proportionation caused the phosphorus analysis to be 0.5–0.6% too low.

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## The Behavior of Diphenylmagnesium Bromide with Benzoquinone

By David E. Worrall and Sumner Cohen

Bamberger and Blangey[1] got tars only by the action of methylmagnesium iodide on benzoquinone, although small amounts of several crystalline products were obtained from xyloquinone. Using duroquinone, Smith and Crawford[2] found that a non-tractable oil was the principal product with phenylmagnesium bromide. It occurred to us that the substitution of a heavier radical (diphenyl) not only would raise the melting point but might act as a brake on the reactivity, thereby facilitating the isolation of the primary addition product or products. Actually it increased the Wurtz-like effect inherent in the Grignard reagent and the principal products, aside from unchanged reactants, are dibiphenyl and hydroquinone.

### Experimental

The reagent was prepared from 0.1 g. mole of *p*-bromodiphenyl, using activated magnesium with a small volume of ether. An average of about 2 g. of dibiphenyl separated out, an amount that varied somewhat in different preparations. Benzoquinone (0.4 m. equiv.) dissolved in ether was added to the mixture. A dark blue precipitate formed, doubtless a molecular compound, that reacted with excess of reagent on shaking, losing its color and depositing a yellow oil that partially solidified. A permanent blue color was reached before all of the material was introduced. After decomposition with dilute acid the portion insoluble in ether and in water yielded 6.8 g. or 44% of crude dibiphenyl, which was recrystallized from nitrobenzene and identified by analysis. Hydroquinone was identified in the aqueous portion although most of the original quinone turned up as a tar after steam distillation of the ether layer. A mixture of 6–7 g. of diphenyl with bromodiphenyl was recovered by steam distillation. Slightly more than a gram of non-volatile material soluble in hot water and in alkali was isolated and identified as *p*-hydroxydiphenyl. About the same amount of hydrocarbon was obtained by treating the Grignard reagent with azobenzene. If the reactants in the original experiment were mixed rapidly or in the reverse order, little decolorization of the blue precipitate was observed and even after standing overnight no increase was noted in the quantity of hydrocarbon normally present in the reagent. No attempt was made to isolate addition products from the oil. Similar experiments with phenyl and *n*-butylmagnesium bromides failed to reveal any hydrocarbon formation from the action of benzoquinone. Small amounts of phenol and butyl alcohol from the action of atmospheric oxygen were formed.

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[1] Bamberger and Blangey, *Ann.*, **384**, 272 (1911).

[2] Smith and Crawford, This Journal, **50**, 869 (1928).

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# COMMUNICATIONS TO THE EDITOR

## *l*-EPHEDRINE, A DEGRADATION PRODUCT OF ALKALOIDS IN ACONITUM NAPELLUS

*Sir:*

We have been engaged in a study of degradation reactions of aconitine and its accompanying alkaloids in *aconitum napellus*. The latter, according to H. Schulze and G. Berger [*Arch. d. Pharm.*, **262**, 553 (1924)], is a mixture of the following bases: aconitine, benzoylaconine, homoisaconitine, aconine and neopollin. This material was available in the form of aconitin hydrochloricum, amorph. (Merck).

The destructive distillation of aconitine hydrochloride, amorph., over four to five its amount of crystalline barium hydroxide yields besides a large fraction of hydrocarbons and methylamine, a strongly basic oil which ultimately can be separated by steam distillation.

For purification it is distilled in a vacuum of 0.5 mm. and the fraction obtained at the bath temperature of 50–70° is collected. The analysis showed the presence of one methylimide group, no methoxy, a content of carbon, hydrogen and nitrogen corresponding to the formula $C_{11}H_{17}ON$. The mixture boils at 260–262° (uncorr.) it is slightly levorotatory and has a refractive index of $n^{20}$ 1.528. The total yield is 2%. *Anal.* Calcd. for $C_{11}H_{17}ON$: C, 73.74; H, 9.50; N, 7.82; (N)-$CH_3$, 8.37. Found: C, 74.52; H, 8.98; N, 7.70; (N)-$CH_3$, 7.82.

From this, besides other salts, a hydrochloride was obtained in crystalline form which, after