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N-(Trimethylsilyl)pyridine-3-carboxamide

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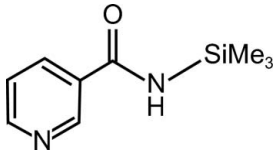
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.126; data-to-parameter ratio = 21.4.

The title compound, $C_9H_{14}N_2OSi$, crystallizes with two molecules of different conformation in the asymmetric unit. As the main intermolecular interactions, C—H···O and N—H···O hydrogen bonds connect the individual molecules into chains along [100]. While N—H donors connect to O-atom acceptors, the potential pyridine N-atom acceptors obviously are of minor importance: one of them accepts a hydrogen bond of a pyridylic C—H function, and the other one does not establish any interactions within a 'sum of the van der Waals radii plus 0.2 Å' criterion. In addition, there are π–π stacking interactions between pyridyl rings (centroid–centroid distance = 3.615 Å).

Related literature

The title compound was prepared according to a published procedure for the bis(trimethylsilyl) derivative (Franchetti *et al.*, 2004).



Experimental

Crystal data

$C_9H_{14}N_2OSi$
M_r = 194.31
Triclinic, $P\overline{1}$
a = 9.9550 (3) Å
b = 11.0928 (4) Å
c = 11.2096 (4) Å
α = 80.5113 (14)°
β = 82.6627 (13)°
γ = 67.2202 (19)°
V = 1122.91 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 200 (2) K
0.18 × 0.06 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
7682 measured reflections
5073 independent reflections
3244 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.126
S = 1.01
5073 reflections
237 parameters
Only H-atom displacement parameters refined
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H721···O2^{i}	0.88	2.10	2.948 (2)	161
N4—H741···O1	0.88	2.13	2.982 (2)	164
C12—H12···O1	0.95	2.30	3.201 (3)	157
C1—H1···O2^{i}	0.95	2.49	3.343 (3)	149
C3—H3···N3^{ii}	0.95	2.58	3.492 (4)	161

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

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

The authors thank Anna Zangl for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2620).

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supplementary materials

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N-(Trimethylsilyl)pyridine-3-carboxamide

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Comment

The title compound, silylated nicotineamide, is a building block for the synthesis of coenzymes of the NAD family. In contrast to the results presented by Franchetti *et al.* (2004), numerous variations of the reaction conditions always yielded the mono- instead of the bis-silyl derivative of nicotineamide. In order to verify Si-NMR-spectroscopic hints on monosilylation, the product was crystallized and structurally characterized.

In the crystal structure, two molecules with a different orientation of the pyridine-N atoms with respect to the amide function are stacked at right angles. The strongest intermolecular interactions are N–H···O hydrogen bonds (Fig. 1), the O acceptor thus being the stronger one compared with the potential pyridine-N acceptor. The latter acts as an acceptor in a weak C–H···N interaction, which, together with supporting π-stacking, connects two N–H···O-connected chains along [100] to double chains (Fig. 2).

Experimental

The title compound was prepared in analogy to a published procedure (Franchetti *et al.*, 2004) upon reaction of nicotineamide with chlorotrimethylsilane in boiling hexamethyldisilazane. Crystals suitable for X-ray analysis were obtained within one day after slowly cooling down the reaction mixture.

Refinement

All H atoms were located in a difference map and refined as riding on their parent atoms with $C_{aromatic}$—H = 0.95 Å, C_{methyl}—H = 0.98Å and N—H = 0.88 Å. Two common isotropic displacement parameters for the H atoms were refined: one for the methyl Hs and one for the remaining Hs.

Figures

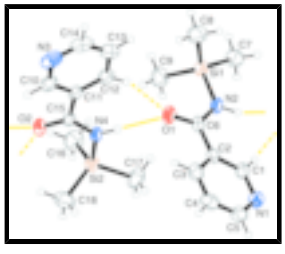

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 40% probability level. Intermolecular N–H···O hydrogen bonds are drawn as yellow bars.

