$S = 2.10$
613 reflections
88 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.04F^2]$

Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($\mathring{A}^2$) for (II)*

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O3	0.3571 (6)	0.0922 (4)	1.0462 (6)	3.5 (1)
C1	0.8269 (8)	0.1561 (5)	1.0585 (9)	3.1 (1)
C2	0.6496 (9)	0.1830 (5)	1.1518 (8)	3.1 (1)
C3	0.4947 (8)	0.1438 (5)	1.0160 (8)	2.4 (1)
C4	0.5470 (8)	0.1832 (5)	0.8294 (8)	2.4 (1)
C5	0.4433 (9)	0.1307 (5)	0.6637 (9)	2.9 (1)
C6	0.3625 (9)	0.1888 (6)	0.5262 (8)	3.6 (1)
C14	0.7659 (8)	0.1846 (5)	0.8543 (8)	2.5 (1)
C15	0.959 (1)	1/4	1.115 (1)	4.1 (2)
C141	0.8692 (9)	0.1219 (6)	0.7168 (9)	4.0 (2)

Table 4. *Selected geometric parameters ($\mathring{A}$, °) for (II)*

O3—C3	1.204 (7)	C1—C2	1.533 (9)
C1—C14	1.546 (9)	C1—C15	1.505 (9)
C2—C2^i	1.610 (9)	C2—C3	1.491 (8)
C3—C4	1.519 (9)	C4—C4^i	1.606 (8)
C4—C5	1.495 (8)	C4—C14	1.564 (8)
C14—C14^i	1.573 (8)	C5—C6	1.309 (9)
C6—C6^i	1.472 (9)	C14—C141	1.504 (9)
C2—C1—C14	102.0 (5)	C2—C1—C15	104.3 (6)
C14—C1—C15	102.0 (6)	C1—C2—C3	103.8 (5)
C1—C2—C2^i	102.2 (5)	C3—C2—C2^i	108.5 (5)
O3—C3—C2	128.0 (6)	O3—C3—C4	127.0 (5)
C2—C3—C4	105.1 (5)	C3—C4—C4^i	108.2 (5)
C3—C4—C14	103.4 (4)	C14—C4—C4^i	89.4 (5)
C3—C4—C5	116.2 (5)	C14—C4—C5	121.0 (5)
C5—C4—C4^i	115.0 (5)	C1—C14—C4	107.3 (5)
C1—C14—C14^i	102.8 (5)	C1—C14—C141	114.6 (5)
C4—C14—C141	118.1 (5)	C4—C14—C14^i	90.6 (5)
C141—C14—C14^i	120.1 (5)	C1—C15—C1^i	97.3 (5)
C4—C5—C6	122.7 (6)	C5—C6—C6^i	122.3 (6)

Symmetry code: (i) $x, \frac{1}{2} - y, z$.

H atoms were located on a difference map. Their geometries were idealized and they were constrained to ride upon their attached non-H atoms, with $U(H) = 1.3U_{eq}$(attached atom). *CAD-4 Software* (Enraf–Nonius, 1989) was used for data collection and cell refinement. The programs used for the structure solution and refinement were *MULTAN*80 (Main *et al.*, 1980) and *MolEN* (Fair, 1990), respectively. The figures were drawn using *ORTEP*II (Johnson, 1976). *MolEN* was also used for data reduction.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1064). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two N-Aryl-Substituted Silyl Carbamates

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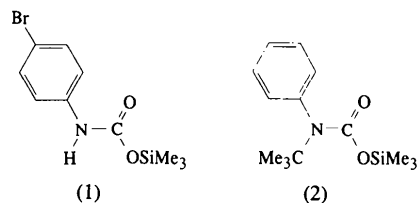
Abstract

Trimethylsilyl N-(4-bromophenyl)carbamate, $C_{10}H_{14}BrNO_2Si$, and trimethylsilyl N-*tert*-butyl-N-phenylcarbamate, $C_{14}H_{23}NO_2Si$, are both important silylating agents. The structures of both molecules are dominated by a plane containing a remarkably large number of atoms. The planes are organized by delocalization around the carbamate moiety. A quasi-pentacoordination of the Si atom in both compounds is also described.

