N-(*tert*-Butyldimethylsilyl)imidazole and related heterocycles: ^{13}C nuclear magnetic resonance study and reaction with dimethylsulfoxide

ALEXANDER F. JANZEN AND GERALD N. LYPKA
Department of Chemistry, University of Manitoba, Winnipeg, Man., Canada R3T 2N2
AND
RODERICK E. WASYLISHEN
Department of Chemistry, University of Winnipeg, Winnipeg, Man., Canada R3B 2E9

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N-*tert*-Butyldimethylsilyl derivatives of imidazole, 2-methylimidazole, 4-methylimidazole, benzimidazole, pyrazole, 1,2,4-triazole, and benzotriazole were prepared from *tert*-butyldimethylsilyl chloride and the corresponding heterocyclic compound. The products were identified by carbon and proton nmr, mass spectrometry, and elemental analysis. The carbon nmr spectra confirmed the absence of intermolecular silyl exchange at ambient temperature. Silyl exchange did occur at elevated temperatures, 130–160°C.

Reaction of *N*-*tert*-butyldimethylsilyl or *N*-trimethylsilyl heterocycles with dimethylsulfoxide gave *N*-(methylthio)methyl derivatives of imidazole, 2-methylimidazole, 4-methylimidazole, benzimidazole, pyrazole, and 1,2,4-triazole. The products were characterized by carbon and proton nmr, mass spectrometry, and elemental analysis. A mechanism involving a Pummerer rearrangement is proposed to account for the results.

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On a préparé des dérivés *N*-*tert*-butyldiméthylsilyles de l'imidazole, de la benzimidazole, des Me-2 et Me-4 imidazoles, du pyrazole, du triazole-1,2,4 et du benzotriazole, à partir du chlorure de *tert*-butyldiméthylsilyle et des composés hétérocycliques correspondants. On a identifié les produits par rmn du ^{1}H et du ^{13}C, par spectrométrie de masse et par analyse élémentaire. Les spectres rmn du ^{13}C confirment l'absence d'un échange intermoléculaire du groupe silyle à température ambiante. L'échange se produit à température élevée, 130–160°C. Les hétérocycles ayant des substituants *tert*-butyldiméthylsilyle ou triméthylsilyle sur l'azote réagissent avec le diméthylsulfoxyde pour donner des dérivés *N*-(méthylthio) méthyle de l'imidazole, des méthyl-2 et méthyl-4 imidazoles, du benzimidazole, du pyrazole et du triazole-1,2,4. On a caractérisé les produits par rmn du ^{1}H et du ^{13}C, par spectrométrie de masse et par analyse élémentaire. Tenant compte de ces résultats, on propose un mécanisme réactionnel qui implique une transposition de Pummerer.

[Traduit par le journal]

Silyl exchange in *N*-(trimethylsilyl)pyrazoles occurs with activation energies of 24–32 kcal/mol, except in dilute solutions of diphenyl ether where inordinately low (3–6 kcal/mol) activation energies were observed (1). The low energy pathway of intermolecular silyl exchange in *N*-(trimethylsilyl)imidazole (~7.7 kcal/mol) and *N*-(trimethylsilyl)-2-methylimidazole was attributed to rapid hydrolysis; silyl exchange was slowed down in two ways: firstly, by re-distillation of products under anhydrous conditions and, secondly, by addition of a large excess of triethylamine which presumably inhibited further exchange reactions at silicon (2).

Since bulky substituents are known to inhibit reaction at silicon (3), it seemed reasonable that introduction of the *tert*-butyl group would reduce the problem of catalyzed silyl exchange in *N*-trialkylsilyl heterocycles. Consequently, a variety of *tert*-butyldimethylsilyl derivatives of imidazole, 2- and 4-methylimidazole, benzimidazole, pyrazole, 1,2,4-triazole, and benzotriazole were prepared and investigated by means of ^{13}C and ^{1}H nmr.

During the course of this work, it was observed that *N*-trialkylsilyl heterocycles interacted with dimethylsulfoxide to give *N*-(methylthio)methyl derivatives. These derivatives were presumably formed via a Pummerer rearrangement.

