone (II) was only isolated as 2,4-dinitrophenylhydrazone derivative.

The present author found that β-acetyl-butylolactone (II) is synthesized in a smooth way from diethyl acetyl-succinate<sup>2)</sup> (I) according to the following equation<sup>3)</sup>.



One hundred and eight grams (0.5 mol.) of diethyl acetyl-succinate (I) (b. p. 140~142°C/14 mmHg,  $n_D^{16}$  1.4380) were added to 480 ml. of 10% sodium hydroxide aqueous solution at 0°C and left for twenty four hours at room temperature (ca. 15~20°C). After the oil was dissolved completely, the aqueous solution was neutralized with 30% sulfuric acid to pH 8~8.2 and then 20 g. of sodium bicarbonate and 54 ml. of 37% formaline were added. After the mixture was allowed to stand overnight at room temperature, acidified with 30% sulfuric acid and extracted continuously with ether. The ether layer was dried with anhydrous sodium sulfate and then ether was evaporated. The residual oil was distilled under the diminish pressure; yield, 28.0 g. (43.8%), b. p. 140~143°C/6 mmHg,  $n_D^{21}$  1.4600.

*Anal.* Found: C, 55.98; H, 6.41. Calcd. for  $\text{C}_6\text{H}_8\text{O}_3$ : C, 56.24; H, 6.29%.

2,4-Dinitrophenylhydrazone; m. p. 191~192°C (recrystallized from acetic acid).

*Anal.* Found: C, 46.61; H, 3.70; N, 18.20. Calcd. for  $\text{C}_{12}\text{H}_{12}\text{O}_6\text{N}_4$ : C, 46.76; H, 3.92; N, 18.18%.

The 2,4-dinitrophenylhydrazone was soluble in sodium bicarbonate aqueous solution and the mixed melting point with the authentic 2,4-dinitrophenylhydrazone<sup>1)</sup> was not depressed.

The author is much indebted to Mr. S. Kitamura for his earnest assistance and wishes to express his thanks to his coworkers for the microanalysis.

Sumitomo Chemical Co., Ltd.  
Osaka Works  
Konohana-ku, Osaka

1) B. R. Baker, *J. Org. Chem.*, **17**, 116 (1952).

2) C. Rach, *Ann.*, **234**, 35 (1886).

3) Y. Nishizawa, Japan. Pat., No. 254,740.