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Bulky 2,6-disubstituted aryl siloxanes and a disilanamine

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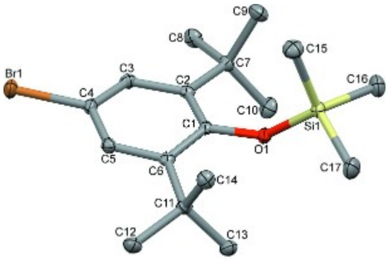
The crystal structures of 5-bromo-1,3-di-*tert*-butyl-2-[(trimethylsilyl)oxy]benzene, $C_{17}H_{29}BrOSi$, (I), 1,3-di-*tert*-butyl-2-[(trimethylsilyl)oxy]benzene, $C_{17}H_{30}OSi$, (II), and *N*-(2,6-diisopropylphenyl)-1,1,1-trimethyl-*N*-(trimethylsilyl)silanamine, $C_{18}H_{35}NSi_2$, (III), are reported. Compound (I) crystallizes in space group $P2_1/c$ with $Z' = 1$, (II) in *Pnma* with $Z' = 0.5$ and (III) in *Cmcm* with $Z' = 0.25$. Consequently, the molecules of (II) are constrained by *m* and those of (III) by *m2m* site symmetries. Despite this, both (I) and (II) are distorted towards mild boat conformations, as is typical of 2,6-di-*tert*-butyl-substituted phenyl compounds, reflecting the high local steric pressure of the flanking alkyl groups. Compound (III) by contrast is planar and symmetric, and this lack of distortion is compatible with the lower steric pressure of the flanking 2,6-diisopropyl substituents.

1. Chemical context

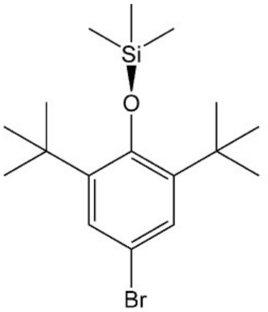
Aryl siloxanes and silanamines are important reaction intermediates, especially as protecting groups for phenols and anilines (Lucente-Schultz *et al.*, 2009). Thus, 5-bromo-1,3-di-*tert*-butyl-2-[(trimethylsilyl)oxy]benzene, (I), is used as a synthetic intermediate to form *inter alia p*-conjugated arylboron radicals (Chung *et al.*, 2018), *p*-quinone methides (Wang *et al.*, 2018) and spin-labelled polymers (Otaki & Goto, 2019). Recently a new cross-coupling reaction using the parent phenol was shown to be more effective than using protected 1,3-di-*tert*-butyl-2-[(trimethylsilyl)oxy]benzene, (II) (Nieves-Quinones *et al.*, 2019). Silanamines such as *N*-(2,6-diisopropylphenyl)-1,1,1-trimethyl-*N*-(trimethylsilyl)silanamine, (III), can support chemistry at the 4-position of the ring, including robust heteroelement derivatives (Maaninen *et al.*, 1999), and are also good sources for the controlled synthesis of early transition-metal amides (Siemeling *et al.*, 1999; Pennington *et al.*, 2005). Similarly, substitution of (II) at the 4-position of the ring leads to numerous intrinsic heteroatom derivatives in addition to follow-up reactivity at oxygen (Kindra *et al.*, 2013; Poverenov *et al.*, 2007; Satoh & Shi, 1994; Healy & Barron, 1990). Herein we report the single-crystal X-ray diffraction structures of (I), (II) and (III).



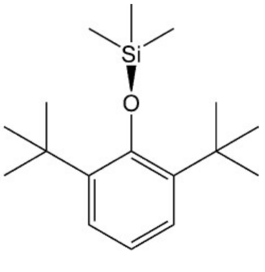
2. Structural commentary

Compound (I) crystallizes on a general position in $P2_1/c$ and adopts a distortion towards boat-shaped (Fig. 1*a*) in which all of the atoms along the central ridge of the substituted benzene ring tilt above a best plane defined by the central C2/C3/C5/C6 ring carbon atoms, whilst the ^{t}Bu groups tilt below. Interestingly, (II) crystallizes with a similar degree of distortion towards a boat conformation (Fig. 1*b*): deviations from the central planes in (I) and (II) are 1.401 (6) and 1.446 (5) Å for Si1, 0.226 (4) and 0.227 (3) Å for O1, 0.111 (3) and 0.107 (2) Å for C1, 0.039 (3) and 0.040 (3) Å for C4 and an average of −0.117 (4) and −0.112 (2) Å for the two ^{t}Bu central carbon atoms; note, however, that (II) has bilateral symmetry from the occupation of Wyckoff site 4*c* in *Pnma* with Si1, O1, C1, C4 and C15 on the mirror. The Si1—O1 bond lengths in (I) and (II) are closely comparable at 1.6617 (15) and 1.6655 (12) Å, respectively, as are the C1—O1 lengths at 1.379 (2) and 1.3821 (19) Å. Noticeably, all these dimensions are long, corresponding to the upper quartiles of the compiled values (1.652 and 1.373 Å, respectively; Lide, 2004) for all organic Si—O bond lengths. In both molecules, the Me_3SiO groups are strongly tilted out of the molecular planes and the C1—O1—Si1 angles are similar but not identical at 139.75 (13) and 137.9 (1)°. Consideration of space-filling representations strongly suggest that these angles allow the best fitting of the bulky Me_3Si groups between flanking ^{t}Bu groups, with very specific orientations of the H atoms on all the components.

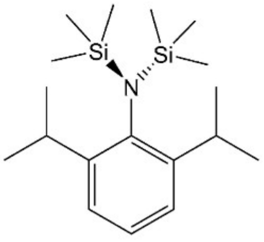
In contrast to the two siloxanes, the silanamine (III) is rigorously planar with the $N(SiMe_3)_2$ moiety strictly orthogonal to the aryl ring (Fig. 1*c*) as required by *m*2*m* symmetry at Wyckoff site 4*c* in space group *Cmcm*. Consideration of a space-filling model also confirms the tight fit of the two Me_3Si groups between the flanking isopropyl moieties, and the constraints on orientations of the Me groups of all the substituents are also considerable, inducing a constrained internal orientation in (III). The N1—Si1 bond lengths are 1.7529 (13) Å, approaching the upper quartile of the compiled standard values of 1.755 Å for all aromatic N—Si bond lengths (Lide, 2004). The C1—N1—Si1 angles are 116.92 (7)°, considerably smaller than the C—O—Si angles in (I) and (II), consistent with trigonal substitution at N1.



(I)



(II)



(III)

The close interlocking of the methyl group atoms belonging to the ^{t}Bu/^{i}Pr and Me_3Si substituents in all three molecules is very evident in Fig. 1.