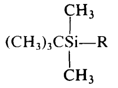
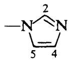
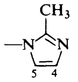
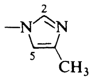
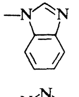
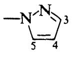
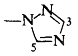
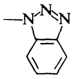
Experimental

Natural abundance ^{13}C nmr spectra were measured at 20 MHz using a Varian CFT-20 Fourier Transform nmr spectrometer. Gated proton decoupling was used in order to retain nuclear Overhauser enhancements of the proton coupled spectra. Typically, flip angles of 40 to 60° were used with acquisition time > 1 s and a pulse delay of 2–5 s. Proton nmr spectra were obtained on a Varian A-56/60A spectrometer. Mass spectra were obtained on a Finnigan 1015 quadrupole mass spectrometer. Elemental analyses were performed by Micro-Tech Laboratories, Skokie, Illinois.

Imidazole (Aldrich), 2-methylimidazole (Sigma), 4-methylimidazole (ROC/RIC), benzimidazole (Aldrich), pyrazole (Eastman), 1,2,4-triazole (Aldrich), benzotriazole (Aldrich) were used without further purification. Me_2SiCl_2 (PCR) and $(Me_3Si)_2NH$ (MCB) were redistilled prior to use. Methylene chloride, triethylamine, cyclohexane, and dimethylsulfoxide were dried, redistilled, and stored over molecular sieve. t-$BuMe_2SiCl$ was prepared from t-BuLi and Me_2SiCl_2 (3) and 1-(trimethylsilyl)imidazole, 1-(trimethylsilyl)pyrazole, and 1-(trimethylsilyl)-1,2,4-triazole were prepared from $(Me_3Si)_2NH$ and the corresponding heterocycle (4).

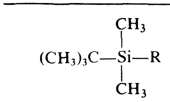
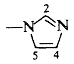
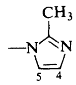
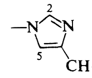
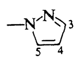
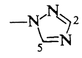
0008-4042/80/010060-05$01.00/0

TABLE 1. ^{13}C nuclear magnetic resonance chemical shifts of some N-(*tert*-butyldimethylsilyl)heterocycles[a]

 $(CH_3)_3C\overset{CH_3}{\underset{CH_3}{Si}}{-}R$	^{13}C chemical shift, ppm								
	C-2	C-4	C-5	CH_3Si	$C(CH_3)_3$	$C(CH_3)_3$	Other		
1 	140.6	130.4	120.7	−5.7	17.7	25.6			
2 	149.0	128.9	121.8	−3.8	19.0	26.2	CH_3—C-2 16.8		
3 	139.2	140.1	117.0	−5.6	17.8	25.8	CH_3—C-4 13.5		
4 	146.3			−5.0	18.7	25.9	C(ring) 113.4 122.5	120.2 137.8	121.8 146.4[b]
5 		105.9	134.0	−5.5	18.2	26.0	C-3 142.8		
6 			149.4	−5.7	17.8	25.7	C-3 154.1		
7 				−4.7	19.0	25.9	C(ring) 112.1 123.8	115.3 125.7	119.8 127.4

[a]Negative chemical shifts are upfield from internal TMS.
[b]Peaks at 137.8 and 146.4 are due to carbons without directly bonded protons.

TABLE 2. ^{13}C,H coupling constants in some N-(*tert*-butyldimethylsilyl)heterocycles

