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Crystallographic report

Fluoro-bis-*iso*-propyl-(2,4,6-tris-*iso*-propylphenyl)-silane, $i\text{-}\mathrm{Pr}_2(2,4,6\text{-}i\text{-}\mathrm{Pr}_3\mathrm{C}_6\mathrm{H}_2)\mathrm{SiF}$

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Owing to steric congestion in $i\text{-}\mathrm{Pr}_2(2,4,6\text{-}i\text{-}\mathrm{Pr}_3\mathrm{C}_6\mathrm{H}_2)\mathrm{SiF}$, the geometry at the Si atom deviates slightly from ideal tetrahedral geometry with an increased C–Si–C angle of 119.02(9)° and elongated Si–C and Si–F bond distances. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; organosilicon; fluorosilane

COMMENT

The title compound (**I**) is an unusually sterically crowded fluorosilane for which the reactivity towards organo-substituted lithium phosphanides is greatly reduced according to earlier work.[1] In a closer investigation of isolated ^{29}Si(^{19}F) NMR spin systems it has been considered as a model compound.[2] The SiF unit connects the three bulky organic substituents. In the crystal structure of **I** the fluorine atom is almost coplanar to the aryl ring, leaving the two silicon-bonded *i*-propyl groups almost perpendicular to each side of the ring plane (Fig. 1). Owing to steric repulsion, the C–Si–C angle between these *i*-propyl groups is significantly increased to 119.02(9)°. The bond distances from silicon to the adjacent carbon atoms are almost equivalent, with the Si–C$_{\mathrm{aryl}}$ bond showing the largest value. Normally, an Si–Csp^2 distance should be significantly shorter than that found for an Si–Csp^3 bond and so this result underlines the steric pressure in the silyl unit. The Si–F bond length of 1.6131(11) Å is slightly elongated compared with similar fluorosilanes having sterically demanding substituents.[3] Usually, hyperconjugative interaction with the aryl π-system is claimed to explain the long Si–F bond in related molecules, which, owing to the perpendicular orientation of the Si–F bond relative to the aryl π-system, is not possible in **I**.[4] The syn-periplanar arrangement of the fluorine atom with C2 and the adjacent *i*-propyl group of the phenyl ring does not cause any significant alterations of the ring geometry.

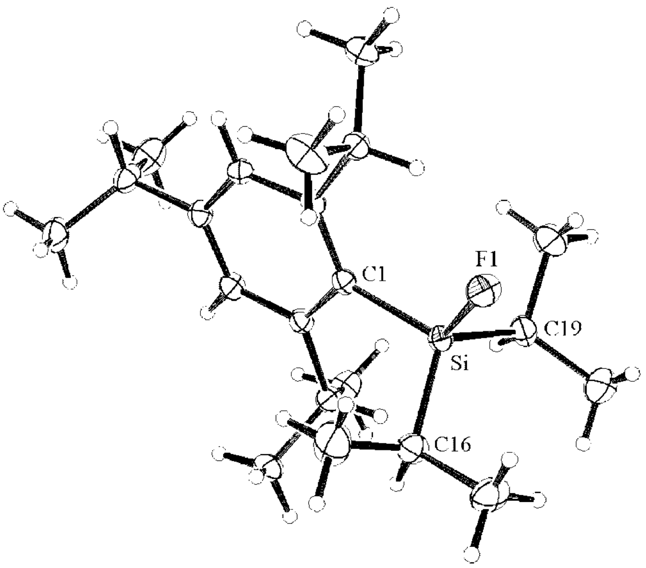

Figure 1. Molecular structure of **I**. Key geometric parameters: Si–F1 1.6131(11), Si–C1 1.8965(17), Si–C16 1.8820(20), Si–C19 1.8852(19), C1–C2 1.419(2), C1–C6 1.422(2), C2–C3 1.398(2), C3–C4 1.385(2), C4–C5 1.382(3), C5–C6 1.395(2) Å; F1–Si–C1 111.69(7), F1–Si–C16 101.45(8), F1–Si–C19 104.36(8), C1–Si–C16 111.35(8), C1–Si1–C19 108.52(8), C16–Si1–C19 119.02(9)°.

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EXPERIMENTAL

Isityllithium (1.05 g, 5 mmol; prepared according to a literature procedure)[5] was reacted with with bis-isopropyldifluorosilane (0.76 g, 5 mmol; prepared according to a literature procedure)[6] in diethyl ether as solvent at −78 °C. After stirring for 2 h the mixture was warmed to room temperature overnight. The solvent was removed in a vacuum and the residue extracted with pentane (2 × 25 ml). Removal of the solvent yields **I** as a colourless deliquescent solid (1.31 g, 78%). ^{19}F NMR ($CDCl_3$, δ ppm): −184.7. ^{29}Si NMR ($CDCl_3$, δ ppm): 18.2 (d, $^1J(^{29}Si-^{19}F) = 296$ Hz). Intensity data were collected at 203 K on a Bruker AXS CCD 1000 diffractometer for a colourless needle 0.1 × 0.1 × 0.2 mm^3. $C_{21}H_{37}FSi$, $M = 336.60$, triclinic, $P1$, $a = 8.6960(16)$, $b = 9.5153(18)$ and $c = 14.021(3)$ Å, $\alpha = 100.781(4)$, $\beta = 105.348(4)$, $\gamma = 99.077(4)°$, $V = 1072.4(3)$ Å^3, $Z = 2$, $R = 0.049$ (3096 data with $I > 2\sigma(I)$), $wR = 0.147$ (all 3726 data; $\theta_{max} = 25.1°$). Programs used: SHELXS-97, SHELXL-97 and ORTEP. CCDC deposition number: 250 703.

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