Comment

Trimethylsilyl carbamates are efficient silylating agents (Knausz, Meszticzky, Szakács, Csákvári & Újszászy, 1983; Csató, Keresztes, Szabó & Knausz, 1985; Knausz, Kolos, Rohonczy & Újszászy, 1985). We have long been engaged in exploring the relationship between the structure and reactivity of this class of compounds (Böcskei, Knausz, Gál, Szakács & Csákvári, 1988; Szalay, Knausz, Szakács, Újszászy & Sohár, 1995a,b). As part of this project, we now present the crystal structures of two compounds belonging to this family of silylating agents, namely, trimethylsilyl N-(p-bromophenyl)carbamate, (1), and trimethylsilyl N-tert-butyl-N-phenylcarbamate, (2).

The structure of the molecule of compound (1) (Fig. 1) is dominated by a plane of seven non-H atoms organized around the carbamate moiety by delocalization which involves the C1 atom of the phenyl ring and the Si atom as well as the C10 atom of the SiMe₃ group, apart from the atoms of the carbamate skeleton. The p-bromophenyl moiety and the carbamate plane form a dihedral angle of around 25° indicating a partial delocalization between the two structural constituents. This is reinforced by the relatively short C1—N7 distance [1.419 (7) Å]. A full delocalization and indeed coplanarity of the carbamate and p-bromophenyl planes is probably hindered by the close contact of the H2 and O13 atoms [2.394 (8) Å]. A further decrease of the interplanar angle would involve even shorter H···O distances; molecular modelling shows that by setting the C2—C1—N7—C8 torsion angle to 0°, the H2···O13 distance decreases to a value of 2.159 Å. A further interesting structural feature to note is the quasi-pentacoordination of the Si atom by the four atoms bonded by classical covalent bonds as well as by the
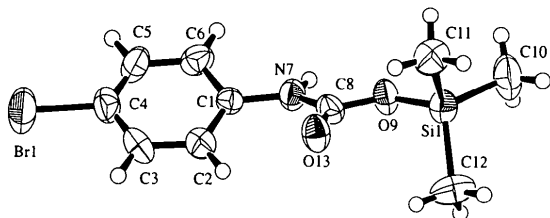
carbonyl O13 atom, the distance of which from the Si1 atom is 2.948 (5) Å. This feature is also present in the structure of compound (2) as well as in a number of other related compounds, the structures of which either have been published (Rohonczy et al., 1988) or are about to appear (Szalay et al., 1996). In the crystal lattice, the carbamate moieties are brought together by N—H···O-type hydrogen bonds into an endless chain.

Compound (2) has two conformationally similar molecules in the asymmetric unit (A and B) (Fig. 2). In fact, the only noteworthy differences between the two molecules are the C2—C1—N7—C8 and related torsion angles. This difference is probably enforced by packing preferences. The carbamate plane again includes the Si atom, although we have shown that in some related structures the delocalization between the carbamate and trimethylsilyl moieties is not strong enough to keep the Si atom in the carbamate plane (Szalay et al., 1996).

Molecule A

Molecule B

Fig. 2. The molecular structure and atomic numbering for compound (2). Displacement ellipsoids are plotted at the 50% probability level.
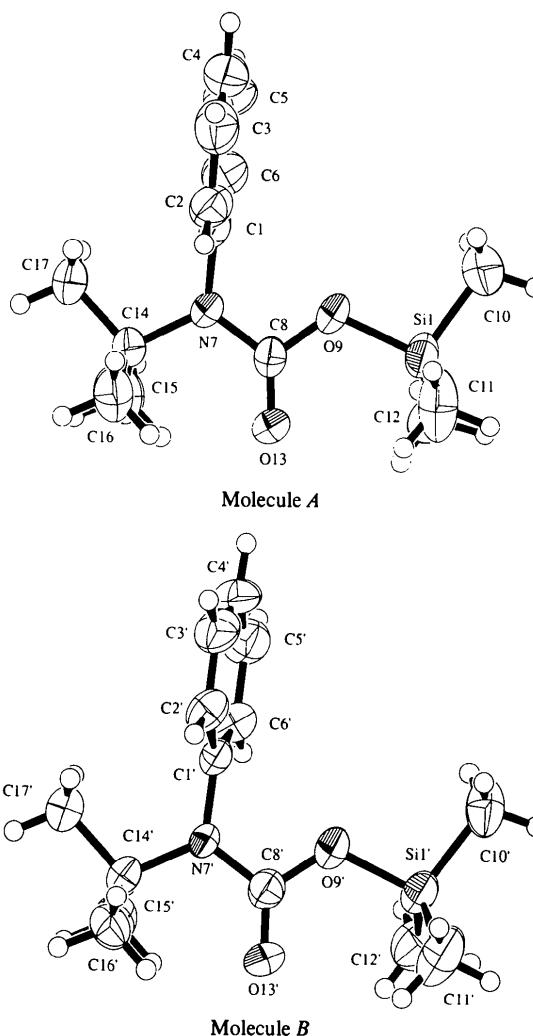

Fig. 1. The molecular structure and atomic numbering for compound (1). Displacement ellipsoids are plotted at the 50% probability level.

