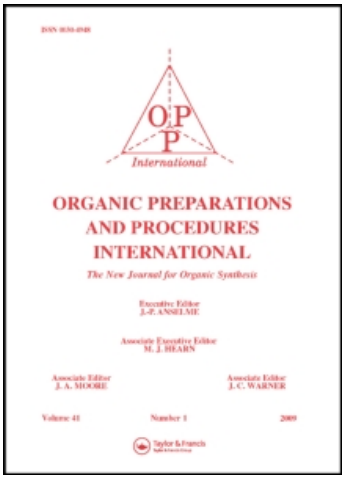

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A CONVENIENT SYNTHESIS OF 2(5H)-FURANONE

Ruzhen Cao[a]; Chong Liu[a]; Lunzu Liu[a]
[a] Institute of Elemento-Organic Chemistry, Nankai University, Tianjin, PR CHINA

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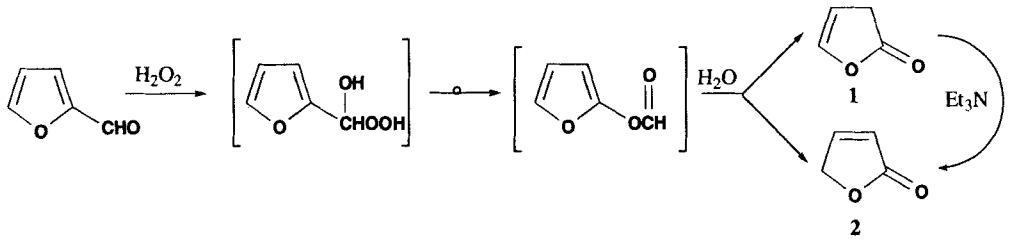
A CONVENIENT SYNTHESIS OF 2(5H)-FURANONE

Submitted by Ruzhen Cao, Chong Liu and Lunzu Liu*
(07/20/95)

Institute of Elemento-Organic Chemistry, Nankai University
Tianjin 300071, P. R. CHINA

2(5H)-Furanone (**2**) has found extensive use as an important chemical,[1] and recently has attracted considerable attention as a plant growth regulator,[2] antiulcer agent[3] and fish growth promoter.[4] It has been prepared by oxidation of furfural with hydrogen peroxide in the presence of formic acid,[5,6] thiourea[7] or cobaltous oxide.[8] Although these methods are one-pot reactions, they are deficient in some respects: the reagents are more expensive, the handling is inconvenient and perhaps most importantly the yield is much lower. We now describe a convenient method which involved refluxing furfural and hydrogen peroxide in dichloroethane and sodium sulfate. This reaction pathway may be described as shown in below.



The oxidation afforded a mixture of 2(3H)-furanone (**1**) and 2(5H)-furanone (**2**) which was separated by distillation. Isomerization of **1** with Et_3N afforded **2** in high yield. In order to avoid hydrolysis of **2** to organic acids, the reaction was carried out in a two phase solution (dichloroethane-sat. Na_2SO_4) to give **2** in good yield. However, with saturated sodium chloride a very low yield of **2** was obtained presumably due to increased hydrolysis.

EXPERIMENTAL SECTION

^{1}H NMR spectra were recorded on a JEOL FX-90Q spectrometer with TMS as the internal standard.

Preparation of 2(5H)-Furanone (2).- In a 1L four-necked flask fitted with a reflux condenser, a mechanical stirrer, a thermometer and a dropping funnel were placed redistilled furfural (96g, 1mol), dichloroethane (200mL) and sodium sulfate (120g). Then 30% hydrogen peroxide (220mL, 2.13mol) was added dropwise with stirring at 60~70° over a period of 2 hrs. After addition, the reaction mixture was heated to reflux for 10 hrs, then cooled to room temperature and filtered. The organic phase of the filtrate was separated, and the aqueous layer was extracted with dichloroethane (2 x 50mL). Triethylamine (2~4g) was added to the combined dichloroethane layers containing **1** and **2**. The mixture was stirred for 2 hrs at 40°. After removal of the dichloroethane, the residual liquid was distilled *in vacuo*

to give **2** (56g, yield 67%). To separate compounds **1** and **2**, the dichloroethane layer was concentrated on a rotary evaporator and the residual liquid was distilled *in vacuo* to give 2(3H)-furanone (15g, yield 18%) and 2(5H)-furanone (31g, yield 37%).

2(3H)-Furanone (**1**): bp. 60-62°/40mmHg. ^{1}H NMR($CDCl_3$): δ 3.10(t, 2H, ^3J = 2.4, ^4J = 2.4, 3-H), 5.52(dt, 1H, ^3J = 3.6, ^4J = 2.4, 5-H), 6.74(dt, 1H, ^3J = 2.4, ^3J = 3.6, 4-H).

Anal. Calcd for $C_4H_4O_2$: C, 57.14; H, 4.76. Found: C, 57.01; H, 4.62

2(5H)-Furanone (**2**): bp. 98-100°/40mmHg. ^{1}H NMR($CDCl_3$): δ 4.88(dd, 2H, ^4J = 2.2, ^3J = 1.7, 5-H), 6.10(dt, 1H, ^4J = 2.2, ^3J = 5.8, 3-H), 7.58(dt, 1H, ^3J = 1.7, ^3J = 5.8, 4-H).

Anal. Calcd for $C_4H_4O_2$: C, 57.14; H, 4.76. Found: C, 57.10; H, 4.71

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