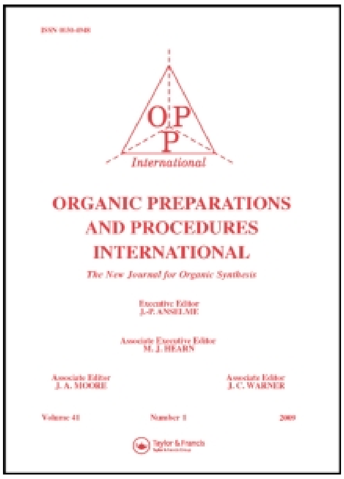

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# A CONVENIENT SYNTHESIS OF 2-ACETYLDIHYDROBENZOFURAN

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# A CONVENIENT SYNTHESIS OF 2-ACETYLDIHYDROBENZOFURAN

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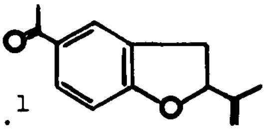
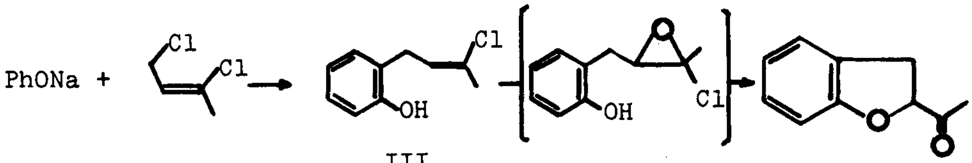


I

Natural products containing isoprene units as a 2-isopropenyldihydrobenzofuran moiety such as tremetone, are quite abundant.[1] A total synthesis of I[2] involves 2-acetyldihydrobenzofuran(II) as a crucial intermediate, prepared in four steps from salicylaldehyde. We now describe a convenient synthesis of synthon II from phenol.

ortho-Alkylation of sodium phenoxide with 1,3-dichlorobut-2-ene in benzene readily gave III. Epoxidation of the double bond of III was the accomplished with m-chloroperbenzoic acid. The resulting epoxide was not isolated and



III

II

apparently underwent ring opening via internal attack by the phenolic hydroxy group to give II directly. Although the overall yield of this sequence is only moderate(23%), the operational simplicity and the low cost of the materials makes the present procedure an advantageous route to II.

## EXPERIMENTAL

2-(3-Chlorobut-2-enyl)phenol(III).- A suspension of sodium phenoxide was prepared by the addition (15 min.) of a solution of 9.0g.(0.096 mole) of phenol in 200 ml. of benzene to 4.5g. of sodium hydride(57% dispersion in mineral oil), previously washed with small portions of dry benzene and contained in a 500 ml. flask. The 1,3-dichlorobut-2-ene(17.5g., 0.14 mole) was added as a thin film from a dropping funnel and the mixture was heated under reflux for 24 hrs. with continuous stirring. It was cooled and extracted with 2N sodium hydroxide. The alkaline extract was acidified and extracted with ether. The dried($MgSO_4$) ethereal extract was evaporated to dryness and the residual oil was distilled to give 8.22g. (47%) of III, bp. 104-106°/0.4 Torr; IR(neat): 3600-3200, 1670, 1620, 1600 $cm^{-1}$; nmr($CCl_4$): $\tau$ 2.8-3.5 (4H,m,arH), 4.42 (1H,t, J = 7.5 Hz with further splittings, =CH-), 6.57 (2H,d, J = 7.5 Hz, $ArCH_2$), 7.95 (3H,m,$CH_3$).

Anal. Calcd. for $C_{10}H_{11}ClO$: C, 65.75; H, 6.03
Found: C, 65.74; H, 6.06

2-Acetyldihydrobenzofuran(II).- A solution of III (518mg., 2.83 mmole) in 10 ml. of chloroform was treated with 1.0g. of m-chloroperbenzoic acid. The reaction mixture was allowed to stand 20 hrs. at room temperature and then diluted by the addition of 50 ml. of ether; m-chlorobenzoic acid was removed by extraction with aqueous sodium bicarbonate. The ethereal solution was then shaken briefly with 1N sodium hydroxide, acidified and the layers separated. The dried($MgSO_4$) organic phase was evaporated and the residue was distilled to yield 218mg.(48%) of II, bp. 55-58°/0.4 Torr, lit.[2] bp. 60-62°/ 0.5 Torr; IR($CH_2Cl_2$): 1710 $cm^{-1}$; nmr($CCl_4$): τ 2.8-3.4 (4H,m, arH), 5.13 (1H,t, J = 8.5 Hz, OCH ), 6.74 (2H,d, J = 8.5 Hz, $ArCH_2$), 7.84 (3H,s,$COCH_3$).

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