Owing to the presence of the *tert*-butyl group in compound (2), the phenyl group is unable to participate in a partial delocalization with the carbamate moiety, since coplanarity of the phenyl and carbamate groups would involve a very short C2—C17 distance.

The absence of delocalization is apparent if we compare the C1—N7 bond length in both compounds, which is significantly shorter in the case of compound (1) than in either of the two similar molecules of compound (2) (Tables 2 and 4). The C—O—Si bond angle, however, points in both cases to some delocalization between O and Si, with values of around 122°. In the absence of any strong secondary bond, the crystal packing of (2) is dominated by methyl–methyl hydrophobic interactions due to the presence of six methyl groups per molecule.

Experimental

Compound (1) was prepared according to the method of Knausz, Kolos, Rohonczy & Újszászy (1985), while compound (2) was prepared according to the method of Szalay, Knausz, Szakács, Újszászy & Sohár (1995a).

Compound (1)

Crystal data

$C_{10}H_{14}BrNO_2Si$
$M_r = 288.22$
Orthorhombic
$P2_12_12_1$
$a = 10.303 (2)$ Å
$b = 23.585 (3)$ Å
$c = 5.335 (3)$ Å
$V = 1296.3 (9)$ Å^3
$Z = 4$
$D_x = 1.477$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 28.10–55.76°$
$\mu = 5.079$ mm^{-1}
$T = 293 (2)$ K
Needle
$1.00 \times 0.35 \times 0.30$ mm
Transparent

Data collection

Rigaku AFC-6S diffractometer
ω scans with profile analysis
Absorption correction:
  ψ scan (North, Phillips & Mathews, 1968)
  $T_{min} = 0.76$, $T_{max} = 1.00$
1533 measured reflections
1533 independent reflections

1072 observed reflections
$[I > 2\sigma(I)]$
$\theta_{max} = 75.10°$
$h = -7 \rightarrow 12$
$k = -23 \rightarrow 29$
$l = -6 \rightarrow 6$
3 standard reflections
  monitored every 150 reflections
  intensity decay: 0.54%

Refinement

Refinement on F^2
$R(F) = 0.0417$
$wR(F^2) = 0.1257$
$S = 1.098$
1531 reflections
138 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 1.9005P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.004$

$\Delta\rho_{max} = 0.309$ e Å^{-3}
$\Delta\rho_{min} = -0.559$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration:
  Flack (1983) parameter = 0.56 (7)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (1)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Br1	0.40627 (9)	0.51255 (4)	0.1097 (3)	0.0943 (4)
Si1	1.2237 (2)	0.69320 (8)	−0.2377 (4)	0.0525 (5)
O9	1.1113 (4)	0.6712 (2)	−0.0272 (9)	0.0557 (13)
O13	0.9607 (5)	0.6472 (2)	−0.3141 (9)	0.0599 (14)
N7	0.9269 (5)	0.6322 (2)	0.1086 (13)	0.0512 (14)
C1	0.8040 (6)	0.6051 (2)	0.0970 (14)	0.0426 (14)
C2	0.7153 (6)	0.6152 (3)	−0.0909 (17)	0.057 (2)
C3	0.5975 (6)	0.5868 (3)	−0.0857 (16)	0.057 (2)
C4	0.5676 (6)	0.5513 (3)	0.1073 (18)	0.057 (2)
C5	0.6537 (8)	0.5422 (3)	0.2967 (19)	0.067 (2)
C6	0.7739 (8)	0.5691 (3)	0.2952 (15)	0.056 (2)
C8	0.9968 (6)	0.6493 (3)	−0.0955 (16)	0.052 (2)
C10	1.3613 (7)	0.7142 (4)	−0.0355 (19)	0.077 (3)
C11	1.2712 (8)	0.6326 (3)	−0.4360 (16)	0.067 (2)
C12	1.1593 (8)	0.7548 (3)	−0.4122 (17)	0.074 (2)