 $(CH_3)_3C{-}\overset{CH_3}{\underset{CH_3}{Si}}{-}R$	^{13}C,H coupling constant, Hz[a]
1 	$^{1}J_{(\text{C-2,H})} = 204.2$ $^{3}J_{(\text{C-2,H-4})} = 11.6$ $^{3}J_{(\text{C-2,H-5})} = 7.9$ $^{1}J_{(\text{C-4,H})} = 186.1$ $^{2}J_{(\text{C-4,H-5})} \simeq {}^{3}J_{(\text{C-4,H-2})} = 10.9$ $^{1}J_{(\text{C-5,H})} = 186.6$ $^{2}J_{(\text{C-5,H-4})} = 17.1$ $^{3}J_{(\text{C-5,H-2})} = 4.1$ $^{1}J_{(CH_3\text{—Si})} = 120.3$ $^{1}J_{\text{C}(CH_3)_3} = 125.4$
2 	$^{1}J_{(\text{C-4,H})} = 185.3$ $^{2}J_{(\text{C-4,H-5})} = 9.9$ $^{1}J_{(\text{C-5,H})} = 185.7$ $^{2}J_{(\text{C-5,H-4})} = 16.4$ $^{1}J_{(CH_3\text{—Si})} = 120.1$ $^{1}J_{(CH_3\text{—C-2})} = 127.5$ $^{1}J_{\text{C}(CH_3)_3} = 125.6$
3 	$^{1}J_{(\text{C-2,H})} = 203.2$ $^{3}J_{(\text{C-2,H-5})} = 8.1$ $^{1}J_{(\text{C-5,H})} = 184.8$ $^{3}J_{(\text{C-5,H-2})} = 4.1 \pm 1.0$ $^{1}J_{(CH_3\text{—Si})} = 120.3$ $^{1}J_{(CH_3\text{—C-4})} = 126.0$ $^{1}J_{\text{C}(CH_3)_3} = 125.4$
5 	$^{1}J_{(\text{C-3,H})} = 181.7$ $^{2}J_{(\text{C-3,H-4})} = {}^{3}J_{(\text{C-3,H-5})} = 7.3$ $^{1}J_{(\text{C-4,H})} = 174.3$ $^{2}J_{(\text{C-4,H-3})}$ or $^{2}J_{(\text{C-4,H-5})} = 9.3$ $^{2}J_{(\text{C-4,H-5})}$ or $^{2}J_{(\text{C-4,H-3})} = 11.6$ $^{1}J_{(\text{C-5,H})} = 182.3$ $^{2}J_{(\text{C-5,H-4})}$ or $^{3}J_{(\text{C-5,H-3})} = 5.2$ $^{3}J_{(\text{C-5,H-3})}$ or $^{2}J_{(\text{C-5,H-4})} = 9.2$ $^{1}J_{(CH_3\text{—Si})} = 120.4$ $^{1}J_{\text{C}(CH_3)_3} = 125.5$
6 	$^{1}J_{(\text{C-3,H})} = 203.8$ $^{3}J_{(\text{C-3,H-5})} = 11.7$ $^{1}J_{(\text{C-5,H})} = 206.6$ $^{3}J_{(\text{C-5,H-3})} = 8.5$ $^{1}J_{(CH_3\text{—Si})} = 120.6$ $^{1}J_{\text{C}(CH_3)_3} = 125.8$

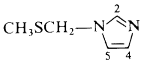
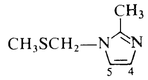
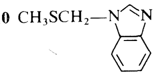
[a]Error ±0.5 Hz, unless otherwise indicated.

N-tert-*Butyldimethylsilyl Heterocycles*

Compounds **1–7** were prepared on a 0.02 mol scale by mixing equimolar amounts of *t*-BuMe$_2$SiCl and the corresponding heterocycle in the presence of excess triethylamine (5, 6). The reaction mixture was either stirred at room temperature for 3–6 days or refluxed for 5–20 h until reaction was complete, as determined by nmr. Et$_3$N·HCl was filtered off and Et$_3$N was removed under vacuum. A trace of Et$_3$N·HCl was removed by adding cyclohexane, refiltering and removing cyclohexane under vacuum. All products (except **6**) were purified by distillation at 10^{-2} Torr and were clear, viscous liquids. Recrystallization of **6** from cyclohexane gave a white solid mp 87–89°C. Yields of final products were 50% or greater.

