(0.01 mole) of the acid I and the mixture was heated at reflux for 24 hours. After removal of the methanol by distillation, the residue was heated under reflux for 1.5 hours with 70 cc. of a 10% aqueous solution of potassium hydroxide. On cooling, the insoluble oil solidified, was removed by filtration, washed and dried. There was obtained 1.5 g. of crude **1,1-di-(*p*-chlorophenyl)-ethylene** (II), m.p. 80–85°, which on recrystallization from pentane gave colorless prisms, m.p. 84–86°.[6] A mixture of it with an authentic specimen of 1,1-di-(*p*-chlorophenyl)-ethylene[7] melted at 84–86°.

Anal. Calcd. for $C_{14}H_{10}Cl_2$: C, 67.49; H, 4.05. Found: C, 67.75; H, 4.27.

The alkaline filtrate was acidified with hydrochloric acid, cooled and the precipitated **di-(*p*-chlorophenyl)-acetic acid** (III) (1.0 g., m.p. 163–167°) was removed by filtration, washed and dried. Recrystallization from 95% ethanol gave the pure acid, m.p. 165–166°.

Anal. Calcd. for $C_{14}H_{10}Cl_2O_2$: C, 59.81; H, 3.59. Found: C, 59.74; H, 3.45.

When mixed with an authentic specimen of III[8] it produced no depression of melting point.

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(6) O. Grummitt, A. C. Buck and E. I. Becker, THIS JOURNAL, **67**, 2265 (1945), report m.p. 84–86°.

(7) Kindly supplied by Prof. Grummitt.

(8) O. Grummitt, A. C. Buck and R. Egan, *Org. Syntheses*, **26**, 21 (1946).

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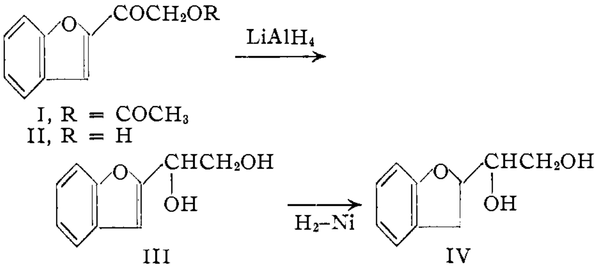
The Preparation of 2-(α,β-Dihydroxyethyl)-benzofuran and 2-(α,β-Dihydroxyethyl)-coumaran

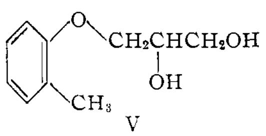
BY HAROLD E. ZAUGG

RECEIVED JULY 6, 1954

Shriner and Anderson[1] prepared ω-acetoxy-2-acetobenzofuran (I), but found that reduction of it either with sodium amalgam or with hydrogen in the presence of Adams catalyst resulted in cleavage to acetic acid and 2-acetobenzofuran. The present note reports the successful reduction of I with lithium aluminum hydride to 2-(α,β-dihydroxyethyl)-benzofuran (III), and further saturation of it by catalytic hydrogenation to 2-(α,β-dihydroxyethyl)-coumaran (IV), isolated as a mixture of diastereoisomers.



Compounds III and IV can be viewed as cyclized forms of the skeletal muscle relaxant, Mephenesin (V), formed by abstraction of hydrogen atoms from the *o*-methyl and α-methylene groups.



V

Both III and IV possess paralytic properties in mice; the activity of IV is comparable to that of V. In rabbits, however, IV is considerably less effective than Mephenesin.

As an incidental outcome of this work a by-product formed during the preparation of I was shown to be ω-hydroxy-2-acetobenzofuran (II) by its identity with an authentic sample prepared by treatment of ω-bromo-2-benzofuran with potassium formate.

Acknowledgment.—The author is indebted to Mr. Morris Freifelder for carrying out the catalytic hydrogenation, to Mr. E. F. Shelberg for the microanalyses, and to Dr. G. M. Everett for the pharmacological tests.

Experimental

2-(α,β-Dihydroxyethyl)-benzofuran (III).—To 2.7 g. (0.071 mole) of lithium aluminum hydride in 100 cc. of dry ether was added dropwise with stirring, a solution of 15.5 g. (0.071 mole) of ω-acetoxy-2-acetobenzofuran (I)[1] in 50 cc. of dry ether. The temperature was maintained below 10° until addition was complete, but the suspension was then stirred at room temperature for four hours. The mixture was decomposed by pouring carefully into ice containing an excess of hydrochloric acid. The ether was separated, washed with dilute sodium bicarbonate solution and dried over anhydrous magnesium sulfate. Filtration and removal of the ether by distillation gave 10.2 g. (82.5%) of product, m.p. 80–85°. Two recrystallizations from benzene gave 9.4 g. of III in the form of colorless leaflets, m.p. 87–88°.

Anal. Calcd. for $C_{10}H_{10}O_3$: C, 67.40; H, 5.66. Found: C, 67.48; H, 5.63.

2-(α,β-Dihydroxyethyl)-coumaran (IV).—A solution of 7 g. of III in 50 cc. of absolute ethanol was hydrogenated at 20 pounds initial pressure in the presence of 2.5 g. of Raney nickel. After filtration, and removal of the solvent by distillation, the residual oil was distilled *in vacuo* to give 4.6 g. (65%) of IV, b.p. 155–162° (1.5 mm.), $n^{25}D$ 1.5653. The product set to a semi-solid material which, after pressing between porous plates for several weeks, melted in the range 55–70°.

Anal. Calcd. for $C_{10}H_{12}O_3$: C, 66.65; H, 6.72. Found: C, 66.65; H, 6.54.

The wide boiling and melting ranges of this material indicate that it is probably a mixture of diastereoisomers of IV.

ω-Hydroxy-2-acetobenzofuran (II).—A solution of 19.1 g. of ω-bromo-2-acetobenzofuran[1] in 400 cc. of 50% aqueous ethanol was refluxed for two hours with 26 g. of potassium formate and 2 cc. of concentrated hydrochloric acid. Pouring into ice, filtering and drying the resulting precipitate gave 12.6 g. of crude yellow product. A recrystallization from one liter of water followed by another from 50 cc. of 95% ethanol gave 5.9 g. of shiny colorless leaflets, m.p. 129–130°.

Anal. Calcd. for $C_{10}H_8O_3$: C, 68.18; H, 4.58. Found: C, 68.08; H, 4.53.

This product proved, by mixed melting point determination, to be identical with the by-product obtained in only 2% yield during the preparation of I by the method of Shriner and Anderson.[1]

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(1) R. L. Shriner and J. Anderson, THIS JOURNAL, **61**, 2705 (1939).

The Bromination of Some N-Substituted Phthalimides with N-Bromosuccinimide

BY HAROLD E. ZAUGG

RECEIVED JULY 6, 1954

In 1898 Sachs[1] obtained N-bromomethylphthalimide in good yield by the high temperature (190°) bromination of N-methylphthalimide. However, N-ethylphthalimide, under the same conditions,

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(1) F. Sachs, *Ber.*, **31**, 1225 (1898).