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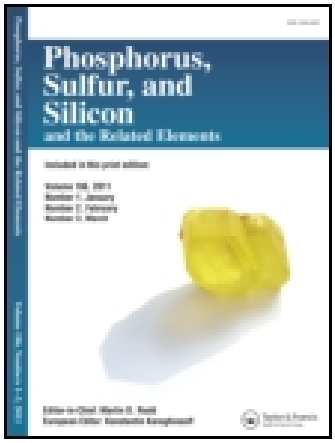

Phosphorus, Sulfur, and Silicon and the Related Elements

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Synthesis of Some Novel 1-(Substituted Phenyl)-2-(4-(Trimethylsilyl)-1H-1,2,3-Triazol-1-YL) Ketones

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SYNTHESIS OF SOME NOVEL 1-(SUBSTITUTED PHENYL)-2-(4-(TRIMETHYLSILYL)-1*H*-1,2,3-TRIAZOL-1-YL) KETONES

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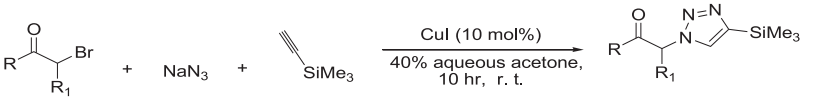
GRAPHICAL ABSTRACT



1a-k **2** **3a-k**

R: substituted phenyl, R_1: H, CH_3.

Abstract *A facile and highly efficient method for the regioselective one-pot synthesis of a series of novel 1-(substituted phenyl)-2-(4-(trimethylsilyl)-1H-1,2,3-triazol-1-yl)ketones from 2-bromo-1-(substituted phenyl)ketones, sodium azide, and trimethylsilyl acetylene, through Cu (I) catalyzed 1,3-dipolar [3+2] cycloaddition reaction is reported. The reaction proceeds smoothly in 40% aqueous acetone at room temperature without the use of any additive. Regioselectivity privileges the formation of the corresponding C-4 silylated-1,2,3-triazoles in high yields in all instances. The methodology offers an entry to new 1,2,3-triazolyl-4-trimethylsilyl anionic synthon scaffolds.*

Keywords [3+2] Cycloaddition; copper (I) catalysis; silylated-1,2,3-triazoles; 2-bromo-1-(substituted phenyl)ketones

INTRODUCTION

Substituted 1,2,3-triazole systems have received significant attention as biologically important heterocycles.[1–3] The triazolyl moiety by itself does not occur in natural products but possesses unique chemical and physical properties.[4] In particular, the trimethylsilyl-containing 1,2,3-triazoles exhibit antibacterial activity against mycobacterium avium tuberculae and anti-HIV activity.[5–7] The development of trimethylsilyl-containing triazole scaffolds has gained interest especially as anionic synthons. Recently, the synthesis of alkyl- and arylethynyl-1,2,3-triazole-fused dihydroisoquinolines using trimethylsilylacetylene and trimethylsilyl-butadiyne is reported.[8] According to the literature, the 1,3-dipolar

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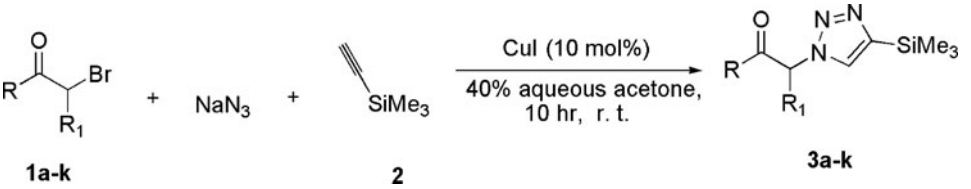
[3+2] cycloaddition of different organic azides with dipolarophile trimethylsilylacetylene and bis(trimethylsilyl)acetylene results in silylated-1,2,3-triazoles[5,7,9–13] and 4,5-bis(trimethylsilyl)-1H-1,2,3-triazoles.[14]