3. Supramolecular features

Compound (I) is gently packed (Fig. 2) in its extended structure with few contacts shorter than $\Sigma r_{\rm vdW}$. By contrast, (II) forms stacks along the *a*-axis direction (Fig. 3) with some contacts from $SiMe_3$ H atoms to aromatic rings at 2.80 Å, within ($\Sigma r_{\rm vdW}$ − 0.1 Å), indicative of weak dispersion interactions; this is consistent with the high crystallinity encountered when (II) is a synthetic by-product. The structure of (III) has very high symmetry as a consequence of space group

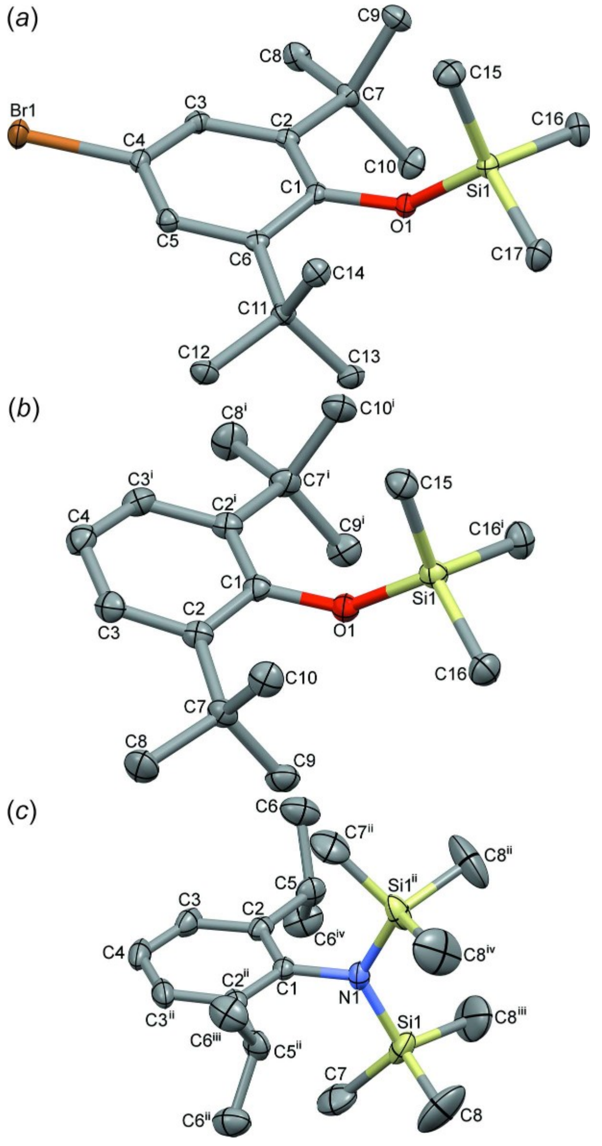


Figure 1
Displacement ellipsoids plot of the molecular structures of (*a*) (I) at the 50% probability level; (*b*) (II), also at the 50% probability level, and (*c*) (III) at the 40% probability level. H atoms have been omitted and the atom numbering schemes are shown. [Symmetry codes: (i): $x, \frac{1}{2} - y, z$; (ii) $1 - x, y, \frac{3}{2} - z$; (iii) $x, y, \frac{3}{2} - z$; (iv) $1 - x, y, z$.]

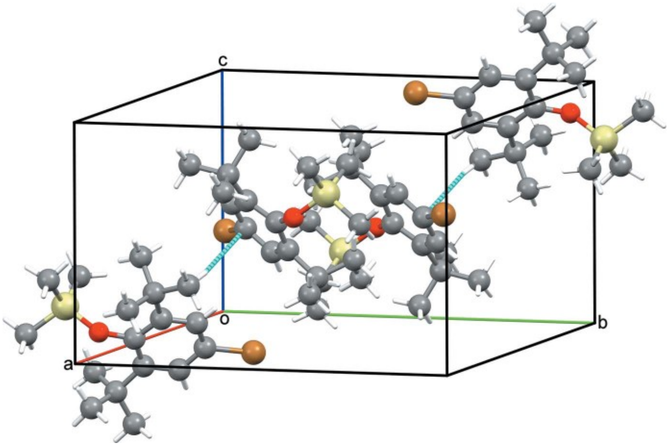


Figure 2
Unit-cell packing diagram for (I) viewed bisecting γ with H atoms shown with arbitrary radii and intermolecular contacts less than ΣrvdW as dashed blue lines.

Cmcm and $Z' = 0.25$ but there are no contacts shorter than $\Sigma r_{\rm vdW}$. The resultant weak packing (Fig. 4) may be a contributing factor to the rather large displacement ellipsoids occurring in the anisotropic refinement of (III).

4. Database survey

The geometry of (I) may be compared to that of 4-bromo-2,6-di-*tert*butylphenol, for which a modern low-temperature area-detector structure has been reported in the Cambridge Structure Database (CSD, Version 5.40, with updates to February 2019; Groom *et al.*, 2016) with refcode BBPHOL02 (Marszaukowski & Boeré, 2019). The C—Br distance of 1.904 (2) Å in (I) is indistinguishable from 1.905 (3) Å in the latter at the 99% confidence level. Both (I) and (II) can be compared with five other reported structures in the CSD that share the same combination of 2,6-di-*tert*-butylphenyl rings and 1-trimethylsiloxane substituents, with CSD refcodes: GIFCEE (Poverenov *et al.*, 2007), JEHDOP (Healy & Barron, 1990), LIKYEJ, which has three independent such moieties attached to a B_3O_3 ring (Satoh & Shi, 1994), TIXZUK, in which two such groups are attached to bismuth atoms that are dimerized through a short $M\cdots M$ contact (Kindra *et al.*, 2013) and TIYBEK (Kindra *et al.*, 2013). All the interatomic distances and angles in (I) and in (II) are indistinguishable from the mean values for the eight independent comparators at the 99% confidence level (nine when BBPHOL02 is included for the non-trimethylsilyl dimensions). This allows for the computation of global mean values (Table 1). Thus, the Si—O distances of 1.6617 (15) and 1.6655 (12) Å in (I) and (II) fit within an average of 1.657 (10) Å for this set of di-^{t}Bu-flanked trimethylsiloxanes, and close to the upper quartile value of 1.652 Å for all organic Si—O bond lengths (Lide, 2004). A comparison of symmetry-averaged interatomic distances (Å) and angles (°) for (I), (II) and (III) with the discussed comparator sets is presented in Table 1.

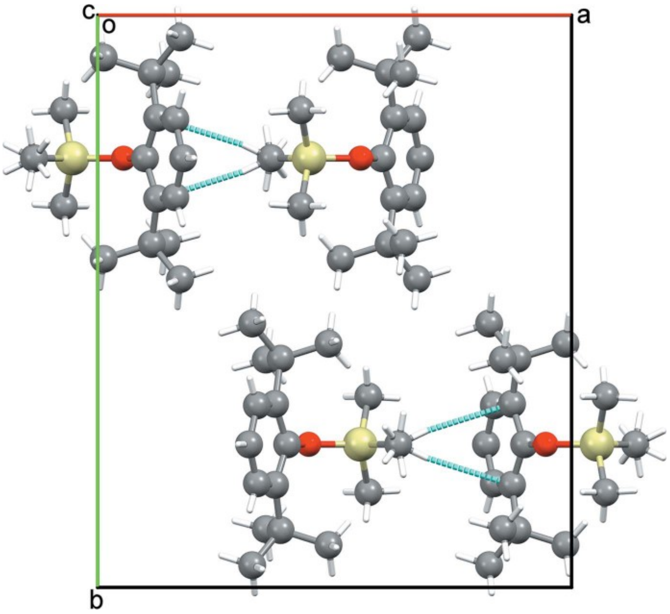


Figure 3
Unit-cell packing diagram for (II) viewed perpendicular to *c* with H atoms shown with arbitrary radii and intermolecular contacts less than ΣrvdW as dashed blue lines.

