N,N'-Di-*n*-butyl-*p*-phenylenediamine

By John V. Capinjola

This compound was prepared by essentially the same procedure as that described by Michaelis, Schubert and Granick[1] for the preparation of the corresponding methyl derivative.

Twenty-five grams of N,N'-di-*p*-toluenesulfonyl-*p*-phenylenediamine, obtained by the reaction of *p*-phenylenediamine with *p*-toluenesulfonyl chloride, was dissolved in dilute (3%) sodium hydroxide solution and treated with 55 g. of *n*-butyl sulfate. The mixture was heated under reflux for several hours, after which the alkylated product was isolated in the usual manner; yield 47%; recrystallized from methyl alcohol, m.p. 132–132.5°.

Anal. Calcd. for $C_{28}H_{36}O_4N_2S_2$: S, 12.14. Found: S, 12.19.

N,N'-Di-*n*-butyl-*p*-phenylenediamine was obtained by hydrolysis of 13.5 g. of the above compound at 90° with a mixture of 20 ml. of glacial acetic acid and 40 ml. of concentrated sulfuric acid; yield 91%. Treatment with activated carbon followed by sublimation *in vacuo* at 100° produced colorless crystals, m.p. 53.2–53.8°.

The identity of the product was confirmed by analysis of the dihydrochloride salt.

Anal. Calcd. for $C_{14}H_{28}N_2Cl_2$: Cl, 24.21. Found: Cl, 24.15.

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[1] Michaelis, Schubert and Granick, This Journal, **61**, 1989 (1939).

Ethyl Corporation
Research Laboratories
Detroit, Michigan

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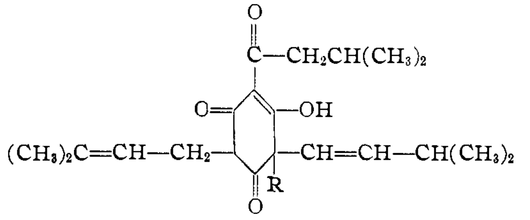
The Synthesis and Absorption Spectra of Some Analogs of Humulon[1]

By Tod W. Campbell and Galvin M. Coppinger

Humulon (I) and lupulon (II) are two substances isolated from hops (*Humulus lupulus*)

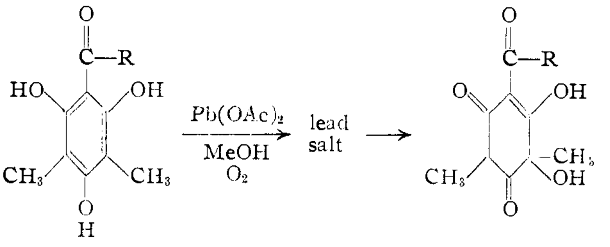


I, R = —OH
II, R = —CH=CH—CH(CH$_3$)$_2$

whose structures were elucidated by Wieland and Wollmer and their co-workers.[2]

In view of the recent interest in humulon and lupulon as antibiotics and tuberculostatic agents,[3] two analogs of humulon have been prepared, and their spectra compared with that of humulon. The similarity of the spectra is added confirmation for the structure of humulon itself, which has yet to be synthesized.

They were synthesized by acylating dimethylphloroglucinol by the Hoesch method[4] to give dimethylacylphloroglucinol (III). A methanol solution of this substance, in the presence of lead acetate, readily absorbed oxygen to give an insoluble lead salt (IV),[2] which was then decomposed with sulfuric acid to give (V), acyl-3,5-dimethyl-2,3-dihydroxy-4,6-diketocyclohexene-1.



III IV V

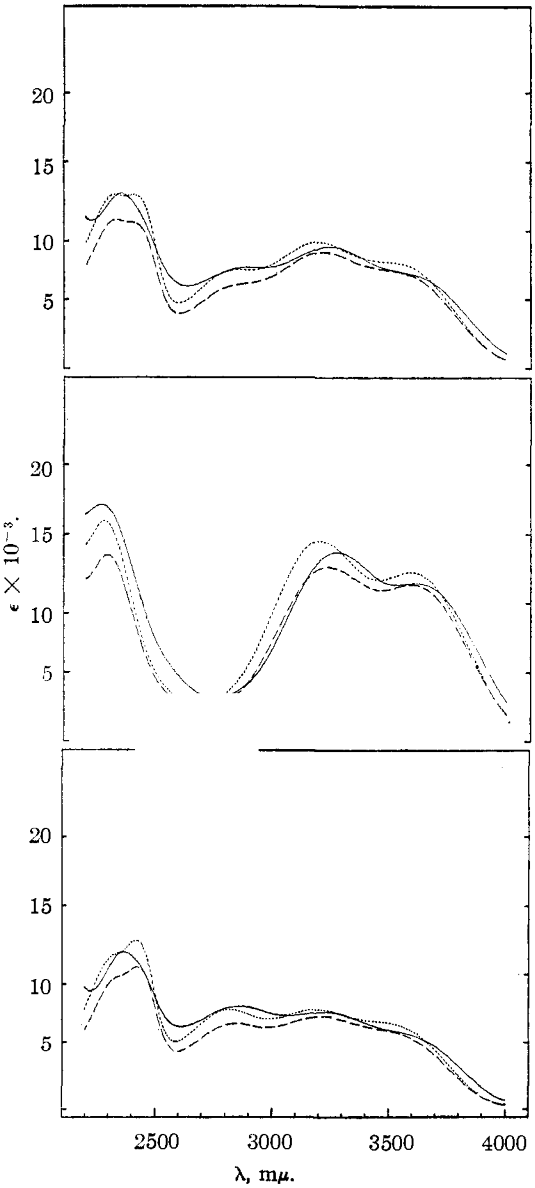


Fig. 1.—Absorption spectra: top, neutral; middle, basic; bottom, acidic; ——, humulon; — —, butyro-3,5-dimethyl-2,3-dihydroxy-4,6-diketocyclohexene-1; . . . aceto-3,5-dimethyl-2,3-dihydroxy-4,6-diketocyclohexene-1.

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[1] Article not copyrighted.

[2] H. Wieland, *Ber.*, **58**, 2012; 102 (1925); H. Wieland and E. Martz, *ibid.*, **59**, 2352 (1926); W. Wollmer, *Ber.*, **49**, 780 (1916); **58**, 672 (1925).

[3] (a) Y. C. Chin, A. A. Andersen, H. Hamilton, G. Alderton and J. C. Lewis, *Soc. Expt. Biol. Med. Proce.*, **70**, 158 (1949); (b) J. C. Lewis, *et al.*, AIC 231, Western Regional Research Laboratory, Bureau of Agricultural and Industrial Chemistry, U. S. Department of Agriculture.

[4] P. E. Spoerri and A. S. DuBois, *Org. Reactions*, **5**, 387 (1949).