Our laboratory concentrates in the synthesis and reactions of organosilicon compounds and silyl-based reagents.[15–18] In further studies, we were interested in synthetic approaches to trimethylsilyl-substituted-1,2,3-triazoles. There are few known routes to the synthesis of C-4 trimethylsilyl-substituted-1,2,3-triazoles. These routes are multistep syntheses of the triazoles employing differing azides and catalysts at elevated temperatures and involving prolonged times.[5,8,9,12]

In the present article, we provide a versatile one-pot route to the synthesis of some novel 1-(substituted phenyl)-2-(4-(trimethylsilyl)-1*H*-1,2,3-triazol-1-yl)ketone**s 3a–k** in good yields. The orientation of the 1,3-dipolar [3+2] cycloaddition of different 2-bromo-1-(substituted phenyl)ketones **1a–k** with sodium azide and fixing as a probe the dipolarophile trimethylsilyl acetylene **2** is reported.

RESULTS AND DISCUSSION

The 2-bromo-1-(substituted phenyl)ketones **1a–k** were prepared by bromination of the corresponding 1-(substituted phenyl)ketones according to reported literature methods.[19] Initially, the 2-bromo-1-(substituted phenyl)ketones **1a–k** were reacted with sodium azide in an attempt to synthesize the corresponding 2-azido-1-(substituted phenyl)ketones. However, the isolation of the corresponding 2-azido-1-(substituted phenyl)ketones was in poor yields. This prompted us to explore the one-pot synthesis of the 2-bromo-1-(substituted phenyl)ketones **1a–k** with sodium azide and trimethylsilylacetylene **2**. We now report that the substrates undergo a one-pot 1,3-dipolar [3+2] cycloaddition reaction with sodium azide and trimethylsilylacetylene **2** to give 1-(substituted phenyl)-2-(4-(trimethylsilyl)-1*H*-1,2,3-triazol-1-yl)ketones **3a–k** compounds in moderate to good yields (Scheme 1).



Scheme 1 Synthetic approach to the 1-(substituted phenyl)-2-(4-(trimethylsilyl)-1*H*-1,2,3-triazol-1-yl)ketones.

The most attractive approaches to the synthesis of 1,2,3-triazoles involve the thermal 1,3-dipolar [3+2] cycloaddition reactions between azides and alkynes as given by Huisgen.[20] However, the major limitations of this methodology are long reaction times, elevated temperatures, and low regioselectivity. These limitations have been overcome by us employing "click chemistry."

Literature reports have specifically shown that the Cu (I)-catalyzed 1,3-dipolar [3+2] cycloaddition is employed in the regioselective synthesis of 1,4-disubstituted-1*H*-1,2,3-triazoles from terminal acetylenes and azides.[21,22] Further, the "click chemistry" reported by Sharpless has been employed by many other research groups using different copper sources as catalyst to synthesize 1,4-disubstituted-1,2,3-triazoles.[23–26]

In this article, we report that the Cu (I) species can be used directly for the reaction as a catalyst to synthesize 1-(substituted phenyl)-2-(4-(trimethylsilyl)-1*H*-1,2,3-triazol-1-yl)ketones **3a–k**.

The reaction proceeds smoothly in a 40% aqueous acetone system at room temperature without use of any additive. Initially, 2-bromo-1-(substituted phenyl)ketones **1a–k** were reacted with sodium azide and trimethylsilyl acetylene **2** in the presence of CuI (10 mol%) in 40% aqueous acetone system. The conditions employed by us showed good reactivity with completion of the reaction within 10 h at room temperature leading to the exclusive formation of 1-(substituted phenyl)-2-(4-trimethylsilyl)-1*H*-1,2,3-triazol-1-yl)ketones **3a–k** as monitored by thin layer chromatography (TLC). After purification by column chromatography, the products **3a–k** were isolated in good yields ranging between 90% and 96%. The C-4 trimethylsilylated-1,2,3-triazoles **3a–k** were formed in a fully regioselective manner exclusively, with the trimethylsilyl group being directed only to the C-4 position, thus fixing the orientation of the [3+2] cycloaddition regiospecifically. A likely mechanism for the formation of the products **3a–k** involves the route as given by the Huisgen [3+2] cycloaddition reaction.[20]

