Aminosilanes derived from 1H-benzimidazole-2(3H)-thione

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Two new molecular structures, namely 1,3-bis(trimethylsilyl)-1H-benzimidazole-2(3H)-thione, $C_{13}H_{22}N_2SSi_2$, (2), and 1-trimethylsilyl-1H-benzimidazole-2(3H)-thione, $C_{10}H_{14}N_2SSi$, (3), are reported. Both systems were derived from 1H-benzimidazole-2(3H)-thione. Noncovalent C—H···π interactions between the centroid of the benzmidazole system and the SiMe$_3$ groups form helicoidal arrangements in (2). Dimerization of (3) results in the formation of $R_2^2(8)$ rings via N—H···S interactions, along with parallel π–π interactions between imidazole and benzene rings.

1. Introduction

1H-Benzimidazole-2(3H)-thione, (1) (see Scheme 1), is a planar molecule with two substitutable acidic H atoms. The N atoms of this molecule have demonstrated the ability to form Lewis acid–base coordination compounds. Under basic conditions, the corresponding salt of (1) has been shown to react with p-block elements (O'Sullivan & Wallis, 1972).

Scheme 1
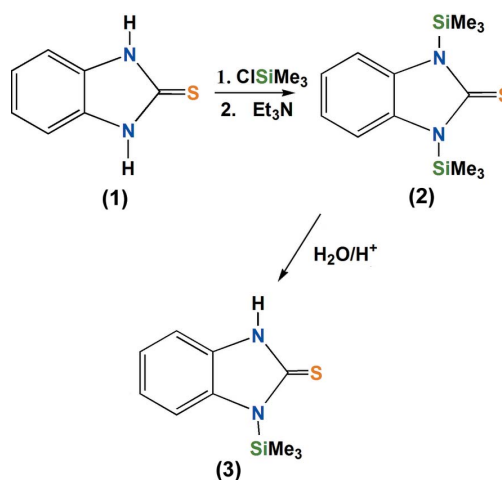
The 1H-benzimidazole-2(3H)-thione heterocycle has been found in compounds with biological activity, such as progesterone agonists (Zhang et al., 2007). Antinematode activity was evaluated for {[(1H-benzimidazol-2-yl)thio]acetyl}-piperazine (Mavrova et al., 2010), while 2-(alkylthio)-benzimidazole with a β-lactam ring presented antibacterial and antifungal activities (Desai & Desai, 2006). Isomeric 2-(methylthio)benzimidazole compounds were synthesized as
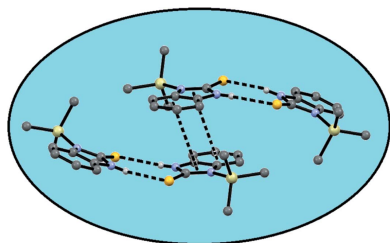
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Table 1
Experimental details.

	(2)	(3)
Crystal data		
Chemical formula	$C_{13}H_{22}N_2SSi_2$	$C_{10}H_{14}N_2SSi$
M_r	294.56	222.38
Crystal system, space group	Orthorhombic, $P2_12_12_1$	Monoclinic, $P2_1/c$
Temperature (K)	293	293
a, b, c (Å)	10.0302 (3), 10.6172 (3), 16.2428 (6)	9.8057 (2), 15.8032 (4), 15.8658 (5)
α, β, γ (°)	90, 90, 90	90, 93.859 (1), 90
V (Å^3)	1729.74 (10)	2453.01 (11)
Z	4	8
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.31	0.33
Crystal size (mm)	$0.25 \times 0.20 \times 0.10 \times 0.15$ (radius)	$0.20 \times 0.20 \times 0.15 \times 0.15$ (radius)
Data collection		
Diffractometer	Nonius KappaCCD area-detector diffractometer	Nonius KappaCCD area-detector diffractometer
Absorption correction	Spherical (Dwiggins, 1975)	Spherical (Dwiggins, 1975)
T_{min}, T_{max}	0.861, 0.862	0.861, 0.862
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15678, 3889, 2472	29355, 5554, 3199
R_{int}	0.064	0.096
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.648	0.649
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.104, 1.01	0.049, 0.138, 1.00
No. of reflections	3889	5554
No. of parameters	164	259
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.17, −0.20	0.21, −0.24
Absolute structure	Flack x parameter determined using 838 quotients, $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons et al., 2013)	–
Absolute structure parameter	0.01 (7)	–

Computer programs: *COLLECT* (Nonius, 2000), *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), and *XPMA* in *ZORTEP* (Zsolnai, 1997).

acyclic analogues of the HIV-1 RT inhibitor ring system (Gardiner & Loyns, 1995). More recently, isoxazole–mercaptobenzimidazole hybrids have presented analgesic and anti-inflammatory activities (Shravankumar et al., 2013). Furthermore, a wide range of biological activities have been reported for the benzimidazole fragment, such as antifungal, antibacterial, vasodilator, antispasmodic, anti-ulcer (Akkurt et al., 2012), antimicrobial (De Almeida et al., 2007), antihistamine (Mor et al., 2004), neutropic (Bakhareva et al., 1996) and analgesic (Anandarajagopal et al., 2010). Additionally, alkylsilyl-substituted benzimidazole has shown in vitro cytotoxicity, for example, 1-[3-(trimethylsilyl)propyl]benzimidazole inhibits carcinoma S180 tumour (Lukevics et al., 2001). In 2012, 1-{[dimethyl(phenyl)silyl]methyl}-3-(2-phenylethyl)-1-benzimidazol-3-ium bromide monohydrate was synthesized and its crystal structure elucidated (Akkurt et al., 2012). Silylated compounds are stable at low temperatures and, in some cases, under atmospheric conditions. Aminosilanes are soluble in nonpolar solvents, while the presence of trimethylsilyl groups increases the volatility of the organic fragments, most of which can be distilled without decomposition and, sometimes, even crystallized (Ghose & Gilchrist, 1991). Alkoxysilanes, thiosilanes and aminosilanes are stable at low temperatures, while the last class become unstable under atmospheric conditions (Colvin, 1981).

We report here the crystal structures of two new trimethylsilyl-substituted derivatives of 1H-benzimidazole-2(3H)-thione, namely 1,3-bis(trimethylsilyl)-1H-benzimidazole-2(3H)-thione, (2), and 1-trimethylsilyl-1H-benzimidazole-2(3H)-thione, (3).

2. Experimental

All reagents were purchased from Aldrich and were used as received. All solvents were dried before use. ^{1}H NMR (300.13185 MHz) and ^{13}C NMR (75.47564 MHz) analyses in CDCl$_3$ were performed on a Bruker 300 MHz spectrometer, using TMS as the internal reference. Chemical shifts (δ) are reported in p.p.m. IR spectra were recorded on a Perkin–Elmer FT–IR 1600 spectrophotometer in the 4000–400 cm^{-1} range. Elemental analyses were performed in a Thermofinniga Flash 112 instrument under standard conditions.

