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Alkylation–Reduction of Aromatic Ketones and Aldehydes. A Convenient Synthesis of Aromatic Hydrocarbons

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Summary Aromatic hydrocarbons are prepared from aromatic ketones and aldehydes by alkylation with an organolithium reagent followed by lithium–ammonia reduction in the same reaction vessel without isolation of intermediates.

WE report a convenient method for the synthesis of aromatic hydrocarbons from aromatic ketones and aldehydes. The advantages of the method which involves the alkylation–reduction of an aromatic ketone or aldehyde is that the entire sequence is carried out in the same reaction vessel

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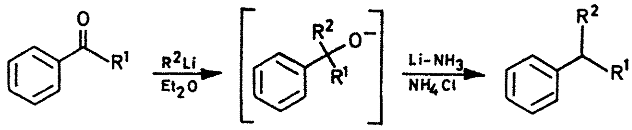
without isolation or purification of intermediates, the procedure consumes only a few hours, and the isolated yield of the aromatic hydrocarbon is excellent.



The general method is to introduce ammonia and lithium to a reaction vessel containing a benzyl alkoxide, generated *in situ* by the alkylation of an aromatic ketone or aldehyde with an organolithium reagent, and then to quench the resulting mixture with ammonium chloride. Recently we have shown that the reduction of a benzyl alkoxide in lithium-ammonia (ether) solutions does not occur until the ammonium chloride is added.[1]

The synthesis of 1,1-diphenylethane from benzophenone is described to illustrate the method. An ethereal solution (10 ml) of benzophenone (0·91 g, 5 mmol) is slowly added to a stirred solution of methyl-lithium† (5·5 mmol) in 15 ml of ether in a metal-ammonia reaction vessel. After 45 min *ca.* 25 ml of ammonia is distilled into the vessel, followed by 0·1 g (15 mmol) of lithium wire.‡ Then, with vigorous stirring, an excess of ammonium chloride (*ca.* 1·5 g) is added cautiously to discharge the dark-blue solution (*ca.* 5 min).§ Normal work-up and purification (alumina, petroleum) yielded a yellow liquid (95%) which was compared with an authentic sample.¶[2]

Other aromatic hydrocarbons synthesised by this method include: 1-methylindane (93%) from indan-1-one, *p*-t-butylethylbenzene (96%) from *p*-t-butylbenzaldehyde, and *p*-isopropylethylbenzene (93%) from *p*-isopropylbenzaldehyde.

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† Foote Mineral Co., Exton, Pa. 19341.

‡ Ventron Corp. (Alfa Inorganics), Beverly, Mass. 01915. The lithium wire was cut into nine pieces and rinsed in petroleum prior to addition.

§ For a convenient method of introducing the quenching agent see ref. 1b.

¶ The spectral and analytical data were also in agreement with the assigned structure. The most revealing were: n.m.r. (CCl_4) τ 8·46 (3H, d, 7 Hz), 5·97 (1H, q, 7 Hz), and 2·88 (10H, s); mass spectrum, m/e (rel intensity), 182 M^+ (32), 167 (100).

[1] (a) S. S. Hall, S. D. Lipsky, and G. H. Small, *Tetrahedron Letters*, 1971, **1853**; (b) S. S. Hall, S. D. Lipsky, F. J. McEnroe, and A. P. Bartels, *J. Org. Chem.*, 1971, **36**, 2588.

[2] J. S. Reichert and J. A. Nieuwland, *J. Amer. Chem. Soc.*, 1923, **45**, 3090.