

with any other phase. The observations do not lend support to the submicroscopic twinning description of the structural layers proposed by Daane *et al.* Investigations are currently under way employing X-ray diffraction and electron diffraction techniques, to find out whether the h.c.p. structure is present only in thin films or whether it exists in bulk specimens as well and also to determine the factors which influence the stabilization of this new h.c.p. structure.

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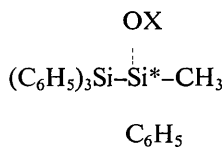
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**Crystal data for (—)-1,2,2,2-tetraphenyl-1-methyl-disilanol.** By ULRICH DE LA CAMP and HÅKON HOPE, Department of Chemistry, University of California, Davis, California, 95616, U.S.A.

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(—)-1,2,2,2-Tetraphenyl-1-methyl-disilanol,  $C_{25}Si_2OH_{24}$ , crystallizes in the monoclinic space group  $P2_1$  with unit-cell dimensions  $a = 20.03$ ,  $b = 11.40$ ,  $c = 20.29$  Å and  $\beta = 96.24^\circ$ . There are eight molecules in the unit cell.

(—)-1,2,2,2-Tetraphenyl-1-methyl-disilanol (Ia) was prepared by allowing diastereomeric phenylmethyl(—)-menthoxychlorosilane to couple with triphenylsilyllithium, yielding ( $\pm$ )-1,2,2,2-tetraphenyl-1-methyl-1-(—)-menthoxydisilane (Ib). The details of the synthesis, the resolution of



I (a) X=H; (b) X=(—)-menthyl,  $C_{10}H_{19}$

the mixture of diastereomers, and the conversion to the disilanol have been described by Sommer & Rosborough (1967). Tentative relative configurations were assigned to a series of disilane derivatives; however, serious discrepancies between the chemical and ORD\* evidence make these assignments doubtful. No absolute configurations have as yet been given for any of these compounds. Preliminary crystallographic work on (Ia) was therefore started.

\* Optical rotatory dispersion.

The space group was determined from oscillation, Weissenberg and precession photographs. Symmetry and systematic absences ( $0k0$  absent for  $k = 2n + 1$ ) indicate the space group to be  $P2_1$ . Cell constants were determined from  $\chi$ ,  $\varphi$  and  $2\theta$  values for axis reflections measured on a Picker diffractometer. The radiation used was  $\text{Cu } K\alpha$  ( $\lambda = 1.542$  Å). The crystals are prisms elongated along **b**.

The crystal data are as follows:  $a = 20.03 \pm 0.02$ ,  $b = 11.40 \pm 0.02$ ,  $c = 20.29 \pm 0.02$  Å;  $\beta = 96.24 \pm 0.05^\circ$ ;  $V = 4606$  Å<sup>3</sup>;  $Z = 8$ ;  $d_{\text{obs}} = 1.14$ ,  $d_{\text{calc}} = 1.15$  g.cm<sup>-3</sup>.

Owing to the large number of molecules in the unit cell, (leading to 112 non-hydrogen atoms in the asymmetric unit) a detailed structure analysis would appear to be rather costly. We have no immediate plans for further work on this compound.

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