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Publisher *Taylor & Francis*

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## Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t902189982>

### AN IMPROVED SYNTHESIS OF 2'-HYDROXYBIPHENYL-2-CARBOXYLIC ACID LACTONE

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**To cite this Article** Gringauz, Alex and Tosk, Eugene(1970) 'AN IMPROVED SYNTHESIS OF 2'-HYDROXYBIPHENYL-2-CARBOXYLIC ACID LACTONE', *Organic Preparations and Procedures International*, 2: 3, 185 – 187

**To link to this Article:** DOI: 10.1080/00304947009458655

**URL:** <http://dx.doi.org/10.1080/00304947009458655>

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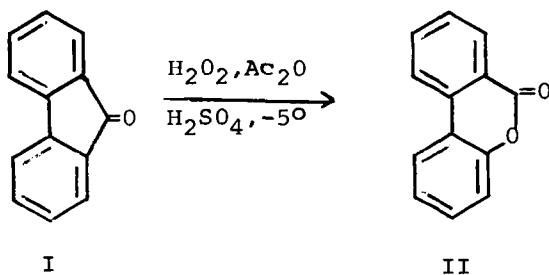
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AN IMPROVED SYNTHESIS  
OF  
2'-HYDROXYBIPHENYL-2-CARBOXYLIC ACID LACTONE

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The Baeyer-Villiger oxidation of ketones to the corresponding esters has undergone various improvements since its original introduction<sup>1</sup>. We required relatively large quantities of the title compound (II) for the preparation of certain biphenyl derivatives.



The oxidation of 9-fluorenone (I) appeared to us more promising than coupling phenol to diazotized anthranilic acid.<sup>2</sup> Wittig and Pieper<sup>3</sup> describe a small scale (10 g) oxidation of I with apparently 30%  $\text{H}_2\text{O}_2$  in a mixture of acetic anhydride-conc.  $\text{H}_2\text{SO}_4$  in the cold for 48 hours. The yield of II was poor, accompanied by a 76% recovery of starting

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material. The workup was complicated by the necessity of separating the lactone from the ketone. Emmons and Lucas<sup>4</sup> utilizing peroxytrifluoroacetic acid and  $\text{Na}_2\text{HPO}_4$  at reflux in  $\text{CH}_2\text{Cl}_2$  describe the oxidation of a series of ketones to the corresponding esters in good yields (53-88%). These were also carried out on a small scale (0.2 mole). Sager and Duckworth<sup>5</sup> similarly report the oxidation of cyclopentanone and cyclohexanone in good yields.

We were able to oxidize I in good yields without requiring large quantities of expensive trifluoroacetic anhydride, inorganic phosphates as neutralizing agents or potentially hazardous elevated temperatures. In our hands oxidations of up to 2 moles of I were carried out without difficulty.

#### EXPERIMENTAL

2'-Hydroxybiphenyl-2-carboxylic acid lactone. To a solution of 135 g of conc.  $\text{H}_2\text{SO}_4$  and 350 g of acetic anhydride there was slowly added with stirring and cooling 55 ml. of 90%  $\text{H}_2\text{O}_2$ .<sup>6</sup> The temperature was maintained below  $15^\circ$ . To this oxidation mixture a solution of 100 g. (0.56 mole) of 9-fluorenone in 100 ml. of  $\text{CH}_2\text{Cl}_2$  was added and stirring continued for 24 hours at  $-5^\circ$ .<sup>7</sup> Addition of 500 ml. of  $\text{H}_2\text{O}$  and subsequent boiling 1-2 hours destroyed excess acetic anhydride and peroxides and removed the  $\text{CH}_2\text{Cl}_2$ . The solid which precipitated on cooling was collected and dissolved in the combined ethereal extracts (3x100 ml.) from the supernatant

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aqueous phase. The ethereal solution was washed with 5%  $\text{Na}_2\text{CO}_3, \text{H}_2\text{O}$ , then brine and finally dried ( $\text{Na}_2\text{SO}_4$ ). Evaporation of the solvent (steam bath or flash evaporator) yielded 96.0 g (88.5%) of crude lactone, m.p. 87-89.5°. Two recrystallizations from ethanol (with Norite) afforded 86.2 g (80%) of fine white crystalline needles, m.p. 93.0-94.0° (lit.<sup>3</sup> 94.5°).

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2. C. Graebe and P. Schestakow, Ann. 284, 306 (1895).
3. G. Wittig and G. Pieper, Ber. 73, 295 (1955).
4. W.D. Emmons and G.B. Lucas, J. Am. Chem. Soc., 77, 2287 (1955).
5. W.F. Sager and A. Duckworth, *ibid.*, 77, 188 (1955).
6. Donation of a generous sample from the FMC Corporation, Inorganic Chemicals Division, New York, N.Y. 10017 is hereby acknowledged. Caution - A very strong oxidizing agent, potentially explosive when mixed with certain organic compounds. Keep away from heat and light.
7. Forma-Temp Jr. Bath, Forma Scientific, Inc., Marietta, Ohio

(Received March 13, 1970)