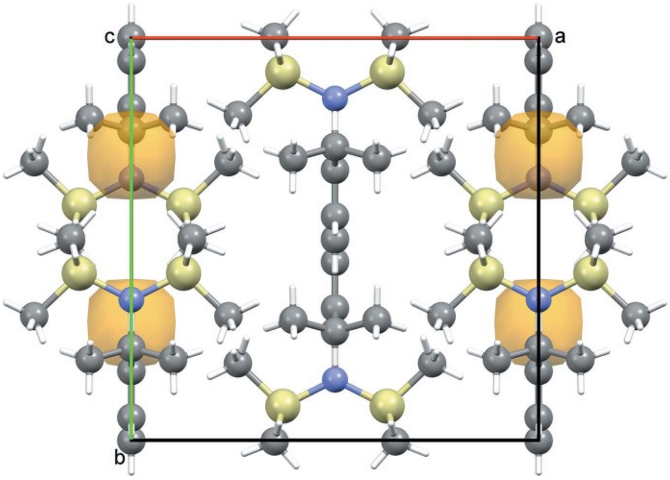


Figure 4
Unit-cell packing diagram for (III) viewed perpendicular to *c* with H atoms shown with arbitrary radii. Small, non solvent-accessible, voids of 22 Å^3 are shaded ochre.

One exception to taking meaningful averages concerns the C1—O1—Si1 angles, which though similar in (I) and (II) at 139.75 (13) and 137.91 (10)°, are both intermediate with respect to an overall range from a low of 126.8 (1) in GIFCEE to a high of 150.3 (2)° in one of the TIKZUK components. Evidently, this angle has a wide variability and a low specificity, so it was of interest to investigate if the values are independent of other structural parameters. For example, attempted correlation of these angles with the C1—O1 bond length shows an almost random scatter. However, *all* members of this series show mild distortions of the substituted benzene rings towards a *boat* conformation in which S11, O1, C1 and C4 deviate in the same direction from planar and the ^{t}Bu group C7 and C11 atoms deviate in the opposite direction. A

Table 1
Average interatomic distances and angles (Å, °) in (I), (II) and (III) with comparators.

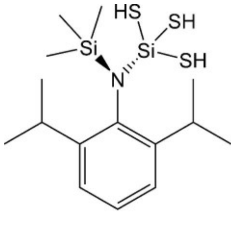
Atom numbers taken from (I).

Parameter	(I)	(II)	Mean siloxane[a]	(III)	Mean silanamine[b]
Si1—O1,N1	1.6617 (15)		1.657 (10)	1.7529 (13)	1.762 (18)
Ave Si—C	1.865 (2)	1.8666 (15)	1.861 (8)	1.861 (2)	1.859 (6)
C1—O1,N1	1.379 (2)	1.3823 (19)	1.385 (7)	1.448 (4)	1.453 (8)
Av C1—C2,6	1.419 (3)	1.4183 (14)	1.415 (5)	1.405 (3)	1.403 (4)
Av C2,5—C3,6	1.395 (3)	1.3977 (17)	1.395 (7)	1.398 (3)	1.389 (3)
Av C2,6—C7,11	1.542 (3)	1.5459 (16)	1.546 (4)	1.513 (3)	1.519 (7)
Av C3,5—C4	1.379 (3)	1.3798 (15)	1.385 (6)	1.373 (3)	1.378 (11)
Av C7,11-methyl	1.540 (3)	1.5386 (17)	1.537 (7)	1.530 (2)	1.523 (6)
Av C—Si—C	109.88 (9)	109.21 (6)	110.0 (8)	110.76 (11)	110.7 (5)
Av O,*N*—Si—C	109.01 (11)	109.68 (7)	108.9 (18)	107.79 (13)	107.84 (9)
C1—O1,N1—Si1	139.75 (13)	137.90 (10)	140 (5)	116.92 (7)	117.0 (10)
Av C2,6—C1—O,*N*	119.42 (17)	119.31 (7)	119.2 (10)	119.81 (13)	119.8 (2)
C2—C1—C6	120.97 (18)	121.23 (15)	121.4 (5)	120.4 (3)	120.5 (4)
Av C1—C2,6—C3,5	117.75 (18)	117.12 (11)	117.9 (6)	118.5 (2)	118.7 (4)
Av C1—C2,6—C7,11	123.92 (17)	124.64 (11)	124.3 (10)	123.05 (19)	123.2 (4)
Av C3,5—C2,6—C7,C11	118.34 (17)	118.24 (10)	112.5 (13)	118.5 (2)	118.6 (11)
Av C2,6—C3,5—C4	120.59 (18)	122.25 (12)	122.5 (11)	121.6 (2)	121.1 (10)
C3—C4—C5	121.40 (18)	119.17 (16)	118.5 (16)	119.5 (3)	119.9 (16)
Av C2,6—C7,11—Me	110.79 (16)	110.86 (10)	110.7 (11)	111.78 (14)	111.83 (15)
Av Me—C7,11—Me	108.12 (16)	108.04 (10)	108.2 (14)	109.5 (2)	109.55 (7)

Notes: (*a*) Mean values taken over (I), (II), BBPHOL02, GIFCEE, JEHDOP, LIKYEJ, TIXZUK and TIYBEK, treating crystallographically independent entities separately. (*b*) Mean values taken over (III), CAQWUW, CORKAV and QOCSEI.

strong correlation is found between the deviation of Si1 from the mean planes defined by C2, C3, C5 and C6 (and hence with the C1—O1—Si1 angle) and similar deviations of smaller magnitude for O1, C1 and C4 (correlation coefficients of 0.98, 0.93 and 0.83, respectively). Thus, bends at the siloxane oxygen atoms smoothly pucker the whole rings toward boat conformations. A consideration of the fits between the ^{t}Bu and Me$_3$Si groups also indicates that the former undergo rotation so as to accommodate the various tilt angles of the latter from the mean molecular planes – a double turnstile motion that accommodates variations in their relative positions despite the interlocking interactions within these structures.

A close structural analogue to (III) has been reported for an aminosilanetrithiol analogue (IV), which has one of the SiMe$_3$ groups replaced by Si(SH)$_3$ (CSD refcode QOCSEI; Li *et al.*, 2014). This is almost isostructural and crystallizes in space group *Cmc*2$_1$ with a unit cell that is imperceptibly different at the 99% confidence level (0.6% shorter in *a* but 0.6% longer in *c*, leading to a volume just 0.1% lower). It has a mirror disorder of the SiMe$_3$ and Si(SH)$_3$ groups as a consequence of being positioned with the aryl ring on a lattice mirror plane. Molecules of (IV) share the same relative lattice positions as those of (III) in *Cmcm*.



(IV)

The reduced site symmetry [compared to *m*2*m* in (III)] results in considerable asymmetry in the benzene ring in QOCSEI and a small deviation from full orthogonality of the N-Si*R*$_3$ units w.r.t. the benzene ring (dihedral angle of 88.1°). By contrast, orthogonal arrangements of the aryl and CNSi$_2$ planes are found in the (ordered) structures of two ring-substituted derivatives of (III) with refcodes CORKAV (4-SeCl$_3$) and QOCSEI (4-ferrocenylethynyl), neither of which have site-symmetry restraints (Maaninen *et al.* 1999; Siemeling *et al.*, 1999). This suggests that it is the interlocking steric constraints of the 2,6-diisopropyl and N(SiMe$_3$)$_2$ groups that induces these highly regular structures, and greater planarity of the aromatic rings and substituents compared to the typical distortions observed for 2,6-di-*tert*-butyl phenol derivatives such as (I) and (II). Notably, there is only one reported crystal structure of a 2,6-di-*tert*-butylaniline with two silyl substituents, in the form of a four-membered N$_2$(Si^{i}Pr$_2$)$_2$ ring (refcode: FOTWEQ; Stalke *et al.*, 1987) and this is severely distorted from planarity towards a boat conformation with the N atoms 0.60 and 0.69 Å out of the planes of the four central ring carbon atoms.

