

Reaction of Silicon Atoms with Hydrogen Cyanide: Generation and Matrix-Spectroscopic Identification of CHNSi and CNSi Isomers

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Cocondensation of thermally generated silicon atoms with hydrogen cyanide in an argon matrix can be used as an access to compounds of the composition CHNSi and CNSi. Isolation, matrix-spectroscopic identification and photo-

chemical behavior of these species are described. Structural assignments are made by the comparison of experimental with calculated IR spectra.

Evaporation of metal atoms and their subsequent condensation, possibly after reaction with another substrate, is the basic step in chemical vapor deposition (CVD) processes, which today are in manifold use to generate thin metallic or organic films. If the metal used is silicon, this method is essential in microelectronics industry and thus of economical importance, for the condensation of silicon vapor – alone and after reaction with substrate gases – is used for the manufacture of silicon wafers, silicon-based semiconductors, and dielectrics. A predictive understanding of the complex chemical processes involved herein requires detailed knowledge of the scope and fundamental reactions of silicon atoms.

The first work in this field goes back to Gaspar et al., who studied the reactions of recoil silicon atoms produced by nuclear transformations with silane, phosphane, and ethylene^[1]. A few years later, Skell and Owen found that silanes react with silicon atoms, which were produced by electron bombardment heating of a silicon electrode or resistance heating of a silicon rod, by successive insertion into Si–H bonds^[2].

The combination of silicon evaporation with matrix isolation techniques made possible the direct study of reactive intermediates emerging from these reactions. In 1977, Weltner et al. obtained SiNN and SiCO after cocondensation of thermally vaporized silicon with argon/N₂ and argon/CO mixtures^[3]. Margrave et al. used small hydrogen-containing molecules as reactants for silicon atoms. They observed hydroxysilylene^[4], fluorosilylene^[5], and the parent silylene^[6] after codeposition of the silicon atoms together with water, hydrogen fluoride, and hydrogen, respectively.

We now report on the cocondensation of thermally generated silicon atoms with hydrogen cyanide in an argon matrix, which led to the observation of a total of four CHNSi isomers and two CNSi isomers.

Experimental Procedure

Silicon is vaporized from a boron nitride crucible, which is surrounded by an aluminum oxide tube. The oven is resistively heated to temperatures of 1490–1550°C by means of a tungsten wire wound around the alumina tube. The silicon atoms are codeposited with gaseous mixtures of the substrate and argon onto a CsI window at 12 K. The substrate/argon ratio is usually in the range of 1:100. If cocondensed with argon alone, the high reactivity of the silicon atoms can be seen in the formation of many products, such as SiNN, SiCO, SiO, SiH₂, SiH₄, Si₂H₂^[7], and Si₂H₆^[8]. The formation of the silicon hydrides is most likely due to hydrogen impurities in the metal surfaces of the cryostat, whereas the other observable products may result from reactions of the silicon atoms with residual gases in the apparatus.