2.1. Synthesis and crystallization

Compound (2) was obtained by mixing 1H-benzimidazole-2(3H)-thione (0.5 g, 3.3 mmol) and chlorotrimethylsilane (0.89 ml, 75.9 mg, 6.9 mmol) in triethylamine (15 ml). The reaction was kept under constant stirring and reflux for 6 h. The resulting compound was a yellow liquid (yield 92%,
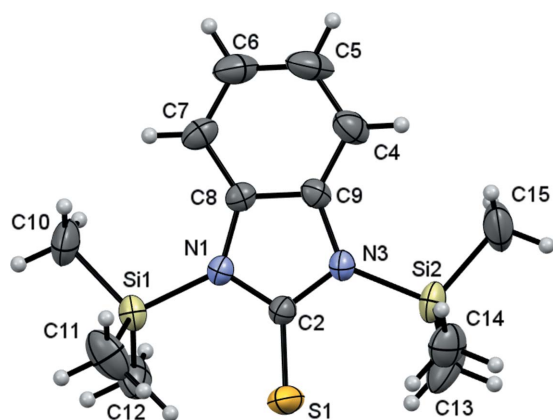
Figure 1
The molecular structure of compound (2), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.
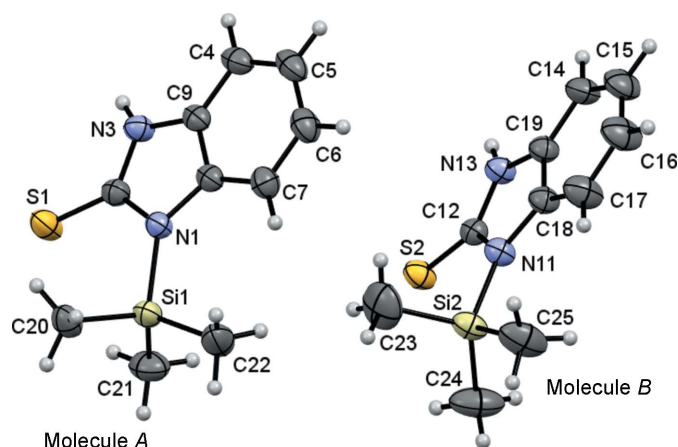

Figure 2
The molecular structures of the two independent molecules of compound (3), showing the atom-numbering schemes. Displacement ellipsoids are drawn at the 30% probability level.

1.87 g) which solidified after 24 h. Crystals of (2) suitable for X-ray diffraction analysis were collected. MS: m/z (intensity,

%): 294 (M^+, 100), 206 (25), 150 (11); IR (KBr, ν_{max}, cm^{-1}): 1623 (C=N), 1514 and 1470 (N—C—S), 1181 (Si—N), 714 and 710 (Si—C); ^{1}H NMR (C$_6$D$_6$/THF, 1:1): δ $AA'BB'$ 7.26 (m, H4, H7), 7.04 (m, H5, H6), 0.73 (s, H$_{Me}$); ^{13}C NMR: δ 182.3 (C2), 112.2 (C4, C7), 122.6 (C5, C6), 2.5 (C$_{Me}$). Elemental analysis calculated for C$_{13}$H$_{22}$N$_2$SSi$_2$: C 53.01, H 7.53, N 9.51, S 10.89%; found: C 53.03, H 7.60, N 9.60, S 10.69%.

Compound (3) was obtained from the partial hydrolysis of (2); both (2) and (3) are readily hydrolysed under atmospheric conditions. This compound was not analysed by spectroscopic techniques. However, crystals of (3) suitable for X-ray diffraction analysis were obtained from a hexane solution and a single crystal immersed in oil was analysed.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were included in geometrically calculated positions, riding on the C or N atoms to which they were bonded. C—H distances were restrained to 0.93 (aromatic) or 0.96 Å (methyl) and the N—H bond length was restrained to 0.86 Å. H-atom displacement parameters were set at U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and at 1.2U_{eq}(C,N) otherwise.

3. Results and discussion

Compound (2) crystallizes in the orthorhombic space group $P2_12_12_1$. The average N1—Si1—Me$_{10,11,12}$ angle is 109.0 (2)° and the average N1—Si1—Me$_{13,14,15}$ angle is 109.1 (2)°. The Si—N distances of 1.809 (3) and 1.803 (3) Å are slightly longer than those reported previously for 1,3-bis(trimethylsilyl)-imidazolidin-2-one [1.739 (7) Å] and 4-methyl-1,3-bis(tri-methylsilyl)imidazolidin-2-one [1.745 (3) Å] (Szalay *et al.*, 2005), which might be caused by the difference in electro-negativities of the O and S atoms.

Compound (3) crystallizes with two independent molecules, A and B, in the asymmetric unit in the monoclinic space group
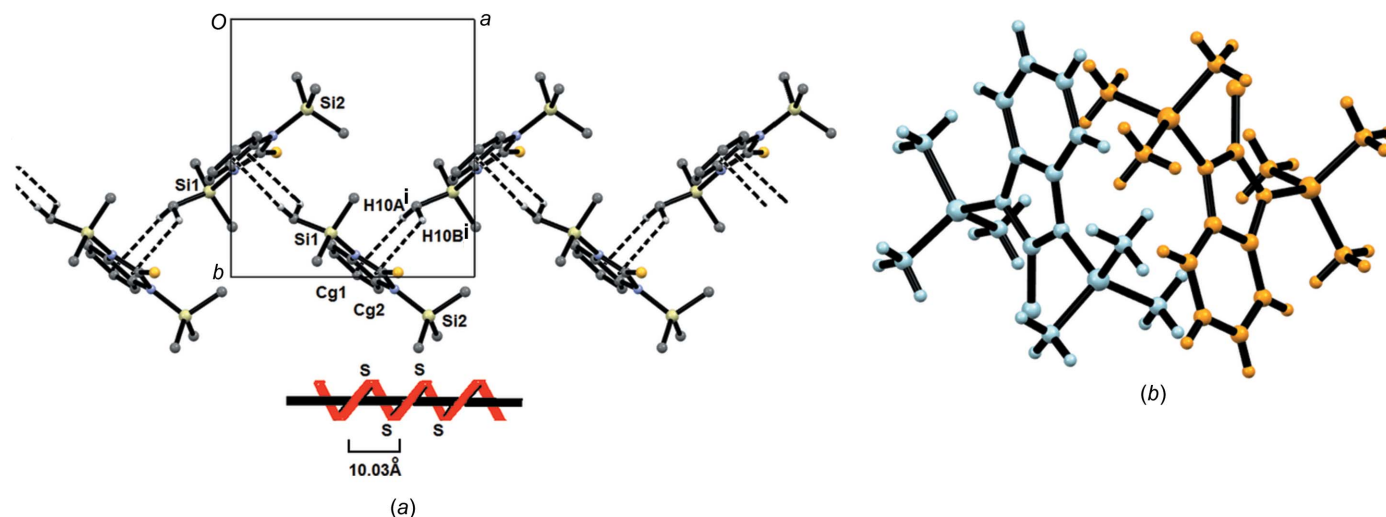

Figure 3
(a) The spiral arrangement for (2) and (b) the overlap of the helix along the direction of the a axis.

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

Si1—N1	1.809 (3)	Si2—C13	1.839 (6)
Si1—C11	1.842 (5)	Si2—C15	1.854 (6)
Si1—C12	1.842 (5)	Si2—C14	1.861 (5)
Si1—C10	1.847 (5)	S1—C2	1.669 (4)
Si2—N3	1.803 (3)		
N1—Si1—C11	109.0 (2)	N3—Si2—C14	109.3 (2)
N1—Si1—C12	109.53 (19)	C13—Si2—C14	113.7 (3)
C11—Si1—C12	113.9 (3)	C15—Si2—C14	107.7 (3)
N1—Si1—C10	108.4 (2)	C2—N1—Si1	121.7 (3)
C11—Si1—C10	109.4 (3)	C8—N1—Si1	130.9 (3)
C12—Si1—C10	106.4 (3)	C2—N3—Si2	120.8 (3)
N3—Si2—C13	109.4 (2)	C9—N3—Si2	132.3 (2)
N3—Si2—C15	108.5 (2)	N1—C2—S1	125.1 (3)
C13—Si2—C15	108.2 (3)	N3—C2—S1	124.8 (3)
C11—Si1—N1—C2	70.3 (4)	C14—Si2—N3—C9	113.9 (4)
C12—Si1—N1—C2	−55.0 (4)	Si2—N3—C9—C4	−4.8 (7)
C10—Si1—N1—C2	−170.7 (3)	Si2—N3—C9—C8	179.1 (3)
C11—Si1—N1—C8	−113.2 (4)	Si1—N1—C8—C7	10.2 (6)
C12—Si1—N1—C8	121.5 (4)	Si1—N1—C8—C9	−174.1 (3)
C10—Si1—N1—C8	5.8 (4)	Si1—N1—C2—N3	173.6 (2)
C13—Si2—N3—C2	59.4 (4)	C8—N1—C2—S1	175.3 (3)
C15—Si2—N3—C2	177.2 (4)	Si1—N1—C2—S1	−7.5 (5)
C14—Si2—N3—C2	−65.7 (4)	Si2—N3—C2—N1	−177.2 (2)
C13—Si2—N3—C9	−121.0 (4)	C9—N3—C2—S1	−175.9 (3)
C15—Si2—N3—C9	−3.2 (4)	Si2—N3—C2—S1	3.9 (5)

Table 3
Selected geometric parameters (Å, °) for (3).