Table 2. *Selected geometric parameters (Å, °) for (1)*

Br1—C4	1.897 (6)	O9—C8	1.339 (7)
Si1—O9	1.695 (5)	O13—C8	1.225 (9)
Si1—C11	1.843 (8)	N7—C8	1.366 (10)
Si1—C12	1.850 (8)	N7—C1	1.419 (7)
Si1—C10	1.848 (8)		
O9—Si1—C11	108.9 (3)	C8—N7—C1	124.6 (7)
O9—Si1—C12	109.2 (3)	O13—C8—O9	122.8 (7)
O9—Si1—C10	102.7 (3)	O13—C8—N7	125.9 (6)
C8—O9—Si1	122.7 (5)	O9—C8—N7	111.2 (7)
C10—Si1—O9—C8	−176.4 (5)	Si1—O9—C8—N7	176.8 (4)
C8—N7—C1—C2	−30.9 (10)	C1—N7—C8—O13	5.1 (11)
Si1—O9—C8—O13	−5.5 (10)	C1—N7—C8—O9	−177.3 (6)

Compound (2)

Crystal data

$C_{14}H_{23}NO_2Si$
$M_r = 265.42$
Monoclinic
Cc
$a = 18.508 (5)$ Å
$b = 6.240 (9)$ Å
$c = 28.128 (6)$ Å
$\beta = 94.57 (2)°$
$V = 3238.2 (49)$ Å^3
$Z = 8$
$D_x = 1.089$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 23 reflections
$\theta = 27.08–51.77°$
$\mu = 1.241$ mm^{-1}
$T = 293 (2)$ K
Needle
$1.00 \times 0.30 \times 0.30$ mm
Transparent

Data collection

Rigaku AFC-6S diffractometer
$\omega/2\theta$ scans
Absorption correction:
  ψ scan (North, Phillips & Mathews, 1968)
  $T_{min} = 0.907$, $T_{max} = 1.000$
3306 measured reflections
3306 independent reflections

1827 observed reflections
$[I > 2\sigma(I)]$
$\theta_{max} = 75.21°$
$h = 0 \rightarrow 23$
$k = 0 \rightarrow 7$
$l = -35 \rightarrow 35$
3 standard reflections
  monitored every 150 reflections
  intensity decay: 0.96%

Refinement

Refinement on F^2
$R(F) = 0.0434$
$wR(F^2) = 0.1435$

$\Delta\rho_{max} = 0.209$ e Å^{-3}
$\Delta\rho_{min} = -0.294$ e Å^{-3}
Extinction correction: none

$S = 1.082$
3303 reflections
327 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 2.6934P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

Atomic scattering factors
from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration:
Flack (1983) parameter = 0.23 (5)