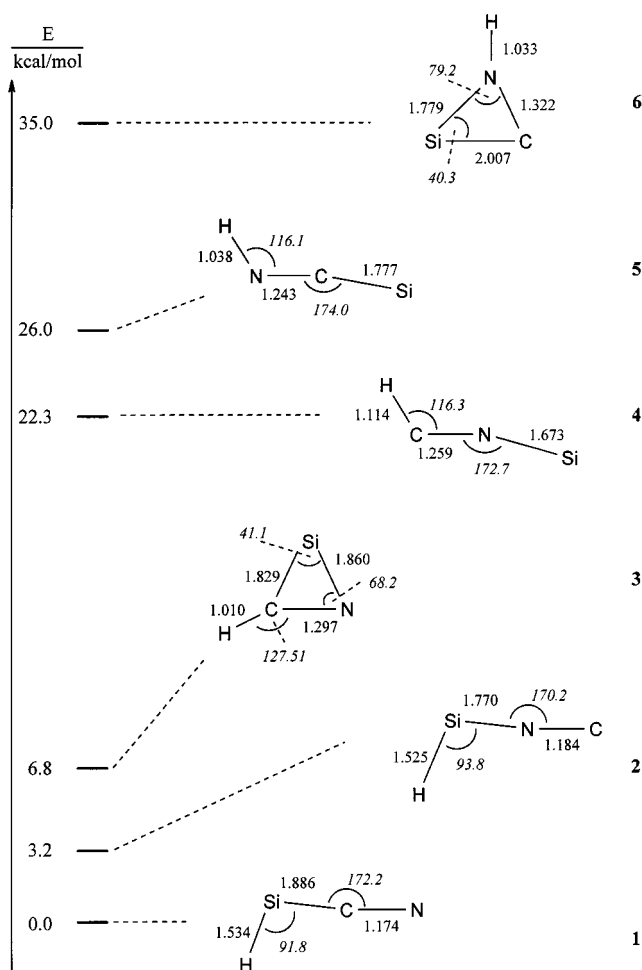
Results and Discussion

Calculations

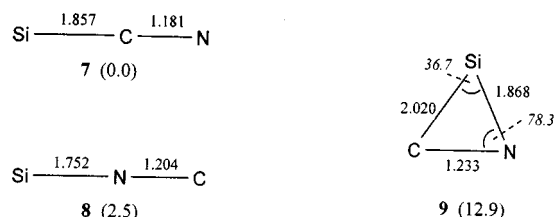
Scheme 1 shows the relative energies of several CHNSi singlet species as calculated with the BLYP functional at the 6-311G** basis^[9]. In this system, the global minimum is not the silacyclopropenylidene system, as it is the case in many other related systems^[10]. Cyanosilylene (**1**) and isocyanosilylene (**2**) are of lower energy. Obviously, the CN group has a great stabilizing effect on an adjacent silylenic centre. Nevertheless, this stabilization does not cause much increase of the bonding angle at silicon, which according to the BLYP calculations is 92° for **1** and 94° for **2**.

[\diamond] Part 26: G Maier, H. P. Reisenauer, J. Jung, H. Pacl, H. Egenolf, *Eur. J. Org. Chem.* **1998**, 1297–1305, preceding paper.

Scheme 1. Calculated (BLYP/6-311G**) relative energies [kcal mol⁻¹] (corrected by zero point vibrational energies) of six CHNSi singlet species

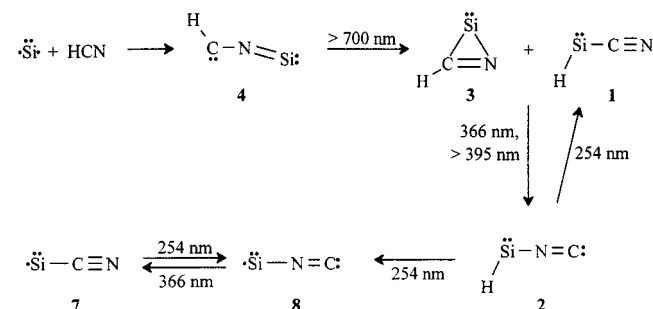


Scheme 2. Calculated (BLYP/6-311G**) relative energies [kcal mol⁻¹] (corrected by zero point vibrational energies) of three CNSi isomers. The products of a homolytic dissociation of **1** into **7** and a hydrogen atom lie 69.3 kcal mol⁻¹ above **1**



Spectroscopic Observations

Apart from the byproducts listed above (SiN_2 etc.) and HCN, the IR spectrum registered after cocondensation of silicon atoms and hydrogen cyanide shows various new absorptions at 2754, 1538, 977, and 735 cm⁻¹. They fit to the calculated spectrum of (silaisocyano)carbene (**4**)^[11]. This means, the lone pair of the nitrogen atom is more basic than the π system. This is analogous to the reaction of atomic silicon with nitrogen, leading to formation of linear SiNN ^[3], instead of the cyclic diazasilacyclopropenylidene^[12]. The lowering of the frequency of the N–N stretching vibration in SiNN by about 600 cm⁻¹ with respect to the N_2 molecule indicates a relatively weak N–N bond. The same holds for **4**, for which the band of the C–N stretching



Scheme 2 shows the relative energies of the three CNSi isomers, which can be expected from the dehydrogenation of the compounds shown in Scheme 1.

Table 1. Calculated (BLYP/6-311G**, C_s symmetry, absolute intensities [km mol⁻¹] in parentheses), corrected [$\tilde{\nu}_{\text{corr}} = \tilde{\nu}_{\text{calc}} \cdot \tilde{\nu}_{\text{obs}}(^1\text{H}^{12}\text{C-4}) / \tilde{\nu}_{\text{calc}}(^1\text{H}^{12}\text{C-4})$], and experimental (Ar matrix, 12 K, intensities relative to the strongest band in parentheses) IR absorptions [cm⁻¹] of (silaisocyano)carbene (**4**)