S1—C2	1.676 (3)	S2—C12	1.675 (2)
Si1—N1	1.817 (2)	Si2—N11	1.804 (2)
Si1—C22	1.841 (3)	Si2—C24	1.827 (3)
Si1—C20	1.846 (3)	Si2—C23	1.830 (4)
Si1—C21	1.850 (3)	Si2—C25	1.841 (3)
N1—Si1—C22	108.72 (12)	N11—Si2—C24	111.21 (15)
N1—Si1—C20	107.62 (12)	N11—Si2—C23	105.51 (15)
C22—Si1—C20	109.24 (16)	C24—Si2—C23	113.3 (2)
N1—Si1—C21	109.12 (13)	N11—Si2—C25	109.27 (13)
C22—Si1—C21	108.81 (18)	C24—Si2—C25	106.95 (19)
C20—Si1—C21	113.23 (16)	C23—Si2—C25	110.6 (2)
C2—N1—Si1	122.00 (16)	C12—N11—Si2	123.12 (16)
C8—N1—Si1	130.56 (17)	C18—N11—Si2	128.88 (17)
N3—C2—S1	125.48 (19)	N13—C12—S2	125.02 (19)
N1—C2—S1	126.65 (18)	N11—C12—S2	127.12 (18)
C22—Si1—N1—C2	176.3 (2)	C24—Si2—N11—C12	56.7 (3)
C20—Si1—N1—C2	−65.5 (2)	C23—Si2—N11—C12	−66.5 (2)
C21—Si1—N1—C2	57.8 (2)	C25—Si2—N11—C12	174.5 (2)
C22—Si1—N1—C8	−1.1 (3)	C24—Si2—N11—C18	−133.4 (3)
C20—Si1—N1—C8	117.1 (2)	C23—Si2—N11—C18	103.4 (3)
C21—Si1—N1—C8	−119.6 (2)	C25—Si2—N11—C18	−15.6 (3)
C9—N3—C2—S1	179.14 (18)	C19—N13—C12—S2	−179.11 (18)
Si1—N1—C2—N3	−177.32 (16)	Si2—N11—C12—N13	171.28 (16)
C8—N1—C2—S1	−178.90 (19)	C18—N11—C12—S2	179.38 (19)
Si1—N1—C2—S1	3.2 (3)	Si2—N11—C12—S2	−8.8 (3)
Si1—N1—C8—C9	177.06 (17)	Si2—N11—C18—C17	8.5 (5)
Si1—N1—C8—C7	−3.0 (4)	Si2—N11—C18—C19	−171.09 (18)

$P2_1/c$. The average N1—Si1—Me$_{20,21,22}$ angle is 108.49 (12)° and the average N11—Si2—Me$_{23,24,25}$ angle is 108.66 (12)°. The Si—N distances are 1.817 (2) and 1.804 (2) Å.

Overall, compounds (2) and (3) have very similar structures, which are shown in Figs. 1 and 2, respectively. Selected bond lengths and angles are listed in Tables 2 and 3, respectively. The average C—Si bond length for both compounds is 1.847 (3) Å and the average C—Si—C angle is 109.5 (2)°, in agreement with sp^3-hybridization of the Si atoms. These values

agree with those in similar structures reported previously (Wagler et al., 2010).

The C=S distances for compounds (2) and (3) range from 1.669 (4) to 1.675 (2) Å. The average N$_{1,3}$—C2=S1 angle is 125.0 (3)° for (2) and the average N$_{1,11}$—C$_{2,12}$=S12 angle is 126.9 (18)° for (3). These angles agree with sp^2-hybridization of the C and S atoms which is typical of thiourea groups (Wagler et al., 2010). The S atom of (3) has a slight displace-
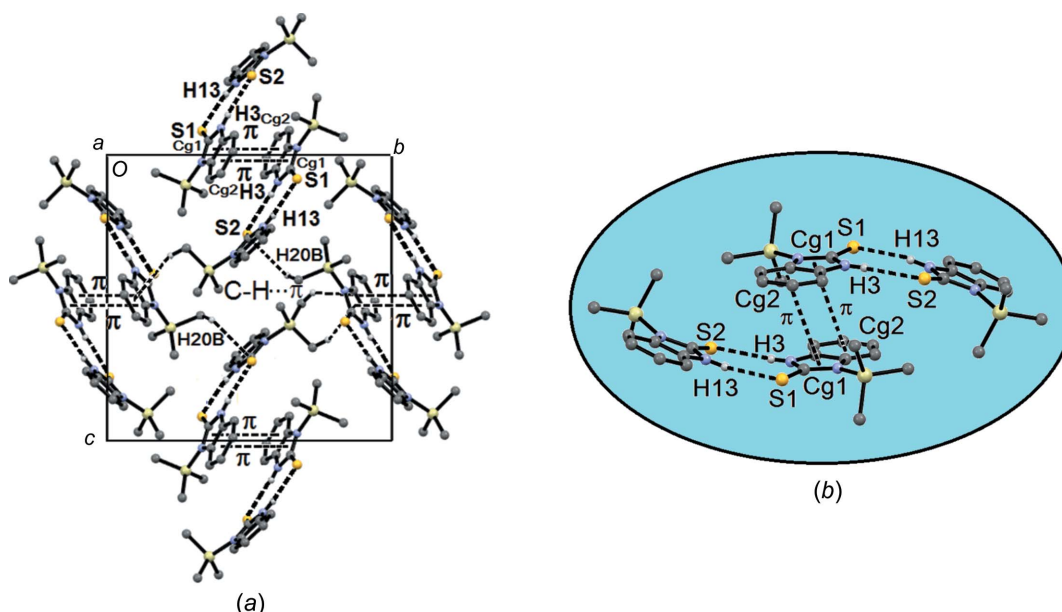

Figure 4
(a) The crystal packing diagram of (3) along the direction of the ab plane. (b) A detailed view of the formation of the $R_2^2(8)$ hydrogen-bonding motif and the π–π stacking interactions.

Table 4
Hydrogen-bond geometry (Å, °) for (2).

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11B···S1	0.96	2.96	3.564 (7)	122
C12—H12C···S1	0.96	2.77	3.415 (5)	125
C13—H13B···S1	0.96	2.79	3.423 (7)	125
C14—H14C···S1	0.96	2.86	3.480 (5)	123

Table 5
Hydrogen-bond geometry (Å, °) for (3).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···S2^i	0.86	2.52	3.374 (2)	170
N13—H13···S1^i	0.86	2.45	3.282 (2)	164
C21—H21B···S1	0.96	2.83	3.480 (4)	126

Symmetry code: (i) $-x + 1, -y + 1, -z$.

ment of 0.007 (1) Å from the benzimidazole molecular plane, whereas in (2), the S atom is out of the plane by 0.155 (2) Å. This displacement could be caused by noncovalent intramolecular interactions between the S-atom nucleus and both Si atoms, or between the methyl H atoms and the S atom. Compound (2) presents four noncovalent C—H···S interactions (Table 4), with C···S distances ranging from 2.77 to 2.96 Å and angles ranging from 122 to 125°, which amount to less than the sum of the van der Waals radii of S and H atoms (3.25 Å; Bondi, 1964).