The products **1** to **7** were identified by ^{13}C nmr (Tables 1 and

TABLE 3. Elemental analyses and ^{13}C nuclear magnetic resonance data of

Compound		Elemental analysis		^{13}C chemical shifts, ppm					
		Calcd.	Found	C-2	C-4	C-5	CH_2	CH_3S	CH_3—C_2
8 	C	46.85	46.89	136.3	129.0	118.1	48.7	13.4	—
	H	6.29	6.19						
	N	21.85	22.20						
9 	C	50.67	51.15	144.4	126.6	119.3	48.2	13.6	12.7
	H	7.09	7.22						
	N	19.70	21.32						
10 	C	60.64	59.35	142.3	—	—	47.2	13.9	—
	H	5.65	5.69						
	N	15.72	15.27						

2), ^1H nmr, and mass spectrometry. Compound **1** has been studied previously (5, 6) and **6** was identified by elemental analysis.

Anal. calcd. for $C_8H_{17}N_3Si$: C 52.41, H 9.35, N 22.92; found: C 51.69, H 9.27, N 22.64.

Reaction with DMSO

N-(Methylthio)methyl derivatives **8–13** were prepared at 140–180°C for 8–24 h in three ways: (*a*) *N*-trimethylsilyl heterocycle and DMSO, (*b*) *N-tert*-butyldimethylsilyl heterocycle and DMSO, (*c*) hexamethyldisilazane, heterocycle, and DMSO. Yields of products were 40–60%.

In a typical reaction, benzimidazole (15 g, 0.13 mol), DMSO (5.0 g, 0.063 mol), and $(Me_3Si)_2NH$ (10.3 g, 0.064 mol) were refluxed for 20 h. The colourless top layer ($Me_3SiOSiMe_3$) was decanted. CCl_4 was added and a white precipitate (benzimidazole, 8.4 g) was removed. The CCl_4 fraction was washed with 3 × 20 mL portions of H_2O and the CCl_4 layer dried ($MgSO_4$) and rotary evaporated to yield a yellow oil which was extracted once more with CCl_4/hexane and dried under vacuum. The oil was identified as *N*-[(methylthio)methyl]benzimidazole on the basis of elemental analysis, carbon nmr (Table 3), proton nmr, and mass spectrometry.

In another reaction, 2-methylimidazole (10 g, 0.12 mol), DMSO (4.8 g, 0.061 mol), and $(Me_3Si)_2NH$ (10 g, 0.062 mol) were refluxed for 8 h. The colourless top layer ($Me_3SiOSiMe_3$) was decanted. CCl_4 was added and the white precipitate (2-methylimidazole) removed. The CCl_4 fraction was rotary evaporated and the product purified by gas chromatography and identified as *N*-[(methylthio)methyl]-2-methylimidazole on the basis of elemental analysis, carbon nmr (Table 3), proton nmr, and mass spectrometry.

Results and Discussion

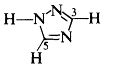
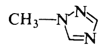
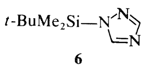
N-(tert-*Butyldimethylsilyl)imidazole and Related Heterocycles*

The ^{13}C nmr spectra (Tables 1 and 2) of compounds **1–7** show clearly that the introduction of the bulky *tert*-butyl substituent gives non-fluxional molecules. In each case, separate signals are observed for non-equivalent ring carbons, as required in the absence of intermolecular silyl exchange. (Only the spectrum of **3** is insensitive to chemical exchange effects.)

The carbon spectra were assigned on the basis of nmr data reported for *N*-methyl analogues of **1–7** (7–9). For the methyl group directly bonded to silicon, $^1J_{C,H} = 120.3^5 \pm 0.2^5$ Hz which is only slightly larger than the value of 118.4 Hz observed for $^1J_{C,H}$ in TMS (10). The $^1J_{C,H}$ values for the methyl carbons of the *tert*-butyl moiety were also the same for all compounds within experimental error, $125.5^5 \pm 0.25$ Hz, slightly larger than the value of 124.04 ± 0.10 Hz observed in neopentane.[1]

$^nJ_{C,H}$ values involving the ring carbons of **1** and **2** are similar to those reported previously for the corresponding *N*-trimethylsilyl derivatives of **1** and **2** (2), while values for **5** and **6** are similar to those observed in 1-methylpyrazole (7, 8) and 1-methyl-1,2,4-triazole (7).