CONCLUSION

An efficient methodology one-pot "click reaction" employing for 2-bromo-1-(substituted phenyl)ketones **1a–k**, sodium azide and trimethylsilylacetylene **2** using Cu (I) as catalyst without use of any additive via 1,3-dipolar [3+2] cycloadditions in a 40% aqueous acetone system at room temperature are reported with high regioselectively leading exclusively to 1-(substituted phenyl)-2-(4-(trimethylsilyl)-1*H*-1,2,3-triazol-1-yl)ketones **3a–k**. The C-4 trimethylsilyl-1,2,3-triazoles **3a–k** may be used as scaffolds in anionic synthon chemistry.

EXPERIMENTAL SECTION

All reagents and solvents were of reagent grade directly used as received commercially. Silica gel (200–400 mesh; Merck, Mumbai, India) was used for column chromatography. Melting points (mp) of compounds are uncorrected (MP Apparatus, Guna Enterprises, Chennai, India). The reaction was monitored using pre-coated TLC silica gel plates (TLC Silica gel 60 F_{254}; Merck KGaA, Barmstadt, Germany). ^{1}H NMR spectra were recorded on a Bruker AC 400 spectrometer (400 MHz; Germany) and Varian (300 and 400 MHz; Varian Instruments, Palo Alto, California) in DMSO-d_6 or $CDCl_3$ solvents (Aldrich, Bangalore, India). ^{13}C NMR spectra were recorded on Bruker (100 MHz; Germany) in $CDCl_3$ solution. Chemical shifts δ are expressed in parts per million referenced to tetramethylsilane (TMS); coupling constants J are given in Hertz. IR spectra were recorded using KBr pellets on Shimadzu FT-IR 8400S spectrometer (Japan). Electrospray mass spectra (ESI) were obtained on HCT ultra ETDII. Elemental analyses were obtained from Variomicro V1.9.7 (Germany) C H N S mode elemental analyser. The Supplemental Materials file contains sample spectra of compounds **3a**–**3k** (Figures S1–S44).

General Procedure for Synthesis of 2-Bromo-1-(substituted phenyl)ketones 1a–k[18]

To a stirred mixture of 1-(substituted phenyl) ketones (1 mmol) and N-bromosuccinimide (1.5 mmol) in dry CCl_4 (20 mL) was added ammonium acetate

(1 mmol). The reaction was stirred at 0°C until the starting material disappeared (normally 1 h, as monitored by TLC, hexane/acetone, 10:1). After stirring at 25°C for 0.5 h, the mixture was filtered and the filtrate was washed with water, dried, and evaporated. The residue was chromatographed (hexane/acetone, 10:1) on silica gel to give 2-bromo-1-(substituted phenyl)ketones **1a–k** (85%–92%).

General Procedure for the Copper (I) Catalyzed 1-(substituted phenyl)-2-(4-(trimethylsilyl)-1*H*-1,2,3-triazol-1-yl)ketones 3a–k

To a solution of 2-bromo-1-(substituted phenyl)ketones **1a–k** (1 mmol), sodium azide (1.5 mmol), and trimethylsilylacetylene **2** (1.2 mmol) in 40% aqueous acetone (20 mL) was added catalytic amount CuI (10 mol%). The reaction was stirred at room temperature until the starting material disappeared (normally 10 h, as monitored by TLC, hexane/ethylacetate, 7:3). The reaction mixture was diluted with ethyl acetate (50 mL). The catalyst was filtered through Celite. The organic layer was separated, and aqueous layer was extracted with ethyl acetate (3 × 20 mL). The combined organic layers were washed with brine solution. The organic layers were dried over anhydrous Na_2SO_4 and filtered. The solvent was removed in vacuo and the residue was purified by column chromatography on silica gel using (hexane/ethylacetate, 7:3) as eluent to isolate 1-(substituted phenyl)-2-(4-(trimethylsilyl)-1*H*-1,2,3-triazol-1-yl)ketones **3a–k**.