Another noncovalent intramolecular interaction (Table 5) was observed in (3), *viz.* C21—H21···S1, with a C···S distance of 2.83 Å and an angle of 126°, similar to that observed in (2).

Comparing the structures of (2) and (3), it becomes obvious that the fused rings in (2) are not completely flat. Specifically, the thiourea unit composed of atoms N1/C2/N3/S1 is offset from the molecular plane defined by the benzene ring. This is a consequence of the intramolecular noncovalent C—H···S interactions present in the system.

Fig. 3(*a*) shows the spiral arrangement of (2), which forms a linking interaction between molecules through the imidazole ring (C10—H10A···Cg1 = 2.94 Å; Cg1 is the centroid of the imidazole ring) and the benzene ring [C10—H10B···Cg2 = 2.83 Å; Cg2 is the centroid of the benzene ring at ($x - \frac{1}{2}, -y + \frac{3}{2}, -z$)]. These interactions form a helicoidal repeat unit of 10.03 Å, which extends along the crystallographic *a* axis. Fig. 3(*b*) presents the helix overlap of this system. A third interaction, *viz.* C13—H13···π($x + \frac{1}{2}, -y + \frac{1}{2}, -z$), has a C···$\pi$ distance of 2.77 Å, which further supports the helicoidal arrangement.

Molecules A and B of (3) are auto-assembled by N—H···S interactions (N3—H3···S2^i = 2.52 Å and N13—H13···S1^i = 2.45 Å; see Table 5 for symmetry code). This arrangement forms a cyclic system with an $R_2^2(8)$ hydrogen-bonding pattern (Bernstein *et al.*, 1995) (Fig. 4). Furthermore, π–π interactions between the imidazole and benzene rings are observed in the dimerization of the compound and extend in the *ab* plane (Fig. 4). The distance between the ring centroids in these interactions is 3.64 Å (symmetry code: $-x + 1, -y + 1, -z$). There is an additional intermolecular C20—H20B···

π(imidazole ring) interaction of 3.03 Å (symmetry code: $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$) which strengthens the crystalline arrangement of (3).

As can be seen, the structures of (2) and (3) have similar parameters around the silyl–amine bond, but while (3) is a dimer formed by classical hydrogen bonding, the structure of (2) is a helix supported by nonclassical interactions.

References

Akkurt, M., Küçükbay, H., Sireci, N. & Büyükgüngör, O. (2012). *Acta Cryst.* E**68**, o2718–o2719.

Anandarajagopal, K., Tiwari, R. N., Venkateshan, N., Vinotha Pooshan, G. & Promwichit, P. (2010). *J. Chem. Pharm. Res.* **2**(3), 230–236.

Bakhareva, E., Voronkov, M., Sorokin, M., Lopyrev, V., Seredenin, S. & Gaidarov, G. M. (1996). *Pharm. Chem. J.* **30**, 89–91.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.

Colvin, E. W. (1981). *Silicon in Organic Synthesis*, ch. 19. London: Butterworth and Co.

De Almeida, M. V., Cardoso, S. H., De Assis, J. V. & De Souza, M. V. N. (2007). *J. Sulfur Chem.* **28**, 17–22.

Desai, K. G. & Desai, K. R. (2006). *Bioorg. Med. Chem.* **14**, 8271–8279.

Dwiggins, C. W. (1975). *Acta Cryst.* A**31**, 146–148.

Gardiner, J. & Loyns, C. (1995). *Tetrahedron*, **51**, 11515–11530.

Ghose, S. & Gilchrist, T. L. (1991). *J. Chem. Soc. Perkin Trans. 1*, pp. 775–780.

Lukevics, E., Arsenyan, P., Shestakova, I., Domracheva, I., Nesterova, A. & Pudova, O. (2001). *Eur. J. Med. Chem.* **36**, 507–515.

Mavrova, A. T., Vuchev, D., Anichina, K. & Vassilev, N. (2010). *Eur. J. Med. Chem.* **45**, 5856–5861.

Mor, M., Bordi, F., Silva, C., Rivara, S., Zuliani, V., Vacondio, F., Rivara, M., Barocelli, E., Bertoni, S., Ballabeni, V., Magnanini, F., Impicciatore, M. & Plazzi, P. V. (2004). *Bioorg. Med. Chem.* **12**, 663–674.

Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.

O'Sullivan, D. G. & Wallis, A. K. (1972). *J. Med. Chem.* **15**, 103–104.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Shravankumar, K., Ranjith, K., Prasad, G., Niranjan, T., Srinivas, N., Mohan, R., Hanmanthu, G., Mukkanti, K., Ravinder, V. & Chandra, S. (2013). *Bioorg. Med. Chem. Lett.* **23**, 1306–1309.

Szalay, R., Pongor, G., Harmat, V., Böcskei, Z. & Knausz, D. (2005). *J. Organomet. Chem.* **690**, 1498–1506.

Wagler, J., Heine, T. & Hill, F. A. (2010). *Organometallics*, **29**, 5607–5613.

Zhang, P., Terefenko, E., Kern, J., Fensome, A., Trybulski, E., Unwalla, R., Wrobel, J., Lockhead, S., Zhu, Y., Cohen, J., LaCava, M., Winneker, R. & Zhang, Z. (2007). *Bioorg. Med. Chem.* **15**, 6556–6564.

Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.

supporting information

Aminosilanes derived from 1*H*-benzimidazole-2(3*H*)-thione

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Computing details

For both compounds, data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: XPMA (Zsolnai, 1997); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015).

(2) 1,3-Bis(trimethylsilyl)-1*H*-benzimidazole-2(3*H*)-thione

Crystal data

$C_{13}H_{22}N_2SSi_2$
$M_r = 294.56$
Orthorhombic, $P2_12_12_1$
$a = 10.0302\ (3)$ Å
$b = 10.6172\ (3)$ Å
$c = 16.2428\ (6)$ Å
$V = 1729.74\ (10)$ Å^3
$Z = 4$
$F(000) = 632$

$D_x = 1.131$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 600 reflections
$\theta = 20–25°$
$\mu = 0.31$ mm^{-1}
$T = 293$ K
Prism, colourless
$0.25 \times 0.20 \times 0.10 \times 0.15$ (radius) mm

Data collection

Nonius Kappa CCD area-detector
  diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 3 pixels mm^{-1}
ω scans

Absorption correction: for a sphere
  Interpolation using Int. Tables Vol. C (1992) p.
  523, Table 6.3.3.3, for values of muR in the
  range 0–2.5, and Int. Tables Vol. II (1959) p.
  302, Table 5.3.6 B, for muR in the range 2.6–
  10.0. The interpolation procedure of Dwiggins
  (1975) is used with some modification.
$T_{min} = 0.861$, $T_{max} = 0.862$
15678 measured reflections
3889 independent reflections
2472 reflections with $I > 2\sigma(I)$
$R_{int} = 0.064$
$\theta_{max} = 27.4°$, $\theta_{min} = 3.8°$
$h = -12{\rightarrow}12$
$k = -12{\rightarrow}13$
$l = -20{\rightarrow}20$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0297P)^2 + 0.4977P]$
Least-squares matrix: full	where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.048$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.104$	$\Delta\rho_{max} = 0.17$ e Å⁻³
$S = 1.01$	$\Delta\rho_{min} = -0.20$ e Å⁻³
3889 reflections	Extinction correction: *SHELXL97* (Sheldrick,
164 parameters	2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.008 (2)
Hydrogen site location: inferred from	Absolute structure: Flack x parameter
neighbouring sites	determined using 838 quotients
H-atom parameters constrained	$[(I+)-(I-)]/[(I+)+(I-)]$ (Parsons *et al.*, 2013)
	Absolute structure parameter: 0.01 (7)