In order to help confirm the assignment of C-3 and C-5 in 1,2,4-triazole derivatives, INDO-MO-FPT level molecular orbital calculations (11) of the various $^nJ_{C,H}$ values in 1,2,4-triazole were carried out. Atomic coordinates were calculated from the bond lengths and angles derived from an X-ray study (12). The results are summarized below.

Parameter	 (Calcd)	 (Obsd, ref. 7)	 **6** (Obsd, this study)
$^1J_{(C\text{-}3,H)}$	180.7 Hz	205.3 Hz	203.8 Hz
$^3J_{(C\text{-}3,H\text{-}5)}$	14.5	11.9	11.8
$^1J_{(C\text{-}5,H)}$	184.4	210.1	206.6
$^3J_{(C\text{-}5,H\text{-}3)}$	11.8	7.3	8.4

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[1] R. E. Wasylishen. Unpublished results.

some *N*-(methylthio)methyl derivatives

^{13}C,H coupling constants, Hz			
$^1J_{(CH_3)} = 139.4$	$^1J_{(CH_2S)} = 152.7$	$^1J_{(C\text{-}2,H)} = 203.8$	
$^1J_{(C\text{-}4,H)} = 189.4$	$^1J_{(C\text{-}5,H)} = 186.5$	$^2J_{(C\text{-}5,H\text{-}4)} = 17.4$	
$^2J_{(C\text{-}4,H\text{-}5)} = 10.4$	$^3J_{(CH_3SCH_2)} = 4.8$	$^3J_{(CH_2SCH_3)} = 4.5$	$^3J_{(C\text{-}4,H\text{-}2)} = 10.4$
$^1J_{(CH_3\text{-}C\text{-}2)} = 128.7$	$^1J_{(CH_3S)} = 139.2$	$^1J_{(CH_2S)} = 151.8$	$^1J_{(C\text{-}4,H)} = 188.8$
$^1J_{(C\text{-}5,H)} = 188.8$	$^2J_{(C\text{-}4,H\text{-}5)} = 9.2$	$^2J_{(C\text{-}5,H\text{-}4)} = 15.9$	
$^3J_{(CH_3SCH_2)} = 4.5$	$^3J_{(CH_2SCH_3)} = 4.8$	$^3J_{(C\text{-}5,CH_2S)} = 3.3$	
$^1J_{(CH_3)} = 139.3$	$^1J_{(CH_2S)} = 152.2$	$^1J_{(C\text{-}2,H)} = 206.0$	
$^3J_{(CH_3SCH_2)} = 4.7$	$^3J_{(CH_2SCH_3)} = 4.8$	$^3J_{(C\text{-}2,CH_2S)} = 3.1$	

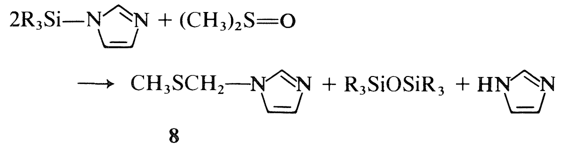
The relative magnitudes of calculated $^1J_{C,H}$ and $^3J_{C,H}$ values are in agreement with the assignments made on the basis of chemical shifts.

The ^{13}C nmr spectra of **1–7** were recorded at elevated temperature. Compounds **1**, **4**, and **6** showed no broadening of the lines at 150°C but **2** and **3** showed broadening at 130°C. In the case of **5**, no exchange was observed at 125°C, but considerable exchange was evident at 150°C and coalescence of C-3 and C-5 occurred at 175 ± 10°C from which we estimate $\Delta G_c^\ddagger = 21.2 \pm 1.0$ kcal/mol (C-4 remained unchanged during this process, as expected).

The proton nmr spectra of **1–7** at 40°C are consistent with rigid structures; however, because of small chemical shift differences the assignment of ring hydrogens could not be made unambiguously.