Within the comparison set of these three previously reported structures, the Si1—N1 distance of 1.7529 (13) Å in (III) compares well with the mean value of 1.750 (2) Å for CAQWUW and CORKAV, whereas the value in QOCSEI of 1.788 (8) Å is different at the 99% confidence level and may have been elongated by the disorder refinement, in agreement with the author's report that the DFT-computed value for this bond is noticeably shorter at 1.744 Å (Li *et al.*, 2014). There is also considerable variation amongst the four structures for the individual Si—C lengths in different positions (*e.g.* Si1—C7 *versus* Si1—C8) but the *average* of Si—C distances of 1.861 (2) Å, and N—Si—C and C—Si—C angles of 110.76 (11)

Table 2
Experimental details.

	(I)	(II)	(III)
Crystal data			
Chemical formula	$C_{17}H_{29}BrOSi$	$C_{17}H_{30}OSi$	$C_{18}H_{35}NSi_2$
M_r	357.40	278.50	321.65
Crystal system, space group	Monoclinic, $P2_1/c$	Orthorhombic, *Pnma*	Orthorhombic, *Cmcm*
Temperature (K)	100	100	173
a, b, c (Å)	13.0955 (1), 15.3457 (2), 9.0449 (1)	14.47237 (14), 17.4657 (2), 6.73933 (7)	12.199 (3), 12.091 (3), 14.177 (3)
α, β, γ (°)	90, 94.617 (1), 90	90, 90, 90	90, 90, 90
V (Å^3)	1811.76 (3)	1703.50 (3)	2091.1 (8)
Z	4	4	4
Radiation type	Cu $K\alpha$	Cu $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	3.67	1.13	0.17
Crystal size (mm)	0.42 × 0.21 × 0.13	0.31 × 0.11 × 0.07	0.19 × 0.16 × 0.10
Data collection			
Diffractometer	Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, Pilatus 200K	Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, Pilatus 200K	Bruker APEXII CCD
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018)	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018)	ψ scan (*SADABS*; Bruker, 2014)
$T_{\min}$, $T_{\max}$	0.507, 1.000	0.773, 1.000	0.667, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	20327, 3935, 3924	17855, 1793, 1663	8996, 1317, 1082
$R_{\rm int}$	0.024	0.046	0.039
$(\sin \theta/\lambda)_{\max}$ (Å^{-1})	0.639	0.626	0.652
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.031, 0.073, 1.10	0.033, 0.093, 1.06	0.047, 0.126, 1.04
No. of reflections	3935	1793	1317
No. of parameters	191	99	64
No. of restraints	0	0	54
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å^{-3})	0.66, −0.37	0.26, −0.28	0.36, −0.26

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

and 107.79 (13)°, respectively in (III) are not significantly different at the 99% confidence level from the corresponding mean values for the comparison set of 1.859 (6) Å, 110.7 (5) and 107.84 (9)°, respectively. All other interatomic distances and angles found for (III) are similarly indistinguishable from the comparison set at the 99% confidence level.

5. Synthesis and crystallization

2,6-Di-*tert*-butyl-phenol (Acros), 2,6-di-*tert*-butyl-4-bromophenol and 2,6-diisopropylanilene (Aldrich) were commercial products and used as received except where noted. The technical grade anilene was purified by vacuum distillation. Solvents (BDH) were chromatographic grade and dried before use by standard methods. NMR spectra were recorded on a 300 MHz Bruker Avance II spectrometer and are referenced to tetramethylsilane at 0 (^{1}H) and $CDCl_3$ at 77.23 (^{13}C) ppm.

5.1. Preparation of (I)

Compound (I) was prepared by modification of a literature method (Lucente-Schultz *et al.*, 2009). A 250 ml side-arm RBF was charged with 2.85 g (10 mmol) of 2,6-di-*tert*-butyl-4-bromophenol in 50 ml of dry THF. The solution was cooled to 195 K for 10 min with stirring. Then 6.0 ml (15 mmol) ^{n}BuLi (2.5 *M* in hexanes) was slowly added and the resulting mixture was stirred for 1 h. Next, chlorotrimethilsylane (2.17 g, 20 mmol) was added to the mixture and the reaction was stirred for 1 h while warming to RT. The product was poured into water (50 ml) and extracted with hexanes twice (2 × 20 ml). The organic layer was washed with water (30 ml), dried with anhydrous $MgSO_4$ and filtered. The product was isolated as a colorless crystalline solid on evaporation and found to be synthetically pure. Yield 3.21 g (90%). ^{1}H NMR (300.13 MHz, $CDCl_3$): δ 0.41 (SiCH_3, *s*, 9H); 1.38 [C(CH_3)$_3$, *s*, 18H]; 7.32 (C*H*, *s*, 2H). ^{13}C NMR (75.48 MHz, $CDCl_3$): δ 3.90 (SiCH_3); 31.04 [C(CH_3)$_3$]; 35.31 [–*C*(CH_3)$_3$]; 113.82 (C_4); 128.69 ($C_{3,5}$); 143.12 ($C_{2,6}$); 152.44 (C_1). Crystals were grown from hexanes.

5.2. Preparation of (II)

Compound (II) was prepared in an analogous manner to (I) from 2.06 g (10 mmol) of 2,6-di-*tert*-butylphenol. Other reagent quantities match those used for (I). The colorless crystalline product solidified on evaporation and was found to be synthetically pure (1.81 g, 65%). ^{1}H NMR (300.13 MHz, $CDCl_3$): δ 0.41 (SiCH_3, *s*, 9H); 1.41 [C(CH_3)$_3$, *s*, 18H]; 6.86 (C*H*, *t*, 1H, $J_{\rm H–H}$ = 7.9 Hz); 7.25 (C*H*, *d*, 2H, $J_{\rm H–H}$ = 7.8 Hz). ^{13}C NMR (75.48 MHz, $CDCl_3$): δ 4.03 (SiCH_3); 31.33 (C(CH_3)$_3$);

35.18 [–$C(CH_3)_3$]; 120.69 (C_3); 125.80 (C_4); 140.87 (C_2); 153.20 (C_1). NMR data was compared to the literature values (Goyal & Singh, 1996). Crystals were grown from hexanes.

5.3. Preparation of (III)

Compound (III) was prepared as reported in the literature (Maaninen *et al.*, 1999). Crystals were grown by sublimation. ^{1}H NMR agrees with the literature.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms in the three structures are attached to C atoms and are treated as riding, with C—H = 0.98 Å and U_{iso} = 1.5U_{eq}(C) for methyl, with C—H = 0.97 Å and U_{iso} = 1.3U_{eq}(C) for methine and with C—H = 0.95 Å and U_{iso} = 1.2U_{eq}(C) for aromatic. H atoms attached to methyl carbon atoms C15 in the structure of (II) and C7 in the structure of (III) are duplicated by the mirror symmetries and have been refined with half-occupancy.

