

An analytical sample, m.p. 174.5–175.5°, was prepared by crystallization from ethyl acetate.

Anal. Calcd. for $C_{22}H_{16}$: C, 94.25; H, 5.75. Found: C, 94.45; H, 5.64.

The hydrocarbon IV gave an unstable, purple picrate, m.p. 182–184°.

Ultraviolet absorption spectrum. The ultraviolet absorption spectrum of 9,10-dimethyl-3,4-benzopyrene in 95% ethanol was measured with a Model DU Beckman spectrophotometer. Maxima and ($\log \epsilon$) values are: 260 $m\mu$ (4.59), 268 $m\mu$ (4.70), 288 $m\mu$ (4.63), 300 $m\mu$ (4.70), 372 $m\mu$ (4.40), and 392 $m\mu$ (4.48).

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Vinyl-Alkali Metal Compounds¹

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Reaction of vinyl chloride with alkali metals in tetrahydrofuran has yielded the vinyl-alkali metal compounds, as might be expected from recent success in making the vinyl-Grignard reagent in that solvent.² The simplicity of the procedure makes it preferable to the exchange reaction through which vinylsodium was first made.³ Propenyllithium and several other alkenyllithium compounds have been made directly from the bromides in diethyl ether or petroleum ether,⁴ but all attempts to similarly prepare vinyl compounds have failed.

The example given below concerns vinylpotassium prepared from liquid 90% potassium-sodium alloy,⁵ but small-scale qualitative observations have shown that potassium, sodium, and lithium will each react.

Though the Grignard reagent is as easily made, vinylsodium or vinylpotassium may offer advantage on those occasions where the solid organometallic compound is needed, since they have little solubility and can be made solvent-free without decomposition by removal of solvent under reduced pressure. This operation leads to decomposition of the vinyl Grignard reagent.²

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As an example the reaction of vinyl Grignard solution with diethyl bromoborane gave only triethylborane, no trivinylborane, and evidence of polymers was seen. In contrast reaction of solid vinylpotassium gave some polymer, but trivinylborane and a mixture of ethylvinylboranes were obtained also.

The solvent should be removed from vinylpotassium (and presumably vinylsodium) as soon as possible because the solid reacts with the tetrahydrofuran. Were the solvent removed at once, the yield would probably be comparable with the best obtained by the exchange method.

EXPERIMENTAL

The vinyl chloride solution (95 g. in 405 g. tetrahydrofuran) was prepared by passing the gas through sodium hydroxide solution and a drying train into the solvent cooled to 0°. This was added slowly to 50 g. 90% potassium-sodium alloy covered with 350 g. tetrahydrofuran cooled to 0° C. in a 3-neck flask fitted with a Hershberg stirrer. A blue precipitate formed which reached a viscous gel-like consistency as the reaction proceeded.

A day after completion the preparation was assayed using a 3/808 g. aliquot from the well stirred slurry. Treatment with isopropyl alcohol in the vacuum apparatus gave 1.8 mmoles of ethylene with the correct vapor pressure, corresponding to a total vinylpotassium content of 485 mmoles or a yield of 32%. Five days later the solvent was removed under reduced pressure, and a similar assay of a 1.81/90 g. aliquot of dry powder gave a yield of 7.4%. Apparently reaction with the solvent had occurred.

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Reaction of Isocyanates with Tris(hydroxymethyl)aminomethane

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During the course of an investigation of certain urea derivatives it was necessary to prepare a series of 1-substituted-3-tris(hydroxymethyl)methylureas. Pierce *et al.*² described aromatic compounds of this type in which the substituents were phenyl, *o*- and *p*-tolyl, and 1- and 2-naphthyl. These compounds were prepared by reaction of equimolar quantities of isocyanate with tris(hydroxymethyl)aminomethane (I) in chloroform solution, but this method was disadvantageous in that I was insoluble in chloroform.

In addition to the phenyl and 1-naphthyl derivatives, made by the method of Pierce, a series of new derivatives has been prepared. It was found

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