trated solutions could be made in dioxane, but these were so hygroscopic that it was difficult to get meaningful measurements since in the instrument used some exposure of the solution to the air was unavoidable. The dipole moment of 8.9 ± 0.2 D. was obtained from the three most dilute solutions in benzene. These three points and the one for the solvent fell on a straight line, but the next point at $\omega_2 = 0.00088499$ fell considerably below the line and the next more concentrated solution precipitated slightly and was still further off the line. It is evident that association begins beyond a concentration of about 6 mg. in 10 ml. in this case. The dipole moment calculated from the dioxane solutions came out 8.1 D., but this is probably not so accurate as the value from the benzene solutions. The value of 8.9 D. is smaller than the value of 9.7 D. obtained by the previous workers and this might, in part, be accounted for if they calculated the MR_D value from tables. There is no doubt, however, that this molecule has a high moment. The value of 8.9 D. indicates that there is a contribution of about 27% from forms with a separation of charge, again no account being taken of the moment present in the normal form of the molecule. This is about the same as the contribution of the forms with a separation of charge in compound I.

Experimental

The dielectric constants were measured using a Dipolmeter Model DM 01. The dipole moments were calculated using the method and equation of Halverstadt and Kumler.[2] The dioxane was purified by refluxing with about one-hundredth of its volume of concentrated hydrochloric acid with an exit tube from the reflux condenser leading outdoors, neutralizing and drying with solid potassium hydroxide, refluxing over sodium, and fractionally distilling twice from sodium through a 30-plate column.

Thiophene-free benzene was refluxed over sodium wire and then fractionally distilled from a 30-plate column.

Compound I,[5] orange needles, had m.p. 74–75°; compound II,[3,6] golden yellow plates, decomposed above 200°. The infrared and ultraviolet-visible spectra of these substances were identical with those reported.[3,5,6]

Acknowledgment.—The author wishes to thank Dr. Jerome Berson for suggesting the measurements be made and for supplying pure samples[5,6] of the compounds, and Mr. Thomas Simpson, Jr., for programming the calculations on the 1620 IBM computer.

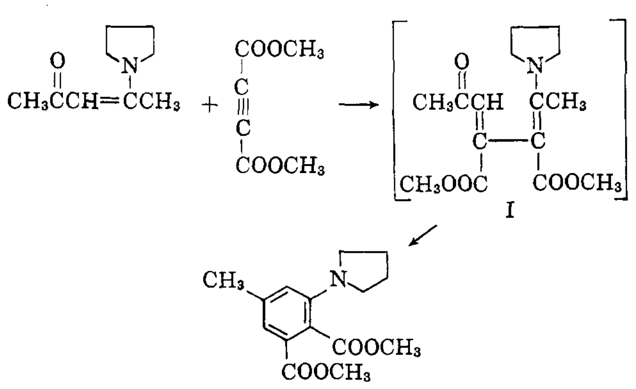
(6) J. A. Berson and E. M. Evleth, *Chem. Ind.* (London), 1362 (1961).

Communication TO THE EDITOR

A Method of Synthesis of the Benzene Ring

Sir:

The introduction by Stork and co-workers in 1954[1] of a new and useful method for the alkylation of carbonyl compounds awakened interest in the chemistry of enamines. A recent interesting new reaction of enamines was cited by Brannock.[2] He indicated that reaction with an acetylenecarboxylic acid ester interposes two carbon atoms between the α and β carbon of the enamine. This reaction, which we had been studying[3] independently, is capable of interesting and useful variations. One of these leads to a new synthesis of the benzene ring under mild conditions. The addition of dimethyl acetylenedicarboxylate to 4-pyrrolidino-3-penten-2-one dissolved in tetrahydrofuran results in a spontaneous reaction (presumably *via* I) with a temperature rise to 90°. The reaction mixture is diluted with water and the solid recrystallized from methanol to give dimethyl-4-methyl-6-pyrrolidinophthalate in 60% yield, m.p. 83–85°. [Calcd. for $C_{15}H_{19}NO_4$: C, 64.96; H, 6.91; N, 5.20. Found: C, 65.25; H, 6.92; N, 5.20. Ultraviolet spectrum in ethanol: λ_{max} 236 mμ (ε 16,400), 274 (7,950), and 347 (3,300).] Its structure is demonstrated by the presence of two isolated aromatic protons at 6.94 δ and 6.62 δ with indications of *meta* splitting and a three proton singlet at 2.27 δ due to an aromatic methyl group (A60, tetramethylsilane standard, deuteriochloroform solvent).



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(1) G. Stork, A. Brizzolara, H. Landesman, J. Szmuskovicz, and R. Terrell, *J. Am. Chem. Soc.*, **85**, 207 (1963), and preceding papers.

(2) K. C. Brannock, Abstracts of Papers, 140th National Meeting of the American Chemical Society, Chicago, Ill., September, 1961.

(3) A paper with M. M. Robison and L. Dorfman is in preparation on the reaction of enamines (especially of cyclic ketones) with acetylenecarboxylic acid esters and other dienophiles.