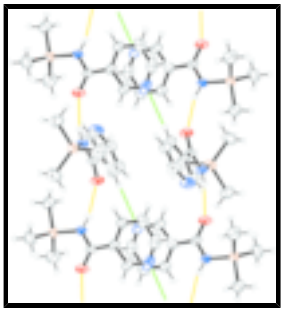

Fig. 2. Two N–H···O-connected chains. The main interactions are C–H···N bonds (green bars) and pyridyl–pyridyl π-stacking (top middle and bottom middle of the figure).

N-(Trimethylsilyl)pyridine-3-carboxamide

Crystal data

$C_9H_{14}N_2OSi$	$Z = 4$
$M_r = 194.31$	$F_{000} = 416$
Triclinic, $P\overline{1}$	$D_x = 1.149$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.9550$ (3) Å	Cell parameters from 3640 reflections
$b = 11.0928$ (4) Å	$\theta = 3.1$–27.5º
$c = 11.2096$ (4) Å	$\mu = 0.18$ mm^{-1}
$\alpha = 80.5113$ (14)º	$T = 200$ (2) K
$\beta = 82.6627$ (13)º	Needle, colourless
$\gamma = 67.2202$ (19)º	0.18 × 0.06 × 0.03 mm
$V = 1122.91$ (7) Å^3	

Data collection

KappaCCD diffractometer	3244 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	$R_{int} = 0.030$
Monochromator: MONTEL, graded multilayered X-ray optics	$\theta_{max} = 27.5$º
$T = 200$(2) K	$\theta_{min} = 3.3$º
φ/ω–scan	$h = -12 \rightarrow 12$
Absorption correction: none	$k = -14 \rightarrow 12$
7682 measured reflections	$l = -13 \rightarrow 14$
5073 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	Only H-atom displacement parameters refined
$wR(F^2) = 0.126$	$w = 1/[\sigma^2(F_o^2) + (0.0385P)^2 + 0.342P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.01	$(\Delta/\sigma)_{max} < 0.001$
5073 reflections	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
237 parameters	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Si1	0.70081 (7)	0.07672 (6)	0.13501 (6)	0.04635 (18)
Si2	0.15496 (6)	0.20555 (7)	0.41313 (6)	0.04568 (18)
O1	0.42748 (15)	0.31010 (16)	0.16768 (15)	0.0530 (4)
O2	−0.05206 (15)	0.23814 (16)	0.23110 (14)	0.0519 (4)
N1	0.6058 (2)	0.5495 (2)	0.3814 (2)	0.0594 (6)
N2	0.66665 (17)	0.22227 (17)	0.19744 (16)	0.0436 (4)
H721	0.7404	0.2323	0.2246	0.056 (2)*
N3	−0.0520 (3)	0.3866 (2)	−0.1337 (2)	0.0665 (6)
N4	0.15327 (17)	0.26542 (17)	0.25745 (15)	0.0384 (4)
H741	0.2223	0.2940	0.2244	0.056 (2)*
C1	0.6209 (2)	0.4456 (2)	0.3272 (2)	0.0500 (6)
H1	0.7110	0.3723	0.3325	0.056 (2)*
C2	0.5146 (2)	0.4372 (2)	0.26416 (19)	0.0392 (5)
C3	0.3823 (2)	0.5446 (2)	0.2575 (2)	0.0504 (6)
H3	0.3052	0.5434	0.2163	0.056 (2)*
C4	0.3658 (3)	0.6531 (2)	0.3123 (2)	0.0560 (6)
H4	0.2775	0.7285	0.3080	0.056 (2)*
C5	0.4783 (3)	0.6506 (2)	0.3727 (2)	0.0555 (6)
H5	0.4645	0.7254	0.4106	0.056 (2)*
C6	0.5334 (2)	0.3194 (2)	0.20598 (19)	0.0411 (5)
C7	0.8928 (3)	−0.0263 (3)	0.1667 (3)	0.0643 (7)
H71	0.9013	−0.0455	0.2545	0.116 (3)*
H72	0.9561	0.0212	0.1304	0.116 (3)*
H73	0.9226	−0.1090	0.1319	0.116 (3)*
C8	0.6801 (3)	0.1152 (3)	−0.0291 (3)	0.0777 (9)
H81	0.5780	0.1699	−0.0437	0.116 (3)*
H82	0.7087	0.0331	−0.0648	0.116 (3)*
H83	0.7427	0.1630	−0.0663	0.116 (3)*
C9	0.5745 (3)	−0.0029 (3)	0.2113 (3)	0.0724 (8)
H91	0.5881	−0.0228	0.2984	0.116 (3)*
H92	0.5949	−0.0848	0.1775	0.116 (3)*
H93	0.4735	0.0568	0.1983	0.116 (3)*
C10	−0.0537 (2)	0.3488 (2)	−0.0142 (2)	0.0526 (6)
H10	−0.1427	0.3475	0.0270	0.056 (2)*
C11	0.0669 (2)	0.3113 (2)	0.05355 (18)	0.0383 (5)
C12	0.1957 (2)	0.3146 (2)	−0.0080 (2)	0.0466 (5)
H12	0.2807	0.2914	0.0345	0.056 (2)*
C13	0.1991 (3)	0.3517 (3)	−0.1311 (2)	0.0589 (6)
H13	0.2869	0.3524	−0.1752	0.056 (2)*

