

coefficient are both much greater than for a diffusion controlled current. A mechanism is proposed for this wave which is based on the catalysis

of hydrogen discharge by the $\text{Re}^{-1}\text{-Re}^{+1}$ couple.

CAMBRIDGE, MASSACHUSETTS

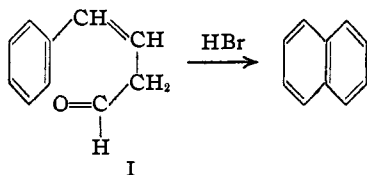
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NOTES

Cyclization of β -Styrylacetaldehyde

BY CHARLES K. BRADSHER¹

It has been reported by Rinkes² that β -styrylacetaldehyde (I) may be cyclized to give naphthalene. Inasmuch as he failed to report either the



yield or the method employed in this cyclization, we have undertaken to reinvestigate this matter.

The required aldehyde (I) was prepared by the more convenient method of Meyer,³ involving the pyrolysis of α -methoxycinnamylacetic acid. When refluxed with hydrobromic and acetic acids, the aldehyde gave naphthalene in 25% yield.

While this is the simplest case of aromatic cyclodehydration to give a naphthalene nucleus, there are a number of other instances in which a cyclization of this type is believed or known to take place.⁴

Experimental

Naphthalene.— β -Styrylacetaldehyde was prepared in 25% yield by the method of Meyer.³ This aldehyde (0.65 g.) was dissolved in acetic acid (13 cc.) and added dropwise to boiling 34% hydrobromic acid (6.5 cc.), the addition requiring about five hours. Refluxing was allowed to continue for a total of forty-eight hours. The mixture was then diluted with water and extracted with ether. The ethereal extract was washed, dried, concentrated and the residue sublimed *in vacuo*. The sublimate consisted of small white plates; m. p. 80–81°; yield, 0.14 g. (25%). This material showed no depression of melting point when mixed with an authentic sample of naphthalene.

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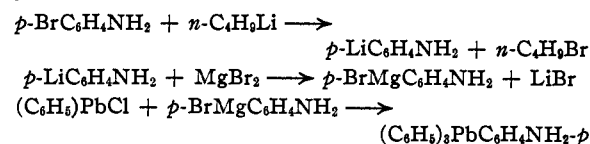
- (1) National Research Fellow (participating basis).
- (2) Rinkes, *Rec. trav. chim.*, **39**, 200 (1920).
- (3) Meyer, *Compt. rend.*, **204**, 508 (1937).
- (4) E. g. Zincke, *Ann.*, **240**, 137 (1887); Erlenmeyer and Kunlin, *Ber.*, **35**, 384 (1902); Haworth and Sheldrick, *J. Chem. Soc.*, 636, 1576 (1935); Omaki, *J. Pharm. Soc. Japan*, **58**, 4 (1938).

Diazotization of an Aminoaryllead Compound¹

BY HENRY GILMAN AND C. G. STUCKWISCH

p-Aminophenyllithium has been used for the introduction of the *p*-aminophenyl group into organoarsenic and organophosphorus compounds.² In attempts to effect a related transformation between triphenyllead chloride and *p*-aminophenyllithium, complex mixtures were obtained. The difficulty probably resides in the metal-metal interconversion reactions between organolead and organolithium compounds.³

However, because M–M interconversions only occur to a highly subordinated extent between Grignard reagents and organolead compounds, we first converted the RLi compound to the corresponding RMgBr compound by means of magnesium bromide.⁴ The following sequence of reactions illustrates the preparation of triphenyl-*p*-aminophenyllead, which was obtained in a 66% yield.



The *p*-aminophenyllead compound was diazotized by conventional procedures, despite the presence in the molecule of a supposed labilizing aminoaryl group. The diazonium compound was coupled with β -naphthol to give an azo-lead compound which was red in acid solution and green in basic solution. The general reactions described now provide the essential means for the resolution of some RM compounds, and the introduction of water-solubilizing groups to vary the lipid-water distribution of the less chemically reactive RM compounds.

- (1) Paper XLIV in the series "Relative Reactivities of Organo-metallic Compounds"; the preceding paper is in THIS JOURNAL, **63**, 2844 (1941).
- (2) Gilman and Stuckwisch, *ibid.*, **63**, 2844 (1941).
- (3) Gilman and Moore, *ibid.*, **62**, 3206 (1940).
- (4) Gilman and Kirby, *ibid.*, **63**, 2046 (1941).