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supporting information

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Bulky 2,6-disubstituted aryl siloxanes and a disilanamine

Flavia Marszaukowski, Karen Wohnrath and René T. Boeré

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2018) for (I), (II); *APEX2* (Bruker, 2014) for (III). Cell refinement: *CrysAlis PRO* (Rigaku OD, 2018) for (I), (II); *SAINT* (Bruker, 2014) for (III). Data reduction: *CrysAlis PRO* (Rigaku OD, 2018) for (I), (II); *SAINT* (Bruker, 2014) for (III). For all structures, program(s) used to solve structure: ShelXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

5-Bromo-1,3-di-*tert*-butyl-2-[(trimethylsilyl)oxy]benzene (I)

Crystal data

$C_{17}H_{29}BrOSi$	$F(000) = 752$
$M_r = 357.40$	$D_x = 1.310$ Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
$a = 13.0955$ (1) Å	Cell parameters from 18499 reflections
$b = 15.3457$ (2) Å	$\theta = 4.4$–$79.8°$
$c = 9.0449$ (1) Å	$\mu = 3.67$ mm^{-1}
$\beta = 94.617$ (1)°	$T = 100$ K
$V = 1811.76$ (3) Å^3	Prism, clear colourless
$Z = 4$	0.42 × 0.21 × 0.13 mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, Pilatus 200K diffractometer	$T_{\min} = 0.507$, $T_{\max} = 1.000$
	20327 measured reflections
	3935 independent reflections
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source	3924 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.024$
Mirror monochromator	$\theta_{\max} = 80.1°$, $\theta_{\min} = 3.4°$
ω scans	$h = -16 \rightarrow 16$
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)	$k = -19 \rightarrow 18$
	$l = -8 \rightarrow 11$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.031$	H-atom parameters constrained
$wR(F^2) = 0.073$	$w = 1/[\sigma^2(F_o^2) + (0.0196P)^2 + 3.634P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	
3935 reflections	$(\Delta/\sigma)_{\max} = 0.002$
191 parameters	$\Delta\rho_{\max} = 0.66$ e Å^{-3}
0 restraints	$\Delta\rho_{\min} = -0.36$ e Å^{-3}
Primary atom site location: dual	

Extinction correction: SHELXL-2018/3 (Sheldrick 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00139 (10)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.51566 (2)	0.21330 (2)	0.45554 (3)	0.01991 (9)
Si1	0.81135 (4)	0.60159 (4)	0.69256 (6)	0.01462 (13)
O1	0.80844 (10)	0.51233 (9)	0.58830 (15)	0.0137 (3)
C1	0.74581 (14)	0.44182 (13)	0.5533 (2)	0.0122 (4)
C2	0.67726 (14)	0.44492 (13)	0.4237 (2)	0.0124 (4)
C3	0.60978 (15)	0.37556 (13)	0.3970 (2)	0.0139 (4)
H3	0.562847	0.376592	0.314197	0.017*
C4	0.61193 (15)	0.30530 (13)	0.4923 (2)	0.0143 (4)
C5	0.68503 (15)	0.29845 (13)	0.6102 (2)	0.0136 (4)
H5	0.686809	0.249093	0.670039	0.016*
C6	0.75644 (15)	0.36495 (13)	0.6406 (2)	0.0128 (4)
C7	0.67316 (15)	0.52100 (13)	0.3119 (2)	0.0141 (4)
C8	0.60959 (17)	0.49792 (14)	0.1665 (2)	0.0183 (4)
H8A	0.636634	0.446032	0.125038	0.027*
H8B	0.612894	0.545090	0.097353	0.027*
H8C	0.539601	0.488246	0.186543	0.027*
C9	0.61968 (17)	0.59939 (14)	0.3782 (2)	0.0196 (4)
H9A	0.548598	0.586074	0.385149	0.029*
H9B	0.625605	0.649347	0.315599	0.029*
H9C	0.651459	0.611686	0.475345	0.029*
C10	0.78100 (17)	0.54395 (15)	0.2675 (2)	0.0200 (4)
H10A	0.823327	0.561482	0.353977	0.030*
H10B	0.776290	0.590827	0.196991	0.030*
H10C	0.810620	0.493866	0.223875	0.030*
C11	0.84163 (15)	0.35033 (13)	0.7659 (2)	0.0140 (4)
C12	0.85418 (16)	0.25267 (14)	0.8049 (2)	0.0184 (4)
H12A	0.793262	0.231898	0.845688	0.028*
H12B	0.911767	0.245176	0.876394	0.028*
H12C	0.865318	0.220270	0.716834	0.028*
C13	0.94702 (15)	0.37909 (14)	0.7187 (2)	0.0183 (4)
H13A	0.963697	0.345661	0.634250	0.027*
H13B	0.998222	0.369667	0.799205	0.027*
H13C	0.944785	0.439842	0.693388	0.027*
C14	0.81303 (17)	0.39676 (14)	0.9074 (2)	0.0181 (4)
H14A	0.800068	0.457243	0.886197	0.027*

H14B	0.868505	0.391592	0.982939	0.027*
H14C	0.752635	0.370455	0.941273	0.027*
C15	0.69125 (17)	0.61128 (15)	0.7890 (2)	0.0223 (5)
H15A	0.645076	0.650692	0.735070	0.033*
H15B	0.707232	0.633097	0.887583	0.033*
H15C	0.659611	0.555032	0.793856	0.033*
C16	0.83126 (18)	0.69902 (14)	0.5749 (3)	0.0238 (5)
H16A	0.888992	0.689285	0.518019	0.036*
H16B	0.843886	0.749167	0.637185	0.036*
H16C	0.771138	0.708748	0.509058	0.036*
C17	0.92756 (18)	0.59976 (15)	0.8263 (3)	0.0247 (5)
H17A	0.921155	0.553917	0.897176	0.037*
H17B	0.934120	0.654670	0.877009	0.037*
H17C	0.987189	0.589737	0.773474	0.037*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.01809 (13)	0.01465 (12)	0.02615 (14)	−0.00553 (8)	−0.00349 (8)	0.00120 (8)
Si1	0.0148 (3)	0.0125 (3)	0.0165 (3)	−0.00183 (19)	0.0007 (2)	−0.0017 (2)
O1	0.0135 (6)	0.0125 (6)	0.0153 (6)	−0.0022 (5)	0.0009 (5)	−0.0006 (5)
C1	0.0107 (8)	0.0117 (9)	0.0145 (9)	−0.0004 (7)	0.0029 (7)	−0.0023 (7)
C2	0.0120 (9)	0.0134 (9)	0.0121 (9)	0.0027 (7)	0.0024 (7)	−0.0002 (7)
C3	0.0128 (9)	0.0154 (9)	0.0132 (9)	0.0006 (7)	−0.0002 (7)	−0.0003 (7)
C4	0.0118 (9)	0.0130 (9)	0.0183 (10)	−0.0021 (7)	0.0021 (7)	−0.0029 (7)
C5	0.0140 (9)	0.0127 (9)	0.0142 (9)	0.0002 (7)	0.0018 (7)	0.0017 (7)
C6	0.0124 (9)	0.0146 (9)	0.0119 (9)	0.0015 (7)	0.0037 (7)	−0.0013 (7)
C7	0.0158 (9)	0.0129 (9)	0.0136 (9)	0.0005 (7)	0.0012 (7)	0.0015 (7)
C8	0.0228 (10)	0.0177 (10)	0.0142 (9)	0.0003 (8)	0.0000 (8)	0.0022 (8)
C9	0.0240 (11)	0.0149 (10)	0.0198 (10)	0.0054 (8)	−0.0002 (8)	−0.0007 (8)
C10	0.0210 (10)	0.0230 (11)	0.0166 (10)	−0.0039 (8)	0.0045 (8)	0.0029 (8)
C11	0.0139 (9)	0.0147 (9)	0.0131 (9)	0.0006 (7)	−0.0004 (7)	0.0006 (7)
C12	0.0175 (10)	0.0176 (10)	0.0197 (10)	0.0027 (8)	−0.0015 (8)	0.0037 (8)
C13	0.0132 (9)	0.0198 (10)	0.0215 (10)	0.0009 (8)	−0.0005 (8)	0.0020 (8)
C14	0.0204 (10)	0.0194 (10)	0.0142 (9)	0.0001 (8)	−0.0006 (8)	−0.0004 (8)
C15	0.0215 (11)	0.0246 (11)	0.0214 (11)	0.0014 (9)	0.0049 (8)	−0.0021 (9)
C16	0.0261 (11)	0.0157 (10)	0.0294 (12)	−0.0049 (9)	0.0021 (9)	0.0007 (9)
C17	0.0230 (11)	0.0209 (11)	0.0291 (12)	−0.0048 (9)	−0.0042 (9)	−0.0035 (9)