Reaction of DMSO with N-*Trialkylsilyl Heterocycles*[2]

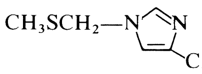
DMSO was found to react with *N*-*tert*-butyldimethylsilyl or *N*-trimethylsilyl heterocycles at elevated temperature 140–180°C in the absence of solvent to give the corresponding *N*-(methylthio)methyl heterocycles **8–13**. Heating a mixture of DMSO, heterocycle, and $(Me_3Si)_2NH$ also gave the same products since, in this case, the *N*-trimethylsilyl heterocycle was formed *in situ* from $(Me_3Si)_2NH$ and heterocycle (4), followed by reaction with DMSO.

[1]


8

The products **8–10** were identified by elemental analysis, carbon nmr (Table 3), proton nmr, and mass spectrometry. Compounds **11–13** were identified by proton nmr and mass spectrometry. The mass spectra of **8–13** showed prominent peaks at M$^+$, M − SCH_3^+, and *m*/*e* 61 ($CH_3SCH_2^+$).

A typical ^{13}C nmr spectrum is shown in Fig. 1. The carbon nmr data confirm the structures of **8–13**



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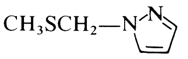
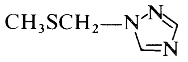


12



13

since the chemical shifts and coupling constants of the ring carbons (Table 3) are very similar to those of the corresponding *tert*-butyldimethylsilyl heterocycles (Table 1 and 2) while the CH_3SCH_2 group in **8–13** is similar to CH_3SCH_2F (13) and CH_3SCH_2Cl (14). The proton nmr of **8–13** showed the CH_3S group at 1.93 to 2.13 ppm and the CH_2 group at 4.78 to 5.37 ppm.

The formation of **8–13** is an example of a Pummerer rearrangement in which a nitrogen, rather than the more familiar oxygen or halogen (15), substituent migrates to the α-position. A possible mechanism is described in Scheme 1. Insertion of

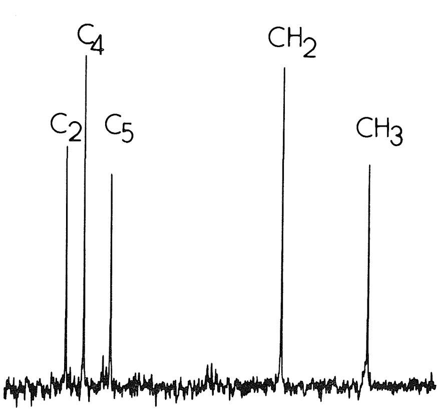


FIG. 1. ^{13}C nmr spectrum of *N*-[(methylthio)methyl]imidazole **8**.

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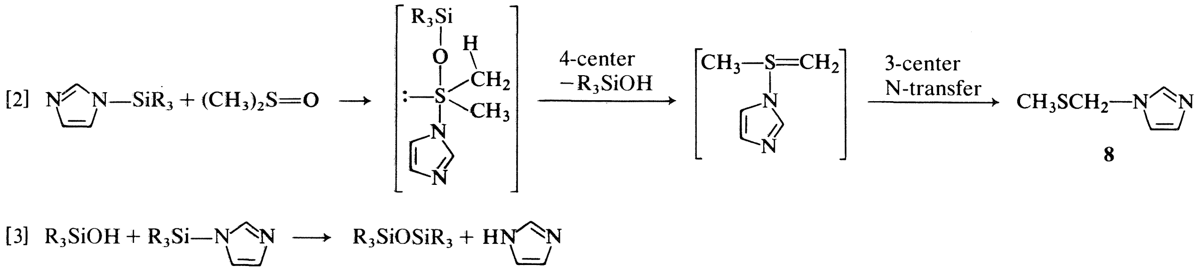
[2] For a preliminary account, see ref. 19.



SCHEME 1

DMSO into the Si—N bond is analogous to SO_2 or SO_3 insertion into Si—N compounds (16) and the 4-center and 3-center steps are similar to the postulated conversion of Me_2SF_2 to $MeSCH_2F$ (13) and Me_2SCl_2 to $MeSCH_2Cl$ (17). Equation [3] is analogous to the reaction of alcohols with Si—N compounds (18).

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