C1—N7—C8—O9	−14.8 (9)	C1'—N7'—C8'—O9'	4.0 (8)
C14—N7—C8—O9	175.3 (6)	C14'—N7'—C8'—O9'	177.9 (5)
N7—C8—O9—Si1	179.4 (4)	N7'—C8'—O9'—Si1'	−170.5 (4)
C10—Si1—O9—C8	174.2 (6)	C10'—Si1'—O9'—C8'	174.7 (5)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Si1	1.03601 (9)	−0.0336 (3)	0.53953 (7)	0.0592 (5)
C1	1.2432 (3)	0.2613 (11)	0.5356 (2)	0.0513 (15)
C2	1.2432 (4)	0.4390 (13)	0.5644 (2)	0.066 (2)
C3	1.2871 (5)	0.4419 (15)	0.6071 (3)	0.085 (2)
C4	1.3297 (4)	0.2720 (16)	0.6195 (3)	0.085 (2)
C5	1.3308 (4)	0.0977 (15)	0.5911 (3)	0.087 (3)
C6	1.2870 (4)	0.0917 (12)	0.5489 (2)	0.067 (2)
N7	1.1992 (2)	0.2572 (9)	0.4909 (2)	0.0561 (13)
C8	1.1300 (3)	0.1829 (11)	0.4901 (2)	0.054 (2)
O9	1.1181 (2)	0.0755 (8)	0.53022 (15)	0.0695 (14)
C10	1.0565 (5)	−0.1795 (15)	0.5959 (3)	0.098 (3)
C11	0.9700 (4)	0.1838 (14)	0.5461 (3)	0.099 (3)
C12	1.0061 (4)	−0.2223 (13)	0.4923 (3)	0.080 (2)
O13	1.0831 (2)	0.2088 (9)	0.4585 (2)	0.0730 (14)
C14	1.2253 (3)	0.3676 (11)	0.4475 (2)	0.057 (2)
C15	1.2120 (4)	0.2220 (12)	0.4045 (2)	0.074 (2)
C16	1.1877 (4)	0.5811 (11)	0.4392 (3)	0.078 (2)
C17	1.3064 (4)	0.4051 (16)	0.4560 (3)	0.091 (3)
Si1'	0.69433 (9)	0.2270 (3)	0.25114 (7)	0.0557 (5)
C1'	0.9077 (3)	0.4816 (9)	0.2665 (2)	0.0440 (14)
C2'	0.9545 (3)	0.4154 (11)	0.2334 (2)	0.057 (2)
C3'	0.9725 (4)	0.5552 (13)	0.1981 (2)	0.068 (2)
C4'	0.9426 (4)	0.7560 (13)	0.1949 (2)	0.070 (2)
C5'	0.8968 (4)	0.8228 (12)	0.2280 (3)	0.070 (2)
C6'	0.8793 (4)	0.6843 (10)	0.2635 (2)	0.0517 (15)
N7'	0.8897 (2)	0.3386 (8)	0.3045 (2)	0.0480 (12)
C8'	0.8231 (3)	0.2459 (11)	0.3012 (2)	0.0515 (14)
O9'	0.7841 (2)	0.2912 (7)	0.26066 (14)	0.0594 (12)
C10'	0.6698 (4)	0.3513 (14)	0.1929 (2)	0.080 (2)
C11'	0.6852 (4)	−0.0661 (14)	0.2483 (3)	0.090 (3)
C12'	0.6431 (4)	0.3452 (14)	0.2975 (3)	0.083 (2)
O13'	0.8011 (2)	0.1291 (8)	0.3316 (2)	0.0704 (14)
C14'	0.9407 (3)	0.3112 (10)	0.3488 (2)	0.0482 (14)
C15'	0.9053 (4)	0.4061 (13)	0.3910 (3)	0.077 (2)
C16'	0.9599 (4)	0.0774 (10)	0.3565 (2)	0.068 (2)
C17'	1.0105 (3)	0.4359 (14)	0.3433 (3)	0.079 (2)

Table 4. *Selected geometric parameters (Å, °) for (2)*

Si1—O9	1.704 (4)	Si1'—O9'	1.710 (4)
C1—N7	1.445 (7)	C1'—N7'	1.450 (7)
N7—C8	1.361 (7)	N7'—C8'	1.358 (7)
N7—C14	1.513 (7)	N7'—C14'	1.512 (7)
C8—O13	1.203 (7)	C8'—O13'	1.218 (7)
C8—O9	1.345 (7)	C8'—O9'	1.330 (7)
O9—Si1—C12	111.6 (3)	O9'—Si1'—C12'	110.3 (3)
O9—Si1—C10	101.8 (3)	O9'—Si1'—C10'	102.1 (3)
O9—Si1—C11	109.1 (3)	O9'—Si1'—C11'	109.0 (3)
C8—N7—C1	119.0 (5)	C8'—N7'—C1'	118.2 (5)
C8—N7—C14	120.3 (5)	C8'—N7'—C14'	120.8 (5)
C1—N7—C14	119.9 (4)	C1'—N7'—C14'	120.7 (4)
O13—C8—O9	122.0 (5)	O13'—C8'—O9'	122.7 (5)
O13—C8—N7	126.0 (6)	O13'—C8'—N7'	124.0 (6)
O9—C8—N7	112.0 (5)	O9'—C8'—N7'	113.2 (5)
C8—O9—Si1	122.4 (4)	C8'—O9'—Si1'	122.5 (4)
C2—C1—N7—C8	−89.7 (8)	C2'—C1'—N7'—C8'	−105.4 (7)
C2—C1—N7—C14	80.2 (7)	C2'—C1'—N7'—C14'	80.7 (7)
C1—N7—C8—O13	164.2 (7)	C1'—N7'—C8'—O13'	−177.4 (6)

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