Geometric parameters (Å, º)

Br1—C4	1.904 (2)	C10—H10A	0.9600
Si1—O1	1.6617 (15)	C10—H10B	0.9600
Si1—C15	1.865 (2)	C10—H10C	0.9600
Si1—C16	1.865 (2)	C11—C12	1.545 (3)
Si1—C17	1.866 (2)	C11—C13	1.542 (3)
O1—C1	1.379 (2)	C11—C14	1.537 (3)
C1—C2	1.419 (3)	C12—H12A	0.9600

C1—C6	1.420 (3)	C12—H12B	0.9600
C2—C3	1.392 (3)	C12—H12C	0.9600
C2—C7	1.543 (3)	C13—H13A	0.9600
C3—H3	0.9300	C13—H13B	0.9600
C3—C4	1.379 (3)	C13—H13C	0.9600
C4—C5	1.378 (3)	C14—H14A	0.9600
C5—H5	0.9300	C14—H14B	0.9600
C5—C6	1.397 (3)	C14—H14C	0.9600
C6—C11	1.541 (3)	C15—H15A	0.9600
C7—C8	1.540 (3)	C15—H15B	0.9600
C7—C9	1.538 (3)	C15—H15C	0.9600
C7—C10	1.540 (3)	C16—H16A	0.9600
C8—H8A	0.9600	C16—H16B	0.9600
C8—H8B	0.9600	C16—H16C	0.9600
C8—H8C	0.9600	C17—H17A	0.9600
C9—H9A	0.9600	C17—H17B	0.9600
C9—H9B	0.9600	C17—H17C	0.9600
C9—H9C	0.9600		

O1—Si1—C15	110.45 (9)	H10A—C10—H10B	109.5
O1—Si1—C16	109.57 (9)	H10A—C10—H10C	109.5
O1—Si1—C17	109.61 (9)	H10B—C10—H10C	109.5
C15—Si1—C16	111.39 (11)	C6—C11—C12	111.50 (16)
C15—Si1—C17	111.88 (11)	C6—C11—C13	111.48 (16)
C16—Si1—C17	103.75 (11)	C13—C11—C12	105.05 (16)
C1—O1—Si1	139.75 (13)	C14—C11—C6	109.51 (16)
O1—C1—C2	119.25 (17)	C14—C11—C12	106.75 (16)
O1—C1—C6	119.59 (17)	C14—C11—C13	112.41 (17)
C2—C1—C6	120.97 (18)	C11—C12—H12A	109.5
C1—C2—C7	123.59 (17)	C11—C12—H12B	109.5
C3—C2—C1	117.91 (18)	C11—C12—H12C	109.5
C3—C2—C7	118.50 (17)	H12A—C12—H12B	109.5
C2—C3—H3	119.7	H12A—C12—H12C	109.5
C4—C3—C2	120.58 (18)	H12B—C12—H12C	109.5
C4—C3—H3	119.7	C11—C13—H13A	109.5
C3—C4—Br1	119.37 (15)	C11—C13—H13B	109.5
C5—C4—Br1	119.17 (15)	C11—C13—H13C	109.5
C5—C4—C3	121.40 (18)	H13A—C13—H13B	109.5
C4—C5—H5	119.7	H13A—C13—H13C	109.5
C4—C5—C6	120.60 (18)	H13B—C13—H13C	109.5
C6—C5—H5	119.7	C11—C14—H14A	109.5
C1—C6—C11	124.24 (17)	C11—C14—H14B	109.5
C5—C6—C1	117.59 (18)	C11—C14—H14C	109.5
C5—C6—C11	118.17 (17)	H14A—C14—H14B	109.5
C8—C7—C2	111.93 (16)	H14A—C14—H14C	109.5
C8—C7—C10	105.93 (16)	H14B—C14—H14C	109.5
C9—C7—C2	109.21 (16)	Si1—C15—H15A	109.5
C9—C7—C8	106.33 (16)	Si1—C15—H15B	109.5

C9—C7—C10	112.26 (17)	Si1—C15—H15C	109.5
C10—C7—C2	111.09 (16)	H15A—C15—H15B	109.5
C7—C8—H8A	109.5	H15A—C15—H15C	109.5
C7—C8—H8B	109.5	H15B—C15—H15C	109.5
C7—C8—H8C	109.5	Si1—C16—H16A	109.5
H8A—C8—H8B	109.5	Si1—C16—H16B	109.5
H8A—C8—H8C	109.5	Si1—C16—H16C	109.5
H8B—C8—H8C	109.5	H16A—C16—H16B	109.5
C7—C9—H9A	109.5	H16A—C16—H16C	109.5
C7—C9—H9B	109.5	H16B—C16—H16C	109.5
C7—C9—H9C	109.5	Si1—C17—H17A	109.5
H9A—C9—H9B	109.5	Si1—C17—H17B	109.5
H9A—C9—H9C	109.5	Si1—C17—H17C	109.5
H9B—C9—H9C	109.5	H17A—C17—H17B	109.5
C7—C10—H10A	109.5	H17A—C17—H17C	109.5
C7—C10—H10B	109.5	H17B—C17—H17C	109.5
C7—C10—H10C	109.5		

Br1—C4—C5—C6	−179.69 (15)	C2—C3—C4—C5	−4.7 (3)
Si1—O1—C1—C2	95.3 (2)	C3—C2—C7—C8	−13.1 (2)
Si1—O1—C1—C6	−89.5 (2)	C3—C2—C7—C9	104.4 (2)
O1—C1—C2—C3	−175.38 (17)	C3—C2—C7—C10	−131.27 (19)
O1—C1—C2—C7	4.2 (3)	C3—C4—C5—C6	3.1 (3)
O1—C1—C6—C5	173.89 (17)	C4—C5—C6—C1	4.7 (3)
O1—C1—C6—C11	−6.3 (3)	C4—C5—C6—C11	−175.10 (17)
C1—C2—C3—C4	−1.6 (3)	C5—C6—C11—C12	18.1 (2)
C1—C2—C7—C8	167.30 (18)	C5—C6—C11—C13	135.20 (19)
C1—C2—C7—C9	−75.2 (2)	C5—C6—C11—C14	−99.8 (2)
C1—C2—C7—C10	49.1 (2)	C6—C1—C2—C3	9.5 (3)
C1—C6—C11—C12	−161.65 (18)	C6—C1—C2—C7	−170.85 (17)
C1—C6—C11—C13	−44.6 (3)	C7—C2—C3—C4	178.75 (18)
C1—C6—C11—C14	80.4 (2)	C15—Si1—O1—C1	−2.0 (2)
C2—C1—C6—C5	−11.0 (3)	C16—Si1—O1—C1	−125.1 (2)
C2—C1—C6—C11	168.74 (17)	C17—Si1—O1—C1	121.7 (2)
C2—C3—C4—Br1	178.09 (15)		

1,3-Di-*tert*-butyl-2-[(trimethylsilyl)oxy]benzene (II)

Crystal data

$C_{17}H_{30}OSi$
$M_r = 278.50$
Orthorhombic, *Pnma*
$a = 14.47237\ (14)$ Å
$b = 17.4657\ (2)$ Å
$c = 6.73933\ (7)$ Å
$V = 1703.50\ (3)$ Å^3
$Z = 4$
$F(000) = 616$