	mode	label	$\tilde{\nu}_{\text{calc.}}$ (4 -singlet)	$\tilde{\nu}_{\text{corr.}}$	$\tilde{\nu}_{\text{obs.}}$	$\tilde{\nu}_{\text{calc.}}$ (4 -triplet)
ν_6	a''	SiNC def oop	222.8 (2)		—	310.3 (11)
		D_{13}C	218.5 (<1)			
			220.6 (2)			
ν_5	a'	SiNC def ip	355.3 (24)		—	291.5 (20)
		D_{13}C	332.3 (25)			
			352.9 (23)			
ν_4	a'	SiN str/CH def	708.3 (20)	734.8	734.8 (2)	521.8 (251)
		D_{13}C	649.2 (69)	698.7	665.3 (11)	
			699.9 (17)	726.1	730.3 (12)	
ν_3	a'	CH def	976.8 (456)	977.0	977.0 (100)	675.7 (124)
		D_{13}C	836.3 (195)	836.5	854.7 (100)	
			964.3 (458)	964.5	964.8 (100)	
ν_2	a'	CN str	1568.6 (83)	1538.1	1538.1 (15)	1660.3 (412)
		D_{13}C	1528.0 (160)	1498.3	1485.9 (63)	
			1549.1 (72)	1519.0	1519.6 (16)	
ν_1	a'	CH str	2821.8 (127)	2753.5	2753.5 (20)	3157.6 (27)
		D_{13}C	2084.9 (46)	2034.4	2059.0 (5)	
			2811.9 (127)	2743.8	2745.3 (22)	

vibration (1538 cm^{-1} , see Table 1) is lowered by about 560 cm^{-1} compared with that of hydrogen cyanide. It even lies below values observed for compounds with C–N double bonds (e. g., 1641 cm^{-1} for methanimine, $\text{H}_2\text{C}=\text{NH}^{[13]}$). The value of the C–H stretching vibration frequency also is very low (2754 cm^{-1}), indicative of a rather unusual structure of **4**. According to our BLYP calculations, the HCN angle in **4** is 116° . Thus, **4** can be assumed to have partly carbene character (resonance structure **4** is more important than other ones).

At the applied level of theory, the triplet form of **4** is 3.7 kcal/mol lower in energy than the singlet form, but this order is reversed when using more sophisticated methods. CAS (4,6)/6-31G** calculations predict the singlet to be 5.9 kcal/mol more stable than the triplet. The experimental observations provide unequivocal evidence that only the singlet species is formed (Table 1), as is the case in other reactions of silicon atoms investigated^[14]. The calculated IR spectrum of the triplet is not compatible with the observed bands of **4**.

The photochemical behavior of **4** is unusual. With light of wavelengths $>700\text{ nm}$, **4** can be quickly transformed into 2-aza-1-silacyclopentenylidene (**3**, Table 2). A rough estimation of the excitation energy to the lowest electronically excited state by means of a CIS/6-31 G** calculation predicts a weak absorption maximum for **4** at 1006 nm representing the HOMO-LUMO transition. The HOMO of **4** is not the $3s$ Si orbital, as it is usually the case for silylenes, but represents a lone pair at the C atom, typical for a singlet carbene.

Table 2. Calculated (BLYP/6-311G**, C_s symmetry, absolute intensities $\sim [\text{km mol}^{-1}]$ in parentheses), corrected [$v_{\text{corr}} = v_{\text{calc}} \cdot v_{\text{obs}}(^1\text{H}^{12}\text{C-3}) / v_{\text{calc}}(^1\text{H}^{12}\text{C-3})$], and experimental (Ar matrix, 12 K , intensities relative to the strongest band in parentheses) IR absorptions [cm^{-1}] of singlet azasilacyclopentenylidene (**3**)

mode	label	$\tilde{\nu}_{\text{calc.}}$	$\tilde{\nu}_{\text{corr.}}$	$\tilde{\nu}_{\text{obs.}}$
v_5 a' ring def		586.6 (32)	635.5	635.5 (96)
	D	553.8 (33)	599.7	591.6 (100)
	^{13}C	584.5 (32)	632.9	633.2 (100)
v_4 a' SiC str		735.2 (28)	769.8	769.8 (100)
	D	679.1 (28)	711.1	718.3 (48)
	^{13}C	723.2 (26)	757.2	757.9 (92)
v_6 a'' CH def oop		799.8 (16)	820.6	820.6 (95)
	D	625.4 (7)	641.7	634.1 (2)
	^{13}C	793.2 (16)	813.8	814.2 (73)
v_3 a' CH def ip		1104.7 (35)	1124.7	1124.7 (94)
	D	919.3 (6)	935.9	944.0 (10)
	^{13}C	1094.4 (37)	1114.2	1114.4 (99)
v_2 a' CN str		1465.3 (9)	1466.2	1466.2 (22)
	D	1443.7 (10)	1444.6	1452.2 (13)
	^{13}C	1436.8 (8)	1437.7	1439.0 (26)
v_1 a' CH str		2977.7 (40)	2974.2	2974.2 (28)
	D	2208.5 (17)	2205.9	—
	^{13}C	2967.9 (40)	2964.4	2962.8 (43)