C14	0.0738 (3)	0.3878 (2)	−0.1894 (2)	0.0640 (7)
H14	0.0770	0.4152	−0.2742	0.056 (2)*
C15	0.0515 (2)	0.2697 (2)	0.18614 (19)	0.0381 (5)
C16	0.1900 (3)	0.0272 (3)	0.4290 (3)	0.0713 (8)
H161	0.2827	−0.0184	0.3845	0.116 (3)*
H162	0.1106	0.0146	0.3958	0.116 (3)*
H163	0.1951	−0.0088	0.5149	0.116 (3)*
C17	0.3083 (3)	0.2331 (3)	0.4661 (2)	0.0678 (7)
H171	0.2875	0.3279	0.4570	0.116 (3)*
H172	0.3984	0.1893	0.4177	0.116 (3)*
H173	0.3201	0.1967	0.5516	0.116 (3)*
C18	−0.0187 (3)	0.2966 (3)	0.4963 (2)	0.0765 (9)
H181	−0.0342	0.3904	0.4864	0.116 (3)*
H182	−0.0141	0.2620	0.5826	0.116 (3)*
H183	−0.0997	0.2857	0.4640	0.116 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0443 (3)	0.0472 (4)	0.0531 (4)	−0.0208 (3)	−0.0041 (3)	−0.0118 (3)
Si2	0.0445 (3)	0.0535 (4)	0.0421 (4)	−0.0217 (3)	−0.0064 (3)	−0.0035 (3)
O1	0.0354 (8)	0.0616 (11)	0.0699 (11)	−0.0227 (7)	−0.0111 (7)	−0.0124 (8)
O2	0.0333 (8)	0.0691 (11)	0.0613 (10)	−0.0260 (7)	−0.0024 (7)	−0.0133 (8)
N1	0.0482 (11)	0.0602 (14)	0.0773 (15)	−0.0230 (10)	0.0032 (10)	−0.0287 (11)
N2	0.0330 (9)	0.0456 (11)	0.0584 (12)	−0.0181 (8)	−0.0070 (8)	−0.0120 (9)
N3	0.0791 (16)	0.0530 (14)	0.0612 (15)	−0.0072 (11)	−0.0350 (12)	−0.0109 (11)
N4	0.0328 (8)	0.0492 (11)	0.0400 (10)	−0.0219 (8)	−0.0061 (7)	−0.0049 (8)
C1	0.0351 (11)	0.0513 (14)	0.0667 (15)	−0.0160 (10)	−0.0003 (10)	−0.0190 (12)
C2	0.0328 (10)	0.0418 (12)	0.0448 (12)	−0.0179 (9)	0.0018 (8)	−0.0038 (9)
C3	0.0406 (12)	0.0540 (15)	0.0517 (14)	−0.0135 (10)	−0.0066 (10)	−0.0013 (11)
C4	0.0543 (14)	0.0454 (14)	0.0564 (15)	−0.0077 (11)	0.0042 (12)	−0.0081 (12)
C5	0.0574 (15)	0.0522 (15)	0.0613 (16)	−0.0245 (12)	0.0078 (12)	−0.0181 (12)
C6	0.0320 (10)	0.0475 (13)	0.0475 (13)	−0.0200 (9)	−0.0024 (9)	−0.0031 (10)
C7	0.0514 (14)	0.0542 (16)	0.0832 (19)	−0.0107 (12)	−0.0054 (13)	−0.0195 (14)
C8	0.082 (2)	0.095 (2)	0.0586 (17)	−0.0306 (17)	−0.0094 (14)	−0.0191 (16)
C9	0.0713 (17)	0.0576 (17)	0.098 (2)	−0.0358 (14)	0.0067 (15)	−0.0151 (15)
C10	0.0473 (13)	0.0516 (14)	0.0590 (16)	−0.0110 (11)	−0.0187 (11)	−0.0148 (12)
C11	0.0364 (10)	0.0363 (12)	0.0435 (12)	−0.0101 (9)	−0.0096 (9)	−0.0122 (9)
C12	0.0468 (12)	0.0511 (14)	0.0445 (13)	−0.0184 (10)	−0.0063 (10)	−0.0104 (11)
C13	0.0734 (17)	0.0567 (16)	0.0459 (14)	−0.0233 (13)	0.0030 (12)	−0.0121 (12)
C14	0.097 (2)	0.0452 (15)	0.0425 (14)	−0.0141 (14)	−0.0152 (14)	−0.0091 (11)
C15	0.0285 (10)	0.0399 (12)	0.0480 (12)	−0.0114 (8)	−0.0065 (8)	−0.0120 (9)
C16	0.0840 (19)	0.0589 (18)	0.0745 (19)	−0.0346 (15)	−0.0060 (15)	0.0026 (14)
C17	0.0763 (18)	0.088 (2)	0.0530 (15)	−0.0437 (16)	−0.0251 (13)	0.0016 (14)
C18	0.0653 (17)	0.101 (2)	0.0498 (16)	−0.0157 (16)	0.0027 (13)	−0.0176 (16)