$D_x = 1.086$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 12570 reflections
$\theta = 5.1–74.4°$
$\mu = 1.13$ mm^{-1}
$T = 100$ K
Needle, clear colourless
0.31 × 0.11 × 0.07 mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, Pilatus 200K diffractometer	$T_{\min} = 0.773$, $T_{\max} = 1.000$
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source	17855 measured reflections
Mirror monochromator	1793 independent reflections
ω scans	1663 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)	$R_{\text{int}} = 0.046$
	$\theta_{\max} = 74.7°$, $\theta_{\min} = 5.1°$
	$h = -18 \rightarrow 17$
	$k = -21 \rightarrow 20$
	$l = -8 \rightarrow 8$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.033$	$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.6892P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.093$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.06$	$\Delta\rho_{\max} = 0.26$ e Å^{-3}
1793 reflections	$\Delta\rho_{\min} = -0.28$ e Å^{-3}
99 parameters	
0 restraints	
Primary atom site location: dual	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Si1	0.44620 (3)	0.250000	0.02273 (7)	0.01982 (15)	
O1	0.55464 (8)	0.250000	0.10549 (17)	0.0194 (3)	
C1	0.59998 (11)	0.250000	0.2860 (2)	0.0181 (3)	
C2	0.62564 (8)	0.32076 (6)	0.37337 (17)	0.0195 (3)	
C3	0.66393 (8)	0.31815 (7)	0.56362 (18)	0.0228 (3)	
H3	0.679079	0.364859	0.628093	0.027*	
C4	0.68049 (12)	0.250000	0.6610 (3)	0.0244 (4)	
H4	0.702993	0.250001	0.793454	0.029*	
C7	0.61394 (8)	0.39980 (7)	0.27322 (18)	0.0219 (3)	
C8	0.67393 (10)	0.46122 (7)	0.3744 (2)	0.0309 (3)	
H8A	0.652833	0.468650	0.511229	0.046*	
H8B	0.668568	0.509554	0.301445	0.046*	
H8C	0.738594	0.444559	0.374947	0.046*	
C9	0.64519 (9)	0.39815 (7)	0.05482 (18)	0.0249 (3)	
H9A	0.710577	0.383808	0.047981	0.037*	
H9B	0.636715	0.448972	−0.004068	0.037*	
H9C	0.608163	0.360625	−0.018420	0.037*	
C10	0.51286 (9)	0.42590 (7)	0.29218 (19)	0.0268 (3)	
H10A	0.472354	0.388099	0.229055	0.040*	
H10B	0.505156	0.475597	0.226734	0.040*	

H10C	0.496631	0.430630	0.432822	0.040*	
C15	0.36580 (12)	0.250000	0.2393 (3)	0.0259 (4)	
H15A	0.306965	0.226595	0.200528	0.039*	0.5
H15B	0.354989	0.302788	0.282753	0.039*	0.5
H15C	0.393242	0.220617	0.348249	0.039*	0.5
C16	0.42870 (9)	0.16623 (8)	−0.1452 (2)	0.0282 (3)	
H16A	0.475983	0.166658	−0.249330	0.042*	
H16B	0.367304	0.169330	−0.206060	0.042*	
H16C	0.433624	0.118748	−0.068507	0.042*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0205 (2)	0.0176 (3)	0.0213 (3)	0.000	−0.00179 (16)	0.000
O1	0.0212 (6)	0.0171 (6)	0.0198 (6)	0.000	−0.0020 (4)	0.000
C1	0.0178 (7)	0.0194 (8)	0.0170 (8)	0.000	0.0020 (6)	0.000
C2	0.0194 (5)	0.0178 (6)	0.0214 (6)	0.0005 (4)	0.0031 (4)	−0.0006 (5)
C3	0.0258 (6)	0.0213 (6)	0.0213 (6)	−0.0011 (5)	0.0027 (5)	−0.0037 (5)
C4	0.0277 (9)	0.0274 (9)	0.0182 (8)	0.000	0.0006 (7)	0.000
C7	0.0266 (6)	0.0154 (6)	0.0237 (6)	−0.0013 (5)	−0.0008 (5)	−0.0012 (5)
C8	0.0402 (8)	0.0188 (6)	0.0338 (7)	−0.0063 (5)	−0.0060 (6)	−0.0002 (5)
C9	0.0303 (6)	0.0189 (6)	0.0256 (6)	−0.0033 (5)	0.0024 (5)	0.0028 (5)
C10	0.0315 (7)	0.0197 (6)	0.0291 (7)	0.0050 (5)	0.0000 (5)	−0.0031 (5)
C15	0.0230 (8)	0.0266 (9)	0.0280 (9)	0.000	0.0007 (7)	0.000
C16	0.0297 (6)	0.0263 (7)	0.0286 (7)	0.0000 (5)	−0.0057 (5)	−0.0048 (5)

Geometric parameters (Å, º)

Si1—O1	1.6655 (12)	C8—H8A	0.9800
Si1—C15	1.8664 (19)	C8—H8B	0.9800
Si1—C16	1.8671 (13)	C8—H8C	0.9800
Si1—C16$^{\text{i}}$	1.8671 (13)	C9—H9A	0.9800
O1—C1	1.3821 (19)	C9—H9B	0.9800
C1—C2	1.4185 (14)	C9—H9C	0.9800
C1—C2$^{\text{i}}$	1.4185 (14)	C10—H10A	0.9800
C2—C3	1.3976 (17)	C10—H10B	0.9800
C2—C7	1.5461 (16)	C10—H10C	0.9800
C3—H3	0.9500	C15—H15A	0.9800
C3—C4	1.3802 (15)	C15—H15B	0.9800
C4—H4	0.9500	C15—H15C	0.9800
C7—C8	1.5393 (17)	C16—H16A	0.9800
C7—C9	1.5400 (16)	C16—H16B	0.9800
C7—C10	1.5376 (17)	C16—H16C	0.9800
O1—Si1—C15	109.00 (7)	H8A—C8—H8B	109.5
O1—Si1—C16$^{\text{i}}$	109.32 (5)	H8A—C8—H8C	109.5
O1—Si1—C16	109.32 (5)	H8B—C8—H8C	109.5
C15—Si1—C16$^{\text{i}}$	112.92 (5)	C7—C9—H9A	109.5

C15—Si1—C16	112.92 (5)	C7—C9—H9B	109.5
C16—Si1—C16^{i}	103.18 (9)	C7—C9—H9C	109.5
C1—O1—Si1	137.91 (10)	H9A—C9—H9B	109.5
O1—C1—C2	119.32 (7)	H9A—C9—H9C	109.5
O1—C1—C2^{i}	119.32 (7)	H9B—C9—H9C	109.5
C2—C1—C2^{i}	121.21 (15)	C7—C10—H10A	109.5
C1—C2—C7	124.62 (10)	C7—C10—H10B	109.5
C3—C2—C1	117.15 (11)	C7—C10—H10C	109.5
C3—C2—C7	118.24 (10)	H10A—C10—H10B	109.5
C2—C3—H3	118.9	H10A—C10—H10C	109.5
C4—C3—C2	122.23 (12)	H10B—C10—H10C	109.5
C4—C3—H3	118.9	Si1—C15—H15A	109.5
C3—C4—C3^{i}	119.16 (16)	Si1—C15—H15B	109.5
C3—C4—H4	120.4	Si1—C15—H15C	109.5
C3^{i}—C4—H4	120.4	H15A—C15—H15B	109.5
C8—C7—C2	111.54 (10)	H15A—C15—H15C	109.5
C8—C7—C9	105.71 (10)	H15B—C15—H15C	109.5
C9—C7—C2	111.61 (9)	Si1—C16—H16A	109.5
C10—C7—C2	109.43 (10)	Si1—C16—H16B	109.5
C10—C7—C8	107.06 (10)	Si1—C16—H16C	109.5
C10—C7—C9	111.37 (10)	H16A—C16—H16B	109.5
C7—C8—H8A	109.5	H16A—C16—H16C	109.5
C7—C8—H8B	109.5	H16B—C16—H16C	109.5
C7—C8—H8C	109.5		