Special details

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

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

	x	y	z	$U_{iso}*/U_{eq}$
Si1	0.40978 (12)	0.83025 (11)	0.87960 (8)	0.0615 (3)
Si2	0.81188 (12)	1.15207 (13)	0.98310 (9)	0.0735 (4)
S1	0.68527 (14)	0.98333 (17)	0.83494 (7)	0.0965 (6)
N1	0.5113 (3)	0.9147 (3)	0.95331 (19)	0.0481 (8)
N3	0.6713 (3)	1.0480 (3)	0.99562 (19)	0.0522 (8)
C9	0.5940 (4)	1.0147 (4)	1.0649 (2)	0.0514 (9)
C8	0.4962 (4)	0.9315 (3)	1.0387 (2)	0.0485 (9)
C7	0.4107 (4)	0.8755 (4)	1.0949 (3)	0.0668 (12)
H7	0.3466	0.8178	1.0780	0.080*
C2	0.6206 (4)	0.9823 (4)	0.9296 (2)	0.0525 (9)
C4	0.6060 (5)	1.0450 (5)	1.1472 (3)	0.0802 (15)
H4	0.6718	1.1003	1.1652	0.096*
C5	0.5186 (6)	0.9917 (7)	1.2017 (3)	0.1007 (19)
H5	0.5237	1.0128	1.2572	0.121*
C6	0.4237 (6)	0.9077 (6)	1.1759 (3)	0.0923 (17)
H6	0.3667	0.8717	1.2145	0.111*
C14	0.9616 (4)	1.0580 (6)	0.9550 (4)	0.0990 (19)
H14A	1.0367	1.1132	0.9482	0.148*
H14B	0.9802	0.9982	0.9978	0.148*
H14C	0.9452	1.0140	0.9043	0.148*
C12	0.3532 (5)	0.9393 (5)	0.7986 (3)	0.1019 (19)
H12A	0.2994	0.8944	0.7596	0.153*
H12B	0.3015	1.0057	0.8230	0.153*
H12C	0.4292	0.9747	0.7711	0.153*
C13	0.7699 (6)	1.2751 (5)	0.9077 (5)	0.134 (3)
H13A	0.8447	1.3306	0.9010	0.201*
H13B	0.7488	1.2367	0.8559	0.201*

H13C	0.6945	1.3222	0.9270	0.201*
C11	0.5048 (7)	0.6943 (5)	0.8405 (4)	0.122 (2)
H11A	0.4515	0.6490	0.8013	0.184*
H11B	0.5849	0.7234	0.8143	0.184*
H11C	0.5274	0.6397	0.8855	0.184*
C10	0.2582 (5)	0.7739 (6)	0.9326 (4)	0.114 (2)
H10A	0.2031	0.7288	0.8943	0.172*
H10B	0.2830	0.7190	0.9770	0.172*
H10C	0.2097	0.8446	0.9540	0.172*
C15	0.8462 (6)	1.2293 (6)	1.0833 (4)	0.124 (2)
H15A	0.9209	1.2852	1.0775	0.186*
H15B	0.7693	1.2765	1.1002	0.186*
H15C	0.8662	1.1664	1.1239	0.186*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0585 (7)	0.0565 (7)	0.0696 (8)	−0.0086 (6)	−0.0095 (6)	−0.0071 (6)
Si2	0.0477 (6)	0.0730 (8)	0.0999 (10)	−0.0160 (6)	−0.0084 (7)	0.0001 (7)
S1	0.0760 (8)	0.1618 (15)	0.0517 (6)	−0.0372 (10)	0.0097 (6)	0.0012 (8)
N1	0.0434 (16)	0.0495 (18)	0.052 (2)	−0.0058 (14)	0.0010 (15)	0.0006 (14)
N3	0.0455 (16)	0.0559 (19)	0.055 (2)	−0.0098 (14)	−0.0040 (16)	−0.0011 (15)
C9	0.045 (2)	0.061 (2)	0.048 (2)	0.008 (2)	−0.0043 (18)	−0.0040 (19)
C8	0.043 (2)	0.051 (2)	0.052 (2)	0.0071 (18)	0.0027 (19)	0.0041 (18)
C7	0.055 (2)	0.074 (3)	0.072 (3)	0.004 (2)	0.013 (2)	0.013 (2)
C2	0.046 (2)	0.062 (2)	0.050 (2)	−0.0074 (18)	−0.0001 (17)	0.003 (2)
C4	0.065 (3)	0.115 (4)	0.061 (3)	0.004 (3)	−0.011 (2)	−0.017 (3)
C5	0.082 (4)	0.175 (6)	0.046 (3)	0.023 (4)	0.002 (3)	−0.003 (4)
C6	0.073 (3)	0.142 (5)	0.061 (3)	0.012 (4)	0.014 (3)	0.021 (3)
C14	0.050 (3)	0.129 (5)	0.118 (5)	−0.004 (3)	−0.001 (3)	0.002 (4)
C12	0.105 (4)	0.103 (4)	0.098 (4)	−0.024 (3)	−0.045 (3)	0.018 (3)
C13	0.096 (4)	0.095 (4)	0.212 (8)	−0.027 (3)	−0.011 (5)	0.063 (5)
C11	0.139 (5)	0.100 (4)	0.129 (5)	0.029 (4)	−0.026 (5)	−0.054 (4)
C10	0.085 (4)	0.126 (5)	0.132 (5)	−0.057 (4)	−0.011 (4)	0.001 (4)
C15	0.098 (4)	0.127 (5)	0.148 (6)	−0.045 (4)	−0.001 (4)	−0.057 (4)

Geometric parameters (Å, º)

Si1—N1	1.809 (3)	C5—H5	0.9300
Si1—C11	1.842 (5)	C6—H6	0.9300
Si1—C12	1.842 (5)	C14—H14A	0.9600
Si1—C10	1.847 (5)	C14—H14B	0.9600
Si2—N3	1.803 (3)	C14—H14C	0.9600
Si2—C13	1.839 (6)	C12—H12A	0.9600
Si2—C15	1.854 (6)	C12—H12B	0.9600
Si2—C14	1.861 (5)	C12—H12C	0.9600
S1—C2	1.669 (4)	C13—H13A	0.9600
N1—C2	1.366 (4)	C13—H13B	0.9600

