the ones reported earlier (K & S), a reasonable reduction in the standard deviations of the variables is gained and also a smaller R_I. The use of the retrieved information content of the powder pattern has led to a further improvement in the form of the anisotropic thermal parameters obtained.

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Crystallization effects on the space group of (+)-α-naphthylphenylmethoxy-(−)-menthoxysilane, $C_{27}H_{34}O_2Si$.

By J. P. VIDAL, *Centre d'Etude de la Dynamique des Etats Ordonnés, USTL, Place Eugène Bataillon*, 34060-*Montpellier Cedex, France*

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Studies made by Vidal, Galigné & Falgueirettes [*C. R. Acad. Sci. Paris* (1970). **270**, 690–691] and Kanters & van Veen [*Cryst. Struct. Commun.* (1973). **2**, 261–267] on crystals given by Lanneau (Thèse de spécialité, Poitiers, 1969) have shown that there are two different space groups depending on the crystallization conditions.

Introduction

(+)-α-Naphthylphenylmethoxy-(−)-menthoxysilane is isolated by fractional crystallization from two diastereomers (+ − and − −). The former diastereomer (+ −), I_a melts at 103 °C and has a specific rotation of −58·8°. The latter (− −), I_b has a specific rotation of −76·4° and a melting point of 79 °C. Under certain crystallization conditions a compound, I, with well defined physical properties can be obtained; its melting temperature is 74 °C, and its specific rotation −68·6°.

These compounds have been synthesized by Lanneau (1969). Chemical properties and a number of reactions have been described by Corriu, Lanneau & Royo (1972).

Results

Vidal, Galigné & Falgueirettes (1970) made a crystallographic study of the compounds I_a, I_b and I and showed that the compound I is a quasi-racemate of the diastereomers I_a and I_b. Use of Weissenberg and Buerger precession cameras enabled us to obtain approximate values for the parameters and to determine the space group.

The parameter values were refined by least-squares calculations, using the Bragg angles measured with precision on a semi-automatic Enraf–Nonius CAD-3 diffractometer. The radiation used was Cu $K\alpha$.

Crystal I_a: $a=18{\cdot}402$ (6), $b=16{\cdot}260$ (7), $c=8{\cdot}362$ (5) Å, $V_{cell}=2500$ Å^3, space group $P2_12_12_1$, $Z=4$, $D_{calc}=1{\cdot}11$, $D_{obs}=1{\cdot}11$ g cm^{-3}.

Crystal I_b: $a=19{\cdot}260$ (7), $b=17{\cdot}636$ (5), $c=7{\cdot}177$ (5) Å, $V_{cell}=2483$ Å^3, space group $P2_12_12_1$, $Z=4$, $D_{calc}=1{\cdot}13$, $D_{obs}=1{\cdot}11$ g cm^{-3}.

Crystal I: $a=18{\cdot}566$ (6), $b=16{\cdot}000$ (7), $c=16{\cdot}553$ (4) Å, $V_{cell}=4917$ Å^3, space group $P2_12_12_1$, $Z=8$, $D_{calc}=1{\cdot}13$, $D_{obs}=1{\cdot}11$ g cm^{-3}.

Recently, Kanters *et al.* (1973) determined the absolute configuration of the compound I_a and obtained the following data.

Crystal I_a: (single-crystal Enraf–Nonius CAD-3 diffractometer with Cu $K\alpha$ radiation), $a=9{\cdot}774$, $b=9{\cdot}520$, $c=8{\cdot}447$ Å, $\alpha=90{\cdot}1$, $\beta=114{\cdot}4$, $\gamma=117{\cdot}5°$, $V_{cell}=617$ Å^3, space group $P1$, $Z=1$, $D_{calc}=1{\cdot}09$, $D_{obs}=1{\cdot}11$ g cm^{-3}.

Discussion

In both the studies, samples of I_a were provided by Lanneau. For our study (Vidal *et al.*, 1970), it was very difficult to obtain good crystals, as these compounds are inclined to give either syrupy solutions or solid agglomerates. Different neutral solvents and different crystallization conditions were tried. The best results were obtained for the compound I_a with anhydrous heptane.

In the other study (Kanters *et al.*, 1973), the crystals of I_a were recrystallized from ethanol.

Lanneau had remarked that if the compound I_a was subjected to a quick crystallization (for example by using a warming plate gently), there was emission of very perceptible sparks with very audible sputterings. I think that if the crystallization conditions are poor space group $P1$ is obtained, but if the crystallization proceeds normally the higher-symmetry space group $P2_12_12_1$ is obtained.

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