Geometric parameters (Å, °)

Si1—N2	1.7604 (18)	C7—H71	0.9800

Si1—C8	1.838 (3)	C7—H72	0.9800
Si1—C9	1.852 (3)	C7—H73	0.9800
Si1—C7	1.853 (2)	C8—H81	0.9800
Si2—N4	1.7617 (18)	C8—H82	0.9800
Si2—C18	1.851 (3)	C8—H83	0.9800
Si2—C17	1.852 (2)	C9—H91	0.9800
Si2—C16	1.852 (3)	C9—H92	0.9800
O1—C6	1.234 (2)	C9—H93	0.9800
O2—C15	1.237 (2)	C10—C11	1.390 (3)
N1—C5	1.331 (3)	C10—H10	0.9500
N1—C1	1.339 (3)	C11—C12	1.387 (3)
N2—C6	1.351 (3)	C11—C15	1.489 (3)
N2—H721	0.8800	C12—C13	1.374 (3)
N3—C14	1.330 (4)	C12—H12	0.9500
N3—C10	1.336 (3)	C13—C14	1.370 (4)
N4—C15	1.350 (2)	C13—H13	0.9500
N4—H741	0.8800	C14—H14	0.9500
C1—C2	1.384 (3)	C16—H161	0.9800
C1—H1	0.9500	C16—H162	0.9800
C2—C3	1.392 (3)	C16—H163	0.9800
C2—C6	1.493 (3)	C17—H171	0.9800
C3—C4	1.385 (3)	C17—H172	0.9800
C3—H3	0.9500	C17—H173	0.9800
C4—C5	1.369 (3)	C18—H181	0.9800
C4—H4	0.9500	C18—H182	0.9800
C5—H5	0.9500	C18—H183	0.9800
N2—Si1—C8	109.72 (12)	H81—C8—H82	109.5
N2—Si1—C9	109.61 (11)	Si1—C8—H83	109.5
C8—Si1—C9	111.13 (14)	H81—C8—H83	109.5
N2—Si1—C7	103.89 (10)	H82—C8—H83	109.5
C8—Si1—C7	110.88 (13)	Si1—C9—H91	109.5
C9—Si1—C7	111.36 (14)	Si1—C9—H92	109.5
N4—Si2—C18	111.29 (11)	H91—C9—H92	109.5
N4—Si2—C17	104.79 (10)	Si1—C9—H93	109.5
C18—Si2—C17	110.26 (14)	H91—C9—H93	109.5
N4—Si2—C16	108.14 (11)	H92—C9—H93	109.5
C18—Si2—C16	111.33 (14)	N3—C10—C11	124.0 (2)
C17—Si2—C16	110.82 (13)	N3—C10—H10	118.0
C5—N1—C1	116.3 (2)	C11—C10—H10	118.0
C6—N2—Si1	123.55 (14)	C12—C11—C10	117.2 (2)
C6—N2—H721	118.2	C12—C11—C15	123.86 (18)
Si1—N2—H721	118.2	C10—C11—C15	119.0 (2)
C14—N3—C10	116.9 (2)	C13—C12—C11	119.2 (2)
C15—N4—Si2	123.40 (14)	C13—C12—H12	120.4
C15—N4—H741	118.3	C11—C12—H12	120.4
Si2—N4—H741	118.3	C14—C13—C12	119.0 (2)
N1—C1—C2	124.7 (2)	C14—C13—H13	120.5
N1—C1—H1	117.6	C12—C13—H13	120.5
C2—C1—H1	117.6	N3—C14—C13	123.6 (2)

