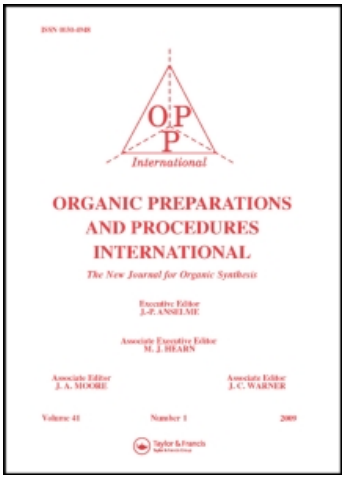

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AN IMPROVED SYNTHESIS OF 2'HYDROXYBIPHENYL-2-CARBOXYLIC ACID LACTONE

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

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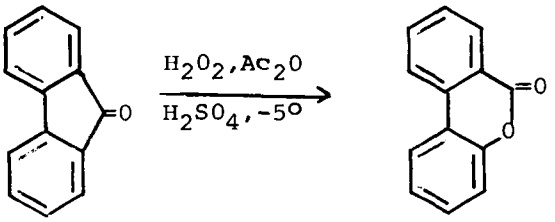
The Baeyer-Villiger oxidation of ketones to the corresponding esters has undergone various improvements since its original introduction[1]. We required relatively large quantities of the title compound (II) for the preparation of certain biphenyl derivatives.



I II

The oxidation of 9-fluorenone (I) appeared to us more promising than coupling phenol to diazotized anthranilic acid.[2] Wittig and Pieper[3] describe a small scale (10 g) oxidation of I with apparently 30% H_2O_2 in a mixture of acetic anhydride-conc. H_2SO_4 in the cold for 48 hours. The yield of II was poor, accompanied by a 76% recovery of starting

material. The workup was complicated by the necessity of separating the lactone from the ketone. Emmons and Lucas[4] utilizing peroxytrifluoroacetic acid and Na_2HPO_4 at reflux in CH_2Cl_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[5] 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 tp 2 moles of I were carried out without difficulty.

EXPERIMENTAL

2'-Hydroxybiphenyl-2-carboxylic acid lactone. To a solution of 135 g of conc. H_2SO_4 and 350 g of acetic anhydride there was slowly added with stirring and cooling 55 ml. of 90% H_2O_2.[6] The temperature was maintained below 15°. To this oxidation mixture a solution of 100 g. (0.56 mole) of 9-fluorenone in 100 ml. of CH_2Cl_2 was added and stirring continued for 24 hours at -5°.[7] Addition of 500 ml. of H_2O and subsequent boiling 1-2 hours destroyed excess acetic anhydride and peroxides and removed the CH_2Cl_2. The solid which precipitated on cooling was collected and dissolved in the combined ethereal extracts (3x100 ml.) from the supernatant

aqueous phase. The ethereal solution was washed with 5% Na_2CO_3,H_2O, then brine and finally dried (Na_2SO_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.[3] 94.5°).

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1. C.H. Hassall,"Organic Reactions", Vol.IX, John Wiley and Sons, Inc. New York, N.Y., 1957, p 73.
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

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