

THE OXIDATION OF FURAN DERIVATIVES WITH PYRIDINIUM CHLOROCHROMATE: A NOVEL SYNTHESIS OF 6-HYDROXY-2 H-PYRAN-3 (6 H)-ONES

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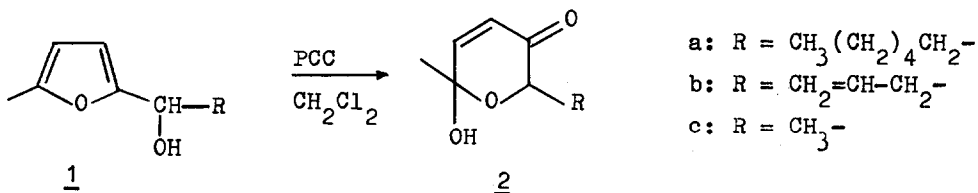
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As recently reported, we have been studying the transformation of 2-furylcarbinols under various conditions: in particular, treatment of these furan derivatives in acidic medium led to the formation of isomeric cyclopentenones (1) and an application of this reaction in the prostaglandin field yielded some interesting intermediates (2).

We wish now to describe a novel and useful oxidation of 5-methyl-2-( $\alpha$ -hydroxyalkyl)-furans 1 to 6-hydroxy-2 H-pyran-3 (6 H)-ones 2 with pyridinium chlorochromate (PCC): this reagent (at present utilized for the oxidation of primary and secondary alcohols to carbonyl compounds) shows a unusual behaviour, as dienophile and oxidant, in the ring enlargement of 2-furylcarbinols 1 to pyran derivatives 2. Furthermore, these compounds are very interesting, since they present antimicrobial and anticoccidial properties (3), and several patent applications have been filed on the subject in the United States (4).

5-methyl-2-furylcarbinols 1a-c (1 g in 10 ml of  $\text{CH}_2\text{Cl}_2$ ), easily obtained through the usual procedure of a Grignard reaction, were rapidly added to a suspension of PCC (4 g) in  $\text{CH}_2\text{Cl}_2$  (20 ml) at room temperature. The reaction was followed by TLC and was complete after 1 h. The crude product, isolated as described by Corey (5), was purified by chromatography on  $\text{SiO}_2$ .

The elution with 2:1 benzene-diethyl ether afforded the pure pyran derivatives 2a-c, as oils, in excellent yields (>90%).



The infrared spectra (neat) showed the presence of an alcoholic function at 3400 cm<sup>-1</sup> and a conjugated ketone at 1690 cm<sup>-1</sup>. <sup>1</sup>H-NMR data (in CCl<sub>4</sub>) confirmed the pyran structure of these derivatives and were completely in agreement with the ones reported for similar compounds (6); for ex., 2c showed signals at 6.70 δ (C<sub>5</sub>-H, d, J=10 Hz), 5.90 δ (C<sub>4</sub>-H, d, J=10 Hz), 4.53 δ (C<sub>2</sub>-H, q, J=6.5 Hz), 1.55 δ (C<sub>6</sub>-Me, s), 1.30 δ (C<sub>2</sub>-Me, d, J=6.5 Hz) (7).

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#### References and remarks

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- 7) The analytical and spectroscopic data (MS, IR, and <sup>1</sup>H-NMR) were completely in accord with the proposed structures for all prepared compounds.