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Crystallographic report

tert-Butyldifluoro(2,4,6-tris-*iso*-propylphenyl)silane, t-Bu(2,4,6-*i*-PrC$_6$H$_2$)SiF$_2$

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Owing to steric congestion within the title compound, the geometry at the silicon atom deviates slightly from ideal tetrahedral geometry with an increased C–Si–C angle of 115.97(12)° and an elongated Si–C$_{aryl}$ bond distance. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; organosilicon; fluorosilane

COMMENT

The title compound, t-Bu(2,4,6-*i*-PrC$_6$H$_2$)SiF$_2$ (**1**, fig.1), is a sterically crowded fluorosilane and has recently been used as a model compound for a detailed investigation of an isolated ^{29}Si(^{19}F)$_2$ NMR spin system.[1] Owing to steric repulsion, the C–Si–C angle is increased to 115.97(12)°. The bond distances from Si to C1 (1.874(3) Å) and C11 (1.870(3) Å) are equivalent. Normally, an Si–C(sp^2) distance should be significantly shorter than that found for an Si–C(sp^3) bond, and so this result underlines the steric pressure in the silyl unit. The bond lengths from silicon to F1 (1.5945(16) Å) and F2 (1.5953(18) Å) are slightly elongated, which is in accordance with a conjugative interaction of Si–F σ^* states with the aryl

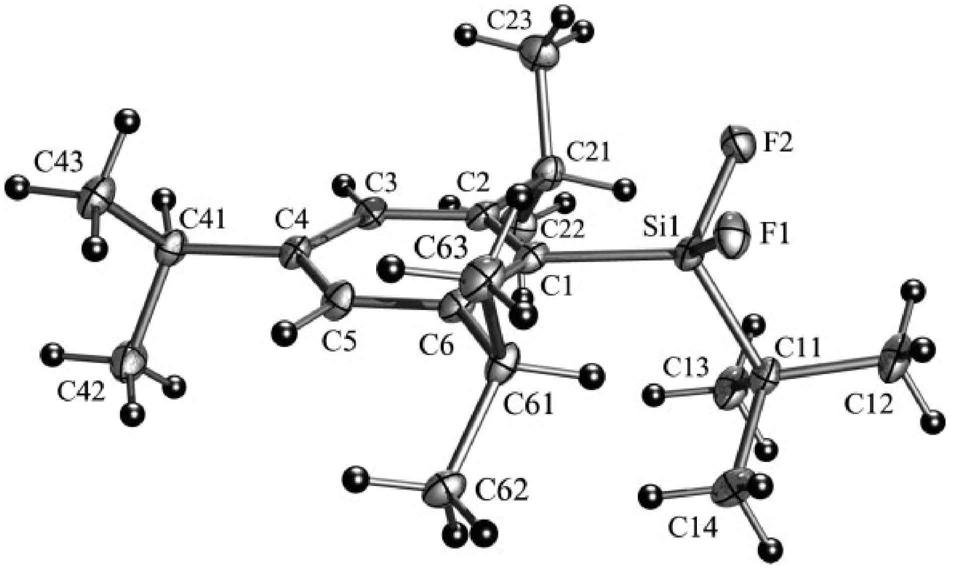

Figure 1. Molecular structure of **1**. Key geometric parameters: C1–C2 1.420(3), C1–C6 1.423(4), C2–C3 1.391(4), C3–C4 1.390(4), C4–C5 1.394(4), C5–C6 1.394(4) Å; C1–Si1–C11 115.97(12), F1–Si1–F2 101.31(9), C1–Si1–F1 111.20(10), C1–Si1–F2 110.82 (11), C11–Si1–F1 107.90(11), C11–Si1–F2 108.58(11)°.

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π-system, as one would expect.[2] This argument is consistent with the C–C bond distances within the phenyl ring.

EXPERIMENTAL

A solution of t-Bu(2,4,6-*i*-PrC$_6$H$_2$)SiCl$_2$ (3.0 g, 8.4 mmol; prepared according to a literature procedure[3]) in toluene (50 ml) was added to a suspension of SbF$_3$ (3.0 g, 16.7 mmol) in toluene (50 ml) at room temperature and stirring was continued to allow complete conversion, as monitored by ^{19}F NMR. The solvent was removed under vacuum and the residue was extracted with pentane (2 × 100 ml). Removal of the solvent and recrystallization from acetone yields **1** as colorless crystals (1.8 g, 66%); m.p. 73 °C (lit. 72–74 °C[3,4]). ^{19}F NMR (CDCl$_3$, δ ppm): −136.0. ^{29}Si NMR (CDCl$_3$, δ ppm): −9.3 (t, $^1J(^{29}\text{Si}-^{19}\text{F}) = 307$ Hz). Other spectroscopic and analytical data are identical to those reported in the literature.[3] Intensity data were collected at 95 K on a Stoe diffractometer for a colorless plate 0.12 × 0.25 × 0.35 mm^3. C$_{19}$H$_{32}$F$_2$Si, $M = 326.54$, triclinic, $P\bar{1}$, $a = 5.941(2)$, $b = 9.743(3)$, $c = 17.328(4)$ Å, $\alpha = 75.76(2)$, $\beta = 80.35(2)$, $\gamma = 80.40(2)°$, $V = 950.1(5)$ Å^3, $Z = 2$, 3713 unique data ($\theta_{\max} = 26.0°$), 2984 data with $I > 2\sigma(I)$, $R = 0.066$ (obs. data), $wR_2 = 0.189$ (all data). Programs used: SHELXS-97, SHELXL-97, ORTEP and Povray. CCDC deposition number: 226798.

REFERENCES

1. Helluy X, Pietschnig R, Sebald A. *Solid State Nucl. Magn. Reson.* 2003; **24**: 286.
2. Reed AE, Schleyer PvR. *J. Am. Chem. Soc.* 1990; **112**: 1434.
3. Nakadaira Y, Oharu K, Sakurai H. *J. Organometal. Chem.* 1986; **309**: 247.
4. Smit CN, Bickelhaupt F. *Organometallics* 1987; **6**: 1156.