[CONTRIBUTION FROM THE SCHOOL OF CHEMISTRY AND PHYSICS OF THE PENNSYLVANIA STATE COLLEGE]

The Preparation of Certain Nitrogen-Substituted Sulfonanilides

BY GEORGE H. YOUNG

In connection with certain crystallographic studies being carried out in this Laboratory we have had occasion to prepare a number of nitrogen-substituted sulfonamides. The Hinsberg reaction[1] between "primary" sulfonamides and alkyl halides has been successfully applied to the synthesis of the *n*-propyl, isopropyl, and *s*-butyl N-substituted *p*-toluene sulfonanilides, three compounds which have not heretofore been described in the literature.

Molar quantities of the sulfonanilide and the appropriate alkyl bromide were caused to react in the presence of potassium hydroxide. The products were recrystallized from methanol, from which they deposited in the form of thick, colorless, monoclinic plates or tablets. Yields of 78–86% were obtained based on the crude air-dried product.

The pure compounds are in general insoluble in water, sparingly soluble in ether and ethyl acetate, and quite soluble in boiling methanol from which they are reprecipitated on cooling. They exhibit increased solubility in ethanol, acetic acid and acetone, and are very soluble in the higher alcohols and other conventional solvents. All are stable in air and melt sharply without decomposition.

The melting points and analytical data for these compounds are given in the table.

TABLE I

p-Toluene-sulfon-N-anilide		*n*-Propyl	Isopropyl[a]	*s*-Butyl
M. p., °C.		56	99.5–100	75.5
Formula		$C_{16}H_{19}O_2NS$	$C_{16}H_{19}O_2NS$	$C_{17}H_{21}O_2NS$
N	Calcd.	4.84	4.84	4.62
	Found	4.79	4.96	4.60
S	Calcd.	11.07	11.07	10.56
	Found	10.98	11.09	10.44

[a] Recrystallized first from 1:1 ethanol–ethyl acetate, then from methanol.

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[1] Hinsberg, *Ann.*, **265**, 178–192 (1891).

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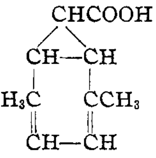
Studies on the Polymethylbenzenes. IX. Addition of Ethyl Diazoacetate to Durene[1,2]

BY LEE IRVIN SMITH AND PLINY O. TAWNEY

The action of diazoacetic ester upon aromatic hydrocarbons has been investigated by Buchner and his students,[3] who showed that the primary product of the reaction was the ester of a norcaradiene acid (I). These esters are characterized by a tendency to rearrange, especially at high temperatures, into derivatives of cycloheptatriene (II), phenylacetic acid (III), and β-phenylpropionic acid (IV), the latter rearrangement, of course, taking place only when the initial aromatic hydrocarbon contains one or more methyl groups.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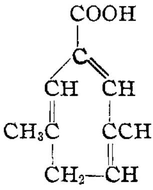
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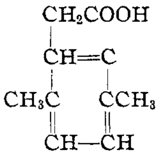
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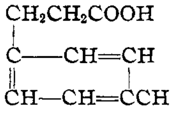
IV

In all the cases studied by Buchner, the addition proceeded in such a way that a norcaradiene con-

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[1] Abstracted from a thesis by Pliny O. Tawney, presented to the Graduate Faculty of the University of Minnesota, in partial fulfilment of the requirements for the degree of Master of Science, July, 1934.

[2] Presented at the 88th meeting of the American Chemical Society held at Cleveland, Ohio, September, 1934.

[3] (a) Buchner, *et al.*, *Ber.*, **18**, 2377 (1885); (b) *ibid.*, **34**, 982 (1901); (c) *ibid.*, **36**, 3502, 3509 (1903); (d) *Ann.*, **358**, 1 (1908); (e) *ibid.*, **377**, 259 (1910); (f) *Ber.*, **53**, 865 (1920).