N1—C8	1.406 (5)	C13—H13C	0.9600
N3—C2	1.376 (5)	C11—H11A	0.9600
N3—C9	1.412 (5)	C11—H11B	0.9600
C9—C4	1.381 (5)	C11—H11C	0.9600
C9—C8	1.387 (5)	C10—H10A	0.9600
C8—C7	1.387 (5)	C10—H10B	0.9600
C7—C6	1.366 (7)	C10—H10C	0.9600
C7—H7	0.9300	C15—H15A	0.9600
C4—C5	1.369 (7)	C15—H15B	0.9600
C4—H4	0.9300	C15—H15C	0.9600
C5—C6	1.370 (8)		
N1—Si1—C11	109.0 (2)	C5—C6—H6	119.2
N1—Si1—C12	109.53 (19)	Si2—C14—H14A	109.5
C11—Si1—C12	113.9 (3)	Si2—C14—H14B	109.5
N1—Si1—C10	108.4 (2)	H14A—C14—H14B	109.5
C11—Si1—C10	109.4 (3)	Si2—C14—H14C	109.5
C12—Si1—C10	106.4 (3)	H14A—C14—H14C	109.5
N3—Si2—C13	109.4 (2)	H14B—C14—H14C	109.5
N3—Si2—C15	108.5 (2)	Si1—C12—H12A	109.5
C13—Si2—C15	108.2 (3)	Si1—C12—H12B	109.5
N3—Si2—C14	109.3 (2)	H12A—C12—H12B	109.5
C13—Si2—C14	113.7 (3)	Si1—C12—H12C	109.5
C15—Si2—C14	107.7 (3)	H12A—C12—H12C	109.5
C2—N1—C8	107.3 (3)	H12B—C12—H12C	109.5
C2—N1—Si1	121.7 (3)	Si2—C13—H13A	109.5
C8—N1—Si1	130.9 (3)	Si2—C13—H13B	109.5
C2—N3—C9	106.9 (3)	H13A—C13—H13B	109.5
C2—N3—Si2	120.8 (3)	Si2—C13—H13C	109.5
C9—N3—Si2	132.3 (2)	H13A—C13—H13C	109.5
C4—C9—C8	120.5 (4)	H13B—C13—H13C	109.5
C4—C9—N3	131.7 (4)	Si1—C11—H11A	109.5
C8—C9—N3	107.7 (3)	Si1—C11—H11B	109.5
C7—C8—C9	120.6 (4)	H11A—C11—H11B	109.5
C7—C8—N1	131.4 (4)	Si1—C11—H11C	109.5
C9—C8—N1	107.9 (3)	H11A—C11—H11C	109.5
C6—C7—C8	117.9 (5)	H11B—C11—H11C	109.5
C6—C7—H7	121.1	Si1—C10—H10A	109.5
C8—C7—H7	121.1	Si1—C10—H10B	109.5
N1—C2—N3	110.1 (3)	H10A—C10—H10B	109.5
N1—C2—S1	125.1 (3)	Si1—C10—H10C	109.5
N3—C2—S1	124.8 (3)	H10A—C10—H10C	109.5
C5—C4—C9	118.3 (5)	H10B—C10—H10C	109.5
C5—C4—H4	120.8	Si2—C15—H15A	109.5
C9—C4—H4	120.8	Si2—C15—H15B	109.5
C4—C5—C6	121.1 (5)	H15A—C15—H15B	109.5
C4—C5—H5	119.5	Si2—C15—H15C	109.5
C6—C5—H5	119.5	H15A—C15—H15C	109.5

C7—C6—C5	121.6 (5)	H15B—C15—H15C	109.5
C7—C6—H6	119.2		
C11—Si1—N1—C2	70.3 (4)	C2—N1—C8—C7	−172.9 (4)
C12—Si1—N1—C2	−55.0 (4)	Si1—N1—C8—C7	10.2 (6)
C10—Si1—N1—C2	−170.7 (3)	C2—N1—C8—C9	2.8 (4)
C11—Si1—N1—C8	−113.2 (4)	Si1—N1—C8—C9	−174.1 (3)
C12—Si1—N1—C8	121.5 (4)	C9—C8—C7—C6	1.8 (6)
C10—Si1—N1—C8	5.8 (4)	N1—C8—C7—C6	177.1 (4)
C13—Si2—N3—C2	59.4 (4)	C8—N1—C2—N3	−3.7 (4)
C15—Si2—N3—C2	177.2 (4)	Si1—N1—C2—N3	173.6 (2)
C14—Si2—N3—C2	−65.7 (4)	C8—N1—C2—S1	175.3 (3)
C13—Si2—N3—C9	−121.0 (4)	Si1—N1—C2—S1	−7.5 (5)
C15—Si2—N3—C9	−3.2 (4)	C9—N3—C2—N1	3.1 (4)
C14—Si2—N3—C9	113.9 (4)	Si2—N3—C2—N1	−177.2 (2)
C2—N3—C9—C4	174.9 (5)	C9—N3—C2—S1	−175.9 (3)
Si2—N3—C9—C4	−4.8 (7)	Si2—N3—C2—S1	3.9 (5)
C2—N3—C9—C8	−1.2 (4)	C8—C9—C4—C5	−0.5 (7)
Si2—N3—C9—C8	179.1 (3)	N3—C9—C4—C5	−176.2 (4)
C4—C9—C8—C7	−1.3 (6)	C9—C4—C5—C6	1.7 (8)
N3—C9—C8—C7	175.3 (3)	C8—C7—C6—C5	−0.6 (8)
C4—C9—C8—N1	−177.6 (4)	C4—C5—C6—C7	−1.2 (9)
N3—C9—C8—N1	−1.0 (4)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11*B*···S1	0.96	2.96	3.564 (7)	122
C12—H12*C*···S1	0.96	2.77	3.415 (5)	125
C13—H13*B*···S1	0.96	2.79	3.423 (7)	125
C14—H14*C*···S1	0.96	2.86	3.480 (5)	123

(3) 1-Trimethylsilyl-1*H*-benzimidazole-2(3*H*)-thione

Crystal data

C₁₀H₁₄N₂SSi	$F(000) = 944$
$M_r = 222.38$	$D_x = 1.204$ Mg m⁻³
Monoclinic, $P2_1/c$	Mo *Kα* radiation, $λ = 0.71073$ Å
$a = 9.8057$ (2) Å	Cell parameters from 60 reflections
$b = 15.8032$ (4) Å	$θ = 20–25°$
$c = 15.8658$ (5) Å	$μ = 0.33$ mm⁻¹
$β = 93.859$ (1)°	$T = 293$ K
$V = 2453.01$ (11) Å³	Block, colourless
$Z = 8$	0.20 × 0.20 × 0.15 × 0.15 (radius) mm

Data collection

Nonius Kappa CCD area-detector diffractometer	Detector resolution: 3 pixels mm⁻¹
Radiation source: fine-focus sealed tube	*ω* scans
Graphite monochromator	

Absorption correction: for a sphere Interpolation using Int. Tables Vol. C (1992) p. 523, Table 6.3.3.3, for values of muR in the range 0–2.5, and Int. Tables Vol.II (1959) p. 302, Table 5.3.6 B, for muR in the range 2.6–10.0. The interpolation procedure of Dwiggins (1975) is used with some modification.
T_{min} = 0.861, T_{max} = 0.862

29355 measured reflections
5554 independent reflections
3199 reflections with $I > 2\sigma(I)$
R_{int} = 0.096
θ_{max} = 27.5°, θ_{min} = 2.1°
$h = -12\rightarrow12$
$k = -20\rightarrow20$
$l = -20\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.138
S = 1.00
5554 reflections
259 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.2886P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.021
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.82440 (7)	0.33390 (5)	−0.08409 (5)	0.0743 (3)
Si1	0.72585 (7)	0.27663 (5)	0.10559 (5)	0.0593 (2)
N1	0.62117 (19)	0.33350 (12)	0.02544 (12)	0.0516 (5)
N3	0.5713 (2)	0.40073 (13)	−0.09430 (13)	0.0567 (5)
H3	0.5788	0.4220	−0.1437	0.068*
C2	0.6702 (3)	0.35663 (16)	−0.05052 (16)	0.0540 (6)
C4	0.3314 (3)	0.44625 (17)	−0.06900 (19)	0.0649 (7)
H4	0.3138	0.4750	−0.1197	0.078*
C5	0.2354 (3)	0.44054 (19)	−0.0107 (2)	0.0740 (8)
H5	0.1501	0.4652	−0.0222	0.089*
C6	0.2631 (3)	0.3988 (2)	0.0649 (2)	0.0743 (8)
H6	0.1958	0.3966	0.1035	0.089*
C7	0.3879 (3)	0.35996 (18)	0.08536 (18)	0.0656 (7)
H7	0.4053	0.3319	0.1365	0.079*
C8	0.4853 (2)	0.36489 (15)	0.02638 (16)	0.0515 (6)
C9	0.4568 (2)	0.40705 (15)	−0.04922 (16)	0.0518 (6)
C20	0.7631 (3)	0.17130 (18)	0.06240 (19)	0.0724 (8)
H20A	0.6952	0.1317	0.0782	0.109*
H20B	0.8517	0.1529	0.0846	0.109*
H20C	0.7618	0.1745	0.0019	0.109*
C21	0.8813 (3)	0.3392 (2)	0.1347 (2)	0.0952 (11)
H21A	0.8557	0.3931	0.1566	0.143*
H21B	0.9320	0.3475	0.0856	0.143*

