

Di-n-Butylberyllium : a Novel Preparative Method

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WE report a new method for the preparation of *ether-free* alkyl- and aryl-beryllium compounds. In the usual preparative method, the organoberyllium is prepared from beryllium chloride and the corresponding Grignard reagent, but may be freed from the very tenacious ether only with difficulty.¹⁻⁴ The use of non-ethereal Grignard reagents⁵ appeared to present an alternate and direct solution to this problem. To illustrate the usefulness of the method, we have prepared di-n-butylberyllium⁶ and examined some of its physical properties.

A filtered solution, from n-butyl iodide and magnesium in benzene,^{5b} was treated with beryllium chloride (in slight excess with respect to the Bu-Mg content of the solution). The filtered product, free from halide and almost free from magnesium, contained beryllium equivalent to a 70% yield of Bu₂Be. The latter was isolated by distillation (ca. 110°, 0.008 m.m.), as a colourless viscous liquid (41% yield, density 0.82) which was dimeric (cryoscopically, 0.23 m in benzene).

The n.m.r. spectrum of Bu₂Be in benzene shows a normal n-propyl pattern and an α -methylene multiplet at $\tau = 9.62$, consisting of *at least eight lines*. Addition of an excess of diethyl ether transforms the multiplet into a well-defined *triplet*, shifted to higher fields. Although other alternatives are possible, we consider the multiplet to be due either to nonequivalence of the α -methylenes in the bridged and terminal positions of a dimeric structure,¹ or to nonequivalence of the individual α -methylene protons, as seen in certain hindered alkylmagnesium compounds.^{7,8}

The method described above appears to offer particular advantages for the preparation of *aryl*-berylliums. The method has yielded solutions of diphenylberyllium (72%), di-n-pentylberyllium (75%), diethylberyllium (35%), and di-*p*-tolylberyllium (90%), although none of the above has been isolated at this time.

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