C1—C2—C3	117.4 (2)	N3—C14—H14	118.2
C1—C2—C6	123.68 (19)	C13—C14—H14	118.2
C3—C2—C6	118.94 (19)	O2—C15—N4	119.9 (2)
C4—C3—C2	118.4 (2)	O2—C15—C11	120.82 (17)
C4—C3—H3	120.8	N4—C15—C11	119.24 (17)
C2—C3—H3	120.8	Si2—C16—H161	109.5
C5—C4—C3	119.2 (2)	Si2—C16—H162	109.5
C5—C4—H4	120.4	H161—C16—H162	109.5
C3—C4—H4	120.4	Si2—C16—H163	109.5
N1—C5—C4	123.9 (2)	H161—C16—H163	109.5
N1—C5—H5	118.0	H162—C16—H163	109.5
C4—C5—H5	118.0	Si2—C17—H171	109.5
O1—C6—N2	120.3 (2)	Si2—C17—H172	109.5
O1—C6—C2	120.15 (18)	H171—C17—H172	109.5
N2—C6—C2	119.52 (17)	Si2—C17—H173	109.5
Si1—C7—H71	109.5	H171—C17—H173	109.5
Si1—C7—H72	109.5	H172—C17—H173	109.5
H71—C7—H72	109.5	Si2—C18—H181	109.5
Si1—C7—H73	109.5	Si2—C18—H182	109.5
H71—C7—H73	109.5	H181—C18—H182	109.5
H72—C7—H73	109.5	Si2—C18—H183	109.5
Si1—C8—H81	109.5	H181—C18—H183	109.5
Si1—C8—H82	109.5	H182—C18—H183	109.5
C8—Si1—N2—C6	68.8 (2)	C3—C2—C6—O1	−10.0 (3)
C9—Si1—N2—C6	−53.5 (2)	C1—C2—C6—N2	−10.2 (3)
C7—Si1—N2—C6	−172.58 (18)	C3—C2—C6—N2	171.14 (19)
C18—Si2—N4—C15	−57.1 (2)	C14—N3—C10—C11	−0.1 (4)
C17—Si2—N4—C15	−176.31 (17)	N3—C10—C11—C12	0.4 (3)
C16—Si2—N4—C15	65.44 (19)	N3—C10—C11—C15	−179.3 (2)
C5—N1—C1—C2	0.1 (4)	C10—C11—C12—C13	−1.1 (3)
N1—C1—C2—C3	−0.5 (4)	C15—C11—C12—C13	178.5 (2)
N1—C1—C2—C6	−179.2 (2)	C11—C12—C13—C14	1.6 (3)
C1—C2—C3—C4	0.9 (3)	C10—N3—C14—C13	0.6 (4)
C6—C2—C3—C4	179.7 (2)	C12—C13—C14—N3	−1.4 (4)
C2—C3—C4—C5	−1.1 (3)	Si2—N4—C15—O2	4.5 (3)
C1—N1—C5—C4	−0.3 (4)	Si2—N4—C15—C11	−174.82 (14)
C3—C4—C5—N1	0.9 (4)	C12—C11—C15—O2	−162.3 (2)
Si1—N2—C6—O1	−0.9 (3)	C10—C11—C15—O2	17.3 (3)
Si1—N2—C6—C2	177.98 (15)	C12—C11—C15—N4	17.0 (3)
C1—C2—C6—O1	168.7 (2)	C10—C11—C15—N4	−163.37 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H721···O2^{i}	0.88	2.10	2.948 (2)	161
N4—H741···O1	0.88	2.13	2.982 (2)	164
C12—H12···O1	0.95	2.30	3.201 (3)	157
C1—H1···O2^{i}	0.95	2.49	3.343 (3)	149