H21C	0.9371	0.3094	0.1770	0.143*
C22	0.6279 (4)	0.2639 (3)	0.19972 (19)	0.0971 (12)
H22A	0.6846	0.2378	0.2441	0.146*
H22B	0.5496	0.2288	0.1861	0.146*
H22C	0.5983	0.3183	0.2181	0.146*
S2	0.41465 (7)	0.49235 (5)	0.27755 (4)	0.0645 (2)
Si2	0.25642 (8)	0.35791 (5)	0.40605 (5)	0.0638 (2)
N11	0.17856 (19)	0.44585 (13)	0.34850 (13)	0.0530 (5)
N13	0.15541 (19)	0.54650 (13)	0.25194 (13)	0.0542 (5)
H13	0.1744	0.5824	0.2137	0.065*
C12	0.2474 (2)	0.49487 (15)	0.29285 (15)	0.0498 (6)
C14	−0.0965 (3)	0.57291 (19)	0.25713 (19)	0.0715 (8)
H14	−0.1033	0.6156	0.2167	0.086*
C15	−0.2088 (3)	0.5458 (2)	0.2971 (2)	0.0868 (10)
H15	−0.2932	0.5711	0.2841	0.104*
C16	−0.1981 (3)	0.4822 (3)	0.3559 (2)	0.0965 (11)
H16	−0.2762	0.4644	0.3810	0.116*
C17	−0.0746 (3)	0.4436 (2)	0.3789 (2)	0.0837 (10)
H17	−0.0689	0.4008	0.4192	0.100*
C18	0.0396 (2)	0.47021 (17)	0.34055 (16)	0.0563 (6)
C19	0.0272 (2)	0.53378 (17)	0.27982 (16)	0.0549 (6)
C23	0.2899 (5)	0.2784 (2)	0.3261 (3)	0.1273 (15)
H23A	0.3674	0.2955	0.2964	0.191*
H23B	0.2113	0.2732	0.2870	0.191*
H23C	0.3085	0.2249	0.3531	0.191*
C24	0.4101 (4)	0.3910 (3)	0.4689 (3)	0.1213 (16)
H24A	0.4233	0.3552	0.5177	0.182*
H24B	0.3999	0.4486	0.4867	0.182*
H24C	0.4878	0.3865	0.4355	0.182*
C25	0.1363 (3)	0.3166 (3)	0.4803 (2)	0.1084 (13)
H25	0.1828	0.2770	0.5181	0.163*
H25B	0.0617	0.2887	0.4494	0.163*
H25C	0.1017	0.3625	0.5122	0.163*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0615 (4)	0.0868 (6)	0.0762 (5)	0.0191 (4)	0.0171 (3)	0.0267 (4)
Si1	0.0585 (4)	0.0614 (5)	0.0576 (4)	0.0110 (3)	0.0009 (3)	0.0068 (3)
N1	0.0481 (11)	0.0519 (12)	0.0544 (12)	0.0052 (9)	0.0019 (9)	0.0042 (10)
N3	0.0563 (12)	0.0593 (13)	0.0539 (12)	0.0033 (10)	−0.0012 (10)	0.0097 (10)
C2	0.0541 (14)	0.0513 (15)	0.0561 (15)	0.0025 (11)	0.0001 (11)	0.0025 (12)
C4	0.0563 (16)	0.0571 (17)	0.0787 (19)	0.0055 (13)	−0.0155 (14)	−0.0023 (14)
C5	0.0488 (16)	0.0658 (18)	0.106 (2)	0.0088 (13)	−0.0080 (16)	−0.0126 (17)
C6	0.0494 (16)	0.081 (2)	0.094 (2)	0.0030 (14)	0.0121 (15)	−0.0082 (18)
C7	0.0565 (16)	0.0690 (18)	0.0720 (18)	0.0012 (13)	0.0105 (13)	−0.0003 (14)
C8	0.0479 (14)	0.0443 (13)	0.0614 (15)	−0.0002 (11)	−0.0029 (11)	−0.0041 (12)
C9	0.0470 (14)	0.0442 (14)	0.0632 (15)	−0.0010 (10)	−0.0046 (11)	−0.0055 (11)

C20	0.0755 (19)	0.0620 (18)	0.080 (2)	0.0176 (14)	0.0106 (15)	0.0116 (15)
C21	0.081 (2)	0.101 (3)	0.098 (2)	0.0015 (19)	−0.0279 (18)	−0.003 (2)
C22	0.103 (2)	0.123 (3)	0.068 (2)	0.044 (2)	0.0187 (17)	0.0232 (19)
S2	0.0470 (4)	0.0746 (5)	0.0727 (5)	0.0043 (3)	0.0094 (3)	0.0152 (4)
Si2	0.0542 (4)	0.0568 (5)	0.0798 (5)	−0.0029 (3)	−0.0002 (4)	0.0199 (4)
N11	0.0459 (11)	0.0509 (12)	0.0621 (12)	−0.0029 (9)	0.0030 (9)	0.0092 (10)
N13	0.0502 (12)	0.0541 (13)	0.0586 (12)	−0.0003 (10)	0.0058 (9)	0.0118 (10)
C12	0.0482 (13)	0.0468 (14)	0.0540 (14)	−0.0013 (11)	0.0011 (11)	0.0000 (11)
C14	0.0561 (17)	0.081 (2)	0.0753 (19)	0.0097 (14)	−0.0088 (14)	0.0132 (16)
C15	0.0458 (16)	0.115 (3)	0.099 (2)	0.0122 (17)	0.0009 (15)	0.012 (2)
C16	0.0466 (17)	0.135 (3)	0.109 (3)	0.0011 (18)	0.0124 (16)	0.030 (2)
C17	0.0556 (17)	0.100 (3)	0.096 (2)	−0.0059 (16)	0.0103 (15)	0.0340 (19)
C18	0.0456 (14)	0.0609 (16)	0.0620 (15)	−0.0030 (12)	0.0008 (11)	0.0045 (13)
C19	0.0461 (14)	0.0594 (16)	0.0587 (15)	−0.0018 (11)	−0.0003 (11)	0.0020 (12)
C23	0.173 (4)	0.061 (2)	0.149 (4)	0.020 (2)	0.021 (3)	0.001 (2)
C24	0.077 (2)	0.158 (4)	0.124 (3)	−0.029 (2)	−0.032 (2)	0.056 (3)
C25	0.080 (2)	0.116 (3)	0.129 (3)	−0.001 (2)	0.011 (2)	0.069 (3)

Geometric parameters (Å, º)

S1—C2	1.676 (3)	S2—C12	1.675 (2)
Si1—N1	1.817 (2)	Si2—N11	1.804 (2)
Si1—C22	1.841 (3)	Si2—C24	1.827 (3)
Si1—C20	1.846 (3)	Si2—C23	1.830 (4)
Si1—C21	1.850 (3)	Si2—C25	1.841 (3)
N1—C2	1.377 (3)	N11—C12	1.384 (3)
N1—C8	1.423 (3)	N11—C18	1.413 (3)
N3—C2	1.348 (3)	N13—C12	1.350 (3)
N3—C9	1.374 (3)	N13—C19	1.375 (3)
N3—H3	0.8600	N13—H13	0.8600
C4—C5	1.366 (4)	C14—C15	1.375 (4)
C4—C9	1.394 (3)	C14—C19	1.388 (4)
C4—H4	0.9300	C14—H14	0.9300
C5—C6	1.380 (4)	C15—C16	1.371 (5)
C5—H5	0.9300	C15—H15	0.9300
C6—C7	1.388 (4)	C16—C17	1.383 (4)
C6—H6	0.9300	C16—H16	0.9300
C7—C8	1.384 (4)	C17—C18	1.375 (4)
C7—H7	0.9300	C17—H17	0.9300
C8—C9	1.384 (3)	C18—C19	1.392 (3)
C20—H20A	0.9600	C23—H23A	0.9600
C20—H20B	0.9600	C23—H23B	0.9600
C20—H20C	0.9600	C23—H23C	0.9600
C21—H21A	0.9600	C24—H24A	0.9600
C21—H21B	0.9600	C24—H24B	0.9600
C21—H21C	0.9600	C24—H24C	0.9600
C22—H22A	0.9600	C25—H25	0.9600
C22—H22B	0.9600	C25—H25B	0.9600