1-(4-Methylphenyl)-2-(4-(trimethylsilyl)-1H-1,2,3-triazol-1-yl)ethanone (3a). White crystals; yield: 94%; mp 103–105°C; FTIR (KBr) ν: 3109, 2954, 1697, 1604, 1498, 1409, 1355, 1251, 1236, 1199, 1117, 1056, 1000, 838, 759, 630, 573, 567 cm^{-1}; ^1H NMR (400 MHz, DMSO) δ: 0.27 (s, 9H), 2.41 (s, 3H), 6.14 (s, 2H), 7.41–7.39 (d, J = 8.0 Hz, 2H), 7.97–7.95 (d, J = 8.2 Hz, 2H), 8.08 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: −1.1 ($SiMe_3$), 21.7 (CH_3), 54.6 (CH_2), 128.2 (C-4), 129.0 (C-1′), 129.8 (C-5), 130.1 (C-2′), 130.7 (C-6′), 131.6 (C-3′), 145.6 (C-5′), 146.9 (C-4′), 190.1 (C=O); ESI-MS *m/z*: 274 [M+H]+; Anal. Calcd. for $C_{14}H_{19}N_3OSi$: C 61.50, H 7.00, N 15.37; found: C 61.48, H 7.20, N 15.45.

1-(3,4-Difluorophenyl)-2-(4-(trimethylsilyl)-1H-1,2,3-triazol-1-yl)ethanone (3b). Brown solid; yield: 95%; mp 108–110°C; FTIR (KBr) ν: 3325, 3122, 2962, 1700, 1600, 1530, 1436, 1249, 1164, 1149, 1051, 1004, 892, 790, 757, 628 cm^{-1}; ^1H NMR (400 MHz, DMSO) δ: 0.27 (s, 9H), 6.18 (s, 2H), 7.73–7.67 (m, 1H), 8.07 (s, 1H), 8.16–8.13 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: −1.1 ($SiMe_3$), 54.5 (CH_2), 117.6 (C-2′), 117.7 (C-5′), 118.1 (C-6′), 118.3 (C-4), 125.4 (C-5), 131.1 (C-1′), 153.2 (C-3′), 155.7 (C-4′), 188.3 (C=O); ESI-MS *m/z* : 296 $[M+H]^+$; Anal. Calcd. for $C_{13}H_{15}F_2N_3OSi$: C 52.86, H 5.12, N 14.23; found: C 52.72, H 5.30, N 14.65.

1-Phenyl-2-(4-(trimethylsilyl)-1H-1,2,3-triazol-1-yl)propan-1-one (3c). Brown color viscous oil; yield: 90%; FTIR (KBr) ν: 3128, 2956, 1693, 1596, 1485, 1450, 1249, 1116, 1045, 966, 842, 759, 700, 639 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 0.32 (s, 9H), 1.81–1.79 (d, J = 6 Hz, 3H), 6.64–6.56 (q, J = 7.2 Hz, 1H), 7.52–7.47 (t, J = 7.5 Hz, 2H), 7.65–7.60 (t, J = 7.5 Hz, 1H), 7.77 (s, 1H), 8.04–8.01 (d, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: −1.2 ($SiMe_3$), 18.8 (CH_3), 58.7 (CH), 127.9 (C-3′), 128.1 (C-5′), 128.5 (C-2′), 128.7 (C-5′), 129.0 (C-4), 129.9 (C-4′), 133.9 (C-5), 138.1 (C-1′), 194.4 (C=O); ESI-MS *m/z* : 274 $[M+H]^+$; Anal. Calcd. for $C_{14}H_{19}N_3OSi$: C 61.50, H 7.00, N 15.37; found: C 61.48, H 7.30, N 15.55.

