Synthesis of Hexakistrimethylsiloxybenzene

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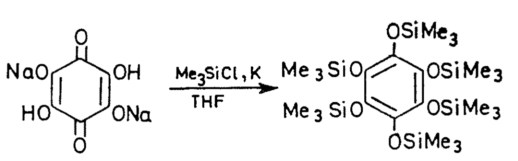
Summary Hexakistrimethylsiloxybenzene has been synthesized by the reaction of disodium salt of tetrahydroxybenzoquinone with potassium and trimethylsilyl chloride.

WE report the very simple synthesis and some spectroscopic properties of the hitherto unknown compound, (I), which has an electron-rich benzene ring surrounded by six bulky substituents.

To a stirred suspension of the disodium salt of tetrahydroxybenzoquinone[1] (II) (0·01 mole) and potassium (0·04 g atom) in tetrahydrofuran was added trimethylsilyl chloride (0·08 mole) over a period of 2 h at 66°. The usual work-up afforded a solid which on recrystallization from hexane gave (I) in a yield of 35%, m.p. 220°.



(II) (I)

Microanalysis and mass spectroscopy confirmed the molecular formula $C_{24}H_{54}O_6Si_6$. The strongest peak above the base peak at *m/e* 73 (100%) was the molecular-ion peak, (*M*), at *m/e* 606 (76%) accompanied with $M + 1$ (42%) and $M+2$ (29%). The observed $M+1$ to M ratio (58·7%) agreed with the calculated value (57·6%). Interestingly, the u.v. spectrum of (I) showed a typical aromatic band at 271 nm (ϵ 488) and was similar to polyalkylbenzenes (270 nm, ϵ 218, for hexamethylbenzene and 273 nm, ϵ 188, for hexaisopropylbenzene[2]) rather than to those of polyalkoxybenzenes (314 nm, ϵ 44, and 276 nm, ϵ 550 for hexamethoxybenzene[3]). Similarly, its i.r. spectrum differs remarkably from spectra of other phenoxy(trialkyl)silanes. The latter usually have a very strong absorption in the range 910—925 cm^{-1} attributed to Ph–O–Si absorption.[4] However, (I) showed no absorption in this range but showed the band of the strongest intensity at 1041 cm^{-1}. The n.m.r. spectrum exhibited only one sharp singlet at τ 9·86 (in CCl_4). This absorption remained unchanged in various solvents (pentane, tetrahydrofuran, toluene) at −80°, and in ether–pentane at −100°. Inspection of a model of (I) revealed the highly restricted rotation of the substituents, so non-equivalent methyl groups might exist. However, the result of the low-temperature n.m.r. study suggests (but does not demand) that, at least, free rotation about the bonds in trimethylsilyl groups and around silicon–oxygen bonds of (I) is allowed.

By the same procedure *p*-benzoquinone gave *p*-bistrimethylsiloxybenzene.

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[1] Commercially available.

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