

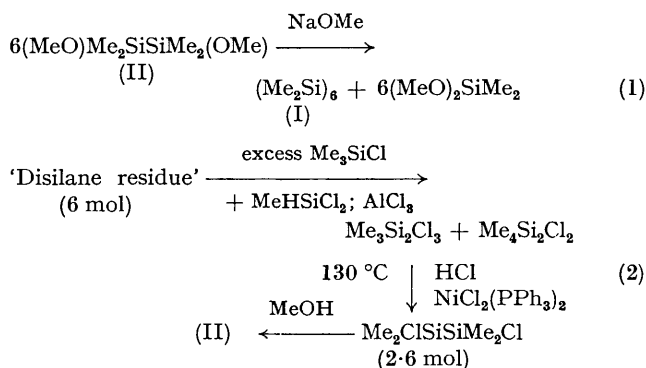
## One-step Synthesis of Dodecamethylcyclohexasilane

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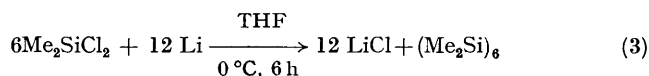
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**Summary** Dodecamethylcyclohexasilane (Me<sub>2</sub>Si)<sub>6</sub> has been conveniently prepared from Me<sub>2</sub>SiCl<sub>2</sub> and Li, in tetrahydrofuran at 0 °C.

DODECAMETHYLCYCLOHEXASILANE (I) is an interesting model compound, and several methods have been described for its synthesis.<sup>1-3</sup> Recently, Nagai *et al.*<sup>4</sup> described a new method [reaction (1)] for the synthesis of (I) (60% yield crude, 40% after recrystallization) from (II) which is not, however, commercially available. The route in reaction (2) was used to prepare (II) from 'disilane residue' (resulting from the industrial synthesis of Me<sub>2</sub>SiCl<sub>2</sub>).<sup>5</sup>



The route we propose [reaction (3)] is more convenient than the disilane route; from commercially available products, (I) is obtained in high yields (90% crude, 80% after recrystallization). Our process has several advantages



over previous organometallic methods; it gives very high yields and does not require the use of a catalyst (Ph<sub>3</sub>SiLi<sup>3</sup>) or the preparation of Na-K alloy.<sup>2</sup>

A typical procedure for the preparation of (I) is as follows. To a mixture of Li (1.6 g) in tetrahydrofuran (THF) (110 ml) at 0 °C was slowly added (1 h) Me<sub>2</sub>SiCl<sub>2</sub> (13 g) in THF (40 ml) with stirring under N<sub>2</sub>. The reaction mixture was stirred for a further 2 h at 0 °C and left overnight at room temperature; cyclohexane (100 ml) was then added and the precipitate filtered off. The THF and C<sub>6</sub>H<sub>12</sub> solvents were removed under reduced pressure, and the residue extracted with further cyclohexane (100 ml). After filtration and evaporation (I) was obtained (6 g, ca. 100%) and recrystallized from ethanol (4.6 g, 80%) in two crops (I), m.p. 250 °C, purity > 99%. Its physico-chemical properties were identical with those reported.<sup>2</sup>

(Received, 29th December 1977; Com. 1319.)

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<sup>2</sup> U. Graf zu Stolberg, *Angew. Chem.*, 1963, **75**, 206; E. Carberry and R. West, *J. Amer. Chem. Soc.*, 1969, **91**, 5440, 5446.

<sup>3</sup> H. Gilman and R. A. Tomasi, *J. Org. Chem.*, 1963, **28**, 1651; M. Kumada, M. Ishikawa, S. Sakamoto, and S. Maeda, *J. Organometallic Chem.*, 1969, **17**, 223.

<sup>4</sup> H. Watanabe, K. Higuchi, M. Kobayashi, T. Kitahara, and Y. Nagai, *J.C.S. Chem. Comm.*, 1977, 704.

<sup>5</sup> H. Matsumoto, T. Motegi, M. Hasagawa, and Y. Nagai, *J. Organometallic Chem.*, 1977, **142**, 149.