1-(2,4-Dimethylphenyl)-2-(4-(trimethylsilyl)-1H-1,2,3-triazol-1-yl)ethanone (3d). White crystals; yield: 96%; mp 106–108°C; FTIR (KBr) ν: 2960, 1706, 1610, 1490, 1352, 1251, 1222, 1101, 1051, 987, 842, 757, 632 cm^{-1}; ^1H NMR (400 MHz,

DMSO) δ: 0.27 (s, 9H), 2.34 (s, 3H), 2.41 (s, 3H), 6.02 (s, 2H), 7.22–7.20 (d, $J = 8.1$ Hz, 2H), 7.95 (s, 1H), 8.07 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: −1.1 ($SiMe_3$), 21.4 (CH_3), 21.8 (CH_3), 56.0 (CH_2), 126.7 (C-5′), 129.1 (C-6′), 130.7 (C-3′), 130.8 (C-4), 133.6 (C-1′), 140.5 (C-5), 144.0 (C-1′), 146.8 (C-4′), 192.2 (C=O); ESI-MS m/z : 288 $[M+H]^+$; Anal. Calcd. for $C_{15}H_{21}N_3OSi$: C 62.68, H 7.36, N 14.62; found: C 62.70, H 7.37, N 14.64.

1-(Naphthalen-2-yl)-2-(4-(trimethylsilyl)-1-H-1,2,3-triazol-1-yl)ethanone (3e). Light yellow solid; yield: 92%; mp 140–142°C; FTIR (KBr) ν: 3105, 2958, 1691, 1629, 1357, 1249, 1112, 840, 719, 644, 474 cm^{-1}; ^{1}H NMR (400 MHz, DMSO) δ: 0.29 (s, 9H), 6.32 (s, 2H), 7.70–7.65 (m, 2H), 8.17–8.01 (m, 5H), 8.84 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: −1.3 ($SiMe_3$), 54.7 (CH_2), 123.2 (C-10′), 127.3 (C-5′), 127.9 (C-7′), 129.2 (C-9′), 129.3 (C-6′), 129.7 (C-2′), 130.2 (C-4′), 130.7 (C-4), 131.4 (C-8′), 131.8 (C-5), 132.3 (C-1′), 136.1 (C-3′), 190.5 (C=O); ESI-MS m/z : 310 $[M+H]^+$; Anal. Calcd. for $C_{17}H_{19}N_3OSi$: C 65.98, H 6.19, N 13.58; found: C 65.87, H 6.30, N 13.65.

1-(4-Flourophenyl)-2-(4-(trimethylsilyl)-1H-1,2,3-triazol-1-yl)ethanone (3f). White solid; yield: 93%; mp 110–112°C; FTIR (KBr) ν: 3124, 2962, 1701, 1596, 1510, 1413, 1352, 1232, 1161, 1112, 1053, 995, 835, 757, 630, 592, 493 cm^{-1}; ^{1}H NMR (400 MHz, DMSO) δ: 0.27 (s, 9H), 6.18 (s, 2H), 7.46–7.42 (t, $J = 8.8$ Hz, 2H), 8.07 (s, 1H), 8.17–8.13 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: −1.1 ($SiMe_3$), 54.6 (CH_2), 116.3 (C-3)′, 116.5 (C-5′), 130.5 (C-2′), 130.5 (C-6′), 130.6 (C-4), 130.9 (C-1′), 131.0 (C-5), 167.7 (C-4′), 189.1 (C=O); ESI-MS m/z : 278 $[M+H]^+$; Anal. Calcd. for $C_{13}H_{16}FN_3OSi$: C 56.29, H 5.81, N 15.15; found: C 56.35, H 5.88, N 15.25.

1-(4-Chlorophenyl)-2-(4-(trimethylsilyl)-1H-1,2,3-triazol-1-yl)ethanone (3g). White solid; yield: 96%; mp 118–120°C; FTIR (KBr) ν: 3384, 3120, 2960, 1697, 1589, 1488, 1402, 1352, 1251, 1093, 993, 