Upon the irradiation mentioned above also a small amount of a second photoproduct is formed. By comparison with its calculated IR spectrum it can be identified as cyanosilylene (**1**, Table 3). Upon irradiation with light of wavelengths 366 or $>395\text{ nm}$, the bands of **3** and **1** disap-

Table 3. Calculated (BLYP/6-311G**, C_s symmetry, absolute intensities $\sim [\text{km mol}^{-1}]$ in parentheses), corrected [$v_{\text{corr}} = v_{\text{calc}} \cdot v_{\text{obs}}(^1\text{H}^{12}\text{C-1}) / v_{\text{calc}}(^1\text{H}^{12}\text{C-1})$], and experimental (Ar matrix, 12 K , intensities relative to the strongest band in parentheses) IR absorptions [cm^{-1}] of singlet cyanosilylene (**1**)

mode	label	$\tilde{\nu}_{\text{calc.}}$	$\tilde{\nu}_{\text{corr.}}$	$\tilde{\nu}_{\text{obs.}}$
v_6 a'' SiCN def oop		205.7 (4)	—	—
	D	203.9 (4)	—	—
	^{13}C	200.0 (4)	—	—
v_1 a' SiH def		264.8 (1)	—	—
	D	250.9 (2)	—	—
	^{13}C	258.5 (1)	—	—
v_2 a' SiC str		538.2 (56)	563.5	563.5
	D	523.9 (33)	548.5	547.3 (10)
	^{13}C	533.4 (55)	558.5	555.1 (12)
v_3 a' SiH def/SiCN def ip		811.2 (79)	826.4	826.4 (87)
	D	635.0 (76)	646.9	642.1 (46)
	^{13}C	807.6 (77)	822.7	823.3 (39)
v_4 a' SiH str		1987.2 (197)	2026.7	2026.7 (100)
	D	1429.8 (107)	1458.2	1470.0 (100)
	^{13}C	1987.3 (197)	2026.8	2024.5 (100)
v_5 a' CN str		2131.0 (38)	2142.7	2142.7 (74)
	D	2131.0 (39)	2142.7	2143.2 (34)
	^{13}C	2083.6 (34)	2095.0	2095.7 (17)

Table 4. Calculated (BLYP/6-311G**, C_s symmetry, absolute intensities $\sim [\text{km mol}^{-1}]$ in parentheses), corrected [$v_{\text{corr}} = v_{\text{calc}} \cdot v_{\text{obs}}(^1\text{H}^{12}\text{C-2}) / v_{\text{calc}}(^1\text{H}^{12}\text{C-2})$], and experimental (Ar matrix, 12 K , intensities relative to the strongest band in parentheses) IR absorptions [cm^{-1}] of singlet isocyanosilylene (**2**)

mode	label	$\tilde{\nu}_{\text{calc.}}$	$\tilde{\nu}_{\text{corr.}}$	$\tilde{\nu}_{\text{obs.}}$
v_6 a'' SiNC def oop		157.4 (1)	—	—
	D	154.4 (1)	—	—
	^{13}C	155.8 (1)	—	—
v_5 a' SiNC def ip		214.7 (<1)	—	—
	D	208.3 (<1)	—	—
	^{13}C	212.3 (<1)	—	—
v_4 a' SiN str		601.3 (86)	622.0	622.0 (41)
	D	583.2 (32)	603.3	607.4 (13)
	^{13}C	594.5 (83)	615.0	615.5 (31)
v_3 a' SiH def		842.3 (98)	867.9	867.9 (30)
	D	647.5 (119)	667.2	662.6 (35)
	^{13}C	842.2 (98)	867.8	867.8 (25)
v_2 a' SiH str		1960.4 (296)	2017.8	2017.8 (100)
	D	1412.2 (144)	1453.5	1470.1 (24)
	$^{13}\text{C}^{[a]}$	1967.2 (186)	2024.8	2024.6 (47)
v_1 a' CN str		1998.1 (244)	2039.5	2039.5 (77)
	D	1996.6 (277)	2038.0	2035.9 (100)
	$^{13}\text{C}^{[a]}$	1952.4 (353)	1992.9	1994.1 (100)

^[a] Better described as $\text{SiH}_{\text{str}} + \text{CN}_{\text{str}}$.