Si1—O1—C1—C2	−92.19 (12)	C2^{i}—C1—C2—C7	170.19 (9)
Si1—O1—C1—C2^{i}	92.19 (12)	C2—C3—C4—C3^{i}	3.9 (2)
O1—C1—C2—C3	174.50 (12)	C3—C2—C7—C8	17.63 (15)
O1—C1—C2—C7	−5.3 (2)	C3—C2—C7—C9	135.63 (11)
C1—C2—C3—C4	2.89 (19)	C3—C2—C7—C10	−100.63 (12)
C1—C2—C7—C8	−162.53 (12)	C7—C2—C3—C4	−177.26 (12)
C1—C2—C7—C9	−44.53 (16)	C15—Si1—O1—C1	0.000 (1)
C1—C2—C7—C10	79.20 (15)	C16^{i}—Si1—O1—C1	123.87 (5)
C2^{i}—C1—C2—C3	−10.0 (2)	C16—Si1—O1—C1	−123.87 (5)

Symmetry code: (i) *x*, −*y*+1/2, *z*.

N-(2,6-Diisopropylphenyl)-1,1,1-trimethyl-*N*-(trimethylsilyl)silanamine (III)

Crystal data

$C_{18}H_{35}NSi_2$
$M_r = 321.65$
Orthorhombic, *Cmcm*
$a = 12.199$ (3) Å
$b = 12.091$ (3) Å
$c = 14.177$ (3) Å
$V = 2091.1$ (8) Å^3
$Z = 4$
$F(000) = 712$

$D_x = 1.022$ Mg m^{-3}
Mo *Kα* radiation, $\lambda = 0.71073$ Å
Cell parameters from 4098 reflections
$\theta = 2.4$–27.5°
$\mu = 0.17$ mm^{-1}
$T = 173$ K
Block, clear colourless
0.19 × 0.16 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer	1317 independent reflections
Graphite monochromator	1082 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.039$
Absorption correction: ψ scan (SADABS; Bruker, 2014)	$\theta_{\max} = 27.6°$, $\theta_{\min} = 2.4°$
$T_{\min} = 0.667$, $T_{\max} = 0.746$	$h = -15 \rightarrow 15$
8996 measured reflections	$k = -15 \rightarrow 15$
	$l = -18 \rightarrow 17$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.047$	$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 2.3874P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.126$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.04$	$\Delta\rho_{\max} = 0.36$ e Å^{-3}
1317 reflections	$\Delta\rho_{\min} = -0.26$ e Å^{-3}
64 parameters	
54 restraints	
Primary atom site location: dual	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Si1	0.62813 (7)	0.91524 (6)	0.750000	0.0459 (3)	
N1	0.500000	0.8496 (2)	0.750000	0.0315 (6)	
C1	0.500000	0.7298 (2)	0.750000	0.0262 (6)	
C2	0.500000	0.67207 (17)	0.66401 (16)	0.0308 (5)	
C3	0.500000	0.55648 (18)	0.6663 (2)	0.0402 (6)	
H3	0.500000	0.516529	0.608650	0.048*	
C4	0.500000	0.4993 (3)	0.750000	0.0446 (9)	
H4	0.500000	0.420746	0.750000	0.053*	
C5	0.500000	0.7292 (2)	0.56902 (17)	0.0396 (6)	
H5	0.500000	0.810796	0.580380	0.047*	
C6	0.39760 (16)	0.70083 (19)	0.51164 (15)	0.0572 (5)	
H6A	0.395926	0.621120	0.499170	0.086*	
H6B	0.399211	0.741161	0.451700	0.086*	
H6C	0.332066	0.722035	0.547291	0.086*	
C7	0.7385 (2)	0.8087 (3)	0.750000	0.0656 (9)	
H7A	0.806954	0.841786	0.727270	0.098*	0.5
H7B	0.717550	0.747465	0.708470	0.098*	0.5
H7C	0.749174	0.780828	0.814260	0.098*	0.5
C8	0.6484 (3)	1.0030 (3)	0.8562 (2)	0.1024 (11)	
H8A	0.595639	1.064092	0.855311	0.154*	
H8B	0.723089	1.032743	0.856153	0.154*	

H8C	0.637079	0.958308	0.913029	0.154*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0650 (5)	0.0344 (4)	0.0382 (4)	−0.0227 (3)	0.000	0.000
N1	0.0452 (14)	0.0211 (11)	0.0281 (13)	0.000	0.000	0.000
C1	0.0233 (13)	0.0231 (13)	0.0323 (15)	0.000	0.000	0.000
C2	0.0253 (9)	0.0293 (10)	0.0377 (12)	0.000	0.000	−0.0060 (9)
C3	0.0345 (11)	0.0283 (11)	0.0578 (16)	0.000	0.000	−0.0132 (10)
C4	0.0329 (16)	0.0222 (14)	0.079 (3)	0.000	0.000	0.000
C5	0.0492 (13)	0.0382 (12)	0.0313 (12)	0.000	0.000	−0.0087 (10)
C6	0.0526 (11)	0.0753 (14)	0.0437 (11)	0.0083 (10)	−0.0095 (9)	−0.0060 (10)
C7	0.0414 (15)	0.076 (2)	0.080 (2)	−0.0221 (15)	0.000	0.000
C8	0.117 (2)	0.0922 (19)	0.098 (2)	−0.0496 (19)	0.0103 (19)	−0.0548 (17)

Geometric parameters (Å, º)

Si1—N1	1.7529 (13)	C1—C2^{i}	1.405 (3)
Si1—C7	1.864 (4)	C2—C3	1.398 (3)
Si1—C8	1.858 (2)	C2—C5	1.513 (3)
Si1—C8^{i}	1.858 (2)	C3—C4	1.373 (3)
N1—C1	1.448 (4)	C5—C6	1.530 (2)
C1—C2	1.405 (3)	C5—C6^{ii}	1.530 (2)
N1—Si1—C7	109.36 (12)	C2—C1—N1	119.81 (13)
N1—Si1—C8	112.15 (10)	C2^{i}—C1—C2	120.4 (3)
N1—Si1—C8^{i}	112.15 (10)	C1—C2—C5	123.05 (19)
C8—Si1—C7	107.37 (13)	C3—C2—C1	118.5 (2)
C8^{i}—Si1—C7	107.37 (13)	C3—C2—C5	118.5 (2)
C8—Si1—C8^{i}	108.2 (2)	C4—C3—C2	121.6 (2)
Si1^{iii}—N1—Si1	126.17 (15)	C3^{i}—C4—C3	119.5 (3)
C1—N1—Si1^{iii}	116.92 (7)	C2—C5—C6^{ii}	111.78 (14)
C1—N1—Si1	116.92 (7)	C2—C5—C6	111.78 (14)
C2^{i}—C1—N1	119.81 (13)	C6—C5—C6^{ii}	109.5 (2)

Symmetry codes: (i) $x, y, -z+3/2$; (ii) $-x+1, y, z$; (iii) $-x+1, y, -z+3/2$.