C3—H3···N3^{ii}	0.95	2.58	3.492 (4)	161

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*, −*y*+1, −*z*.

Fig. 1

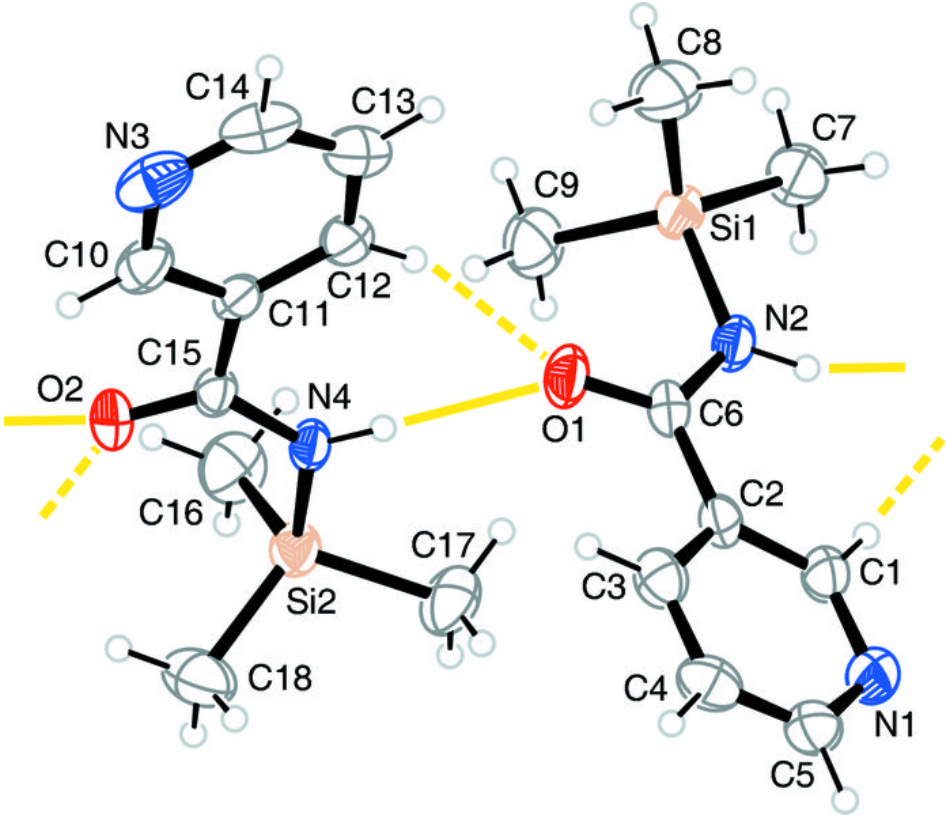

Fig. 2