pear, whereas four small absorptions already present grow rapidly. These can be assigned to isocyanosilylene (**2**, Table 4). An independent proof for structure **2** stems from the observation that the same compound is formed on irradiation of triazidomethylsilane **10**^[15] in an argon matrix. Triazidosilane **10** loses four molecules of nitrogen upon matrix irradiation with $\lambda = 254\text{ nm}$. The reaction sequence initiated by photoexcitation is relatively complex. The main product after longer irradiation is silylisocyanide (**13**; $\nu_{\text{N}=\text{C}} = 2098\text{ cm}^{-1}$), which exists in a photoequilibrium with the already known^[16] silylcyanide (**14**; $\nu_{\text{C}=\text{N}} = 2200\text{ cm}^{-1}$). It is hard to believe that **13** can be a photoproduct

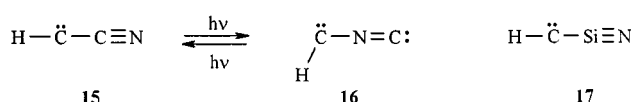


Table 5. Calculated (BLYP/6-311G**, $C_{\infty v}$ symmetry, absolute intensities [km mol^{-1}] in parentheses), corrected [$\nu_{\text{corr}} = \nu_{\text{calc}} \cdot \nu_{\text{obs}}(^1\text{H}^{12}\text{C-8}) / \nu_{\text{calc}}(^1\text{H}^{12}\text{C-8})$], and experimental (Ar matrix, 12 K, intensities relative to the strongest band in parentheses) IR absorptions [cm^{-1}] of the SiNC radical (**8**)

	mode	label	$\tilde{\nu}_{\text{calc.}}$	$\tilde{\nu}_{\text{corr.}}$	$\tilde{\nu}_{\text{obs.}}$
ν_4	π	SiNC def ^[a]	155.3 (1)		—
		^{13}C	153.5 (1)		
ν_3	π	SiNC def ^[a]	247.1 (<1)		—
		^{13}C	244.4 (<1)		
ν_2	σ	SiN str	616.5 (58)	629.1	629.1 (54)
		^{13}C	609.8 (56)	622.3	622.9 (47)
ν_1	σ	C–N str	1878.4 (112)	1945.2	1945.2 (100)
		^{13}C	1841.0 (113)	1906.5	1907.3 (100)

[a] For species with a 2π ground state the degeneracy of the bending vibration is lost (Renner-Teller effect).

Table 6. Calculated (BLYP/6-311G**, $C_{\infty v}$ symmetry, absolute intensities [km mol^{-1}] in parentheses), corrected [$\nu_{\text{corr}} = \nu_{\text{calc}} \cdot \nu_{\text{obs}}(^1\text{H}^{12}\text{C-7}) / \nu_{\text{calc}}(^1\text{H}^{12}\text{C-7})$], and experimental (Ar matrix, 12 K, intensities relative to the strongest band in parentheses) IR absorptions [cm^{-1}] of the SiCN radical (**7**)

	mode	label	$\tilde{\nu}_{\text{calc.}}$	$\tilde{\nu}_{\text{corr.}}$	$\tilde{\nu}_{\text{obs.}}$
ν_4	π	SiCN def ^[a]	197.0 (5)		—
		^{13}C	191.4 (5)		
ν_3	π	SiCN def ^[a]	285.0 (1)		—
		^{13}C	276.9 (1)		
ν_2	σ	SiC str	552.4 (54) —	584.6/548.1	(100) ^[b]
		^{13}C	547.6 (53) —	571.8	(100)
ν_1	σ	C–N str	2046.1 (15)	2077.3	(40)
		^{13}C	2001.0 (13)	2031.5	2032.2 (28)

[a] For species with a 2π ground state the degeneracy of the bending vibration is lost (Renner-Teller effect). — [b] Probably split by anharmonic resonance.

Conclusion

Cocondensation of silicon atoms with hydrogen cyanide in an argon matrix leads to the formation of (silaisocyno)carbene (**4**). By irradiation of this compound with visible or UV light three isomeric silylenes [cyanosilylene (**1**), isocyanosilylene (**2**) and azasilacyclopropenylidene (**3**)] as well as the CNSi radical **8** can be obtained. In all cases the structural assignment is based on the comparison between the experimental and calculated IR spectra of the parent compounds including the corresponding D and ^{13}C isotopomers.

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Experimental Section

Equipment Used for Matrix Isolation, Photolyses, and Pyrolyses: See Ref.^[20].

Pulsed Flash Pyrolyses and High Vacuum Flash Pyrolyses: For a description see Ref.^[20].

General: See Ref.^[20].

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