C22—H22C	0.9600	C25—H25C	0.9600
N1—Si1—C22	108.72 (12)	N11—Si2—C24	111.21 (15)
N1—Si1—C20	107.62 (12)	N11—Si2—C23	105.51 (15)
C22—Si1—C20	109.24 (16)	C24—Si2—C23	113.3 (2)
N1—Si1—C21	109.12 (13)	N11—Si2—C25	109.27 (13)
C22—Si1—C21	108.81 (18)	C24—Si2—C25	106.95 (19)
C20—Si1—C21	113.23 (16)	C23—Si2—C25	110.6 (2)
C2—N1—C8	107.39 (19)	C12—N11—C18	107.40 (19)
C2—N1—Si1	122.00 (16)	C12—N11—Si2	123.12 (16)
C8—N1—Si1	130.56 (17)	C18—N11—Si2	128.88 (17)
C2—N3—C9	110.7 (2)	C12—N13—C19	110.6 (2)
C2—N3—H3	124.6	C12—N13—H13	124.7
C9—N3—H3	124.6	C19—N13—H13	124.7
N3—C2—N1	107.9 (2)	N13—C12—N11	107.9 (2)
N3—C2—S1	125.48 (19)	N13—C12—S2	125.02 (19)
N1—C2—S1	126.65 (18)	N11—C12—S2	127.12 (18)
C5—C4—C9	117.1 (3)	C15—C14—C19	117.0 (3)
C5—C4—H4	121.4	C15—C14—H14	121.5
C9—C4—H4	121.4	C19—C14—H14	121.5
C4—C5—C6	121.1 (3)	C16—C15—C14	121.0 (3)
C4—C5—H5	119.5	C16—C15—H15	119.5
C6—C5—H5	119.5	C14—C15—H15	119.5
C5—C6—C7	122.2 (3)	C15—C16—C17	121.9 (3)
C5—C6—H6	118.9	C15—C16—H16	119.1
C7—C6—H6	118.9	C17—C16—H16	119.1
C8—C7—C6	117.1 (3)	C18—C17—C16	118.3 (3)
C8—C7—H7	121.5	C18—C17—H17	120.9
C6—C7—H7	121.5	C16—C17—H17	120.9
C9—C8—C7	120.3 (2)	C17—C18—C19	119.4 (2)
C9—C8—N1	107.1 (2)	C17—C18—N11	133.3 (3)
C7—C8—N1	132.6 (2)	C19—C18—N11	107.3 (2)
N3—C9—C8	107.0 (2)	N13—C19—C14	130.8 (2)
N3—C9—C4	130.9 (3)	N13—C19—C18	106.8 (2)
C8—C9—C4	122.2 (3)	C14—C19—C18	122.4 (2)
Si1—C20—H20A	109.5	Si2—C23—H23A	109.5
Si1—C20—H20B	109.5	Si2—C23—H23B	109.5
H20A—C20—H20B	109.5	H23A—C23—H23B	109.5
Si1—C20—H20C	109.5	Si2—C23—H23C	109.5
H20A—C20—H20C	109.5	H23A—C23—H23C	109.5
H20B—C20—H20C	109.5	H23B—C23—H23C	109.5
Si1—C21—H21A	109.5	Si2—C24—H24A	109.5
Si1—C21—H21B	109.5	Si2—C24—H24B	109.5
H21A—C21—H21B	109.5	H24A—C24—H24B	109.5
Si1—C21—H21C	109.5	Si2—C24—H24C	109.5
H21A—C21—H21C	109.5	H24A—C24—H24C	109.5
H21B—C21—H21C	109.5	H24B—C24—H24C	109.5
Si1—C22—H22A	109.5	Si2—C25—H25	109.5

Si1—C22—H22B	109.5	Si2—C25—H25B	109.5
H22A—C22—H22B	109.5	H25—C25—H25B	109.5
Si1—C22—H22C	109.5	Si2—C25—H25C	109.5
H22A—C22—H22C	109.5	H25—C25—H25C	109.5
H22B—C22—H22C	109.5	H25B—C25—H25C	109.5
C22—Si1—N1—C2	176.3 (2)	C24—Si2—N11—C12	56.7 (3)
C20—Si1—N1—C2	−65.5 (2)	C23—Si2—N11—C12	−66.5 (2)
C21—Si1—N1—C2	57.8 (2)	C25—Si2—N11—C12	174.5 (2)
C22—Si1—N1—C8	−1.1 (3)	C24—Si2—N11—C18	−133.4 (3)
C20—Si1—N1—C8	117.1 (2)	C23—Si2—N11—C18	103.4 (3)
C21—Si1—N1—C8	−119.6 (2)	C25—Si2—N11—C18	−15.6 (3)
C9—N3—C2—N1	−0.4 (3)	C19—N13—C12—N11	0.8 (3)
C9—N3—C2—S1	179.14 (18)	C19—N13—C12—S2	−179.11 (18)
C8—N1—C2—N3	0.6 (3)	C18—N11—C12—N13	−0.5 (3)
Si1—N1—C2—N3	−177.32 (16)	Si2—N11—C12—N13	171.28 (16)
C8—N1—C2—S1	−178.90 (19)	C18—N11—C12—S2	179.38 (19)
Si1—N1—C2—S1	3.2 (3)	Si2—N11—C12—S2	−8.8 (3)
C9—C4—C5—C6	1.0 (4)	C19—C14—C15—C16	−0.8 (5)
C4—C5—C6—C7	−0.7 (5)	C14—C15—C16—C17	1.3 (6)
C5—C6—C7—C8	0.2 (4)	C15—C16—C17—C18	−0.5 (6)
C6—C7—C8—C9	0.0 (4)	C16—C17—C18—C19	−0.7 (5)
C6—C7—C8—N1	−180.0 (3)	C16—C17—C18—N11	179.8 (3)
C2—N1—C8—C9	−0.6 (2)	C12—N11—C18—C17	179.6 (3)
Si1—N1—C8—C9	177.06 (17)	Si2—N11—C18—C17	8.5 (5)
C2—N1—C8—C7	179.3 (3)	C12—N11—C18—C19	0.1 (3)
Si1—N1—C8—C7	−3.0 (4)	Si2—N11—C18—C19	−171.09 (18)
C2—N3—C9—C8	0.0 (3)	C12—N13—C19—C14	178.8 (3)
C2—N3—C9—C4	−180.0 (3)	C12—N13—C19—C18	−0.7 (3)
C7—C8—C9—N3	−179.6 (2)	C15—C14—C19—N13	−179.9 (3)
N1—C8—C9—N3	0.4 (2)	C15—C14—C19—C18	−0.4 (4)
C7—C8—C9—C4	0.4 (4)	C17—C18—C19—N13	−179.2 (3)
N1—C8—C9—C4	−179.7 (2)	N11—C18—C19—N13	0.4 (3)
C5—C4—C9—N3	179.1 (3)	C17—C18—C19—C14	1.2 (4)
C5—C4—C9—C8	−0.8 (4)	N11—C18—C19—C14	−179.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···S2[i]	0.86	2.52	3.374 (2)	170
N13—H13···S1[i]	0.86	2.45	3.282 (2)	164
C21—H21*B*···S1	0.96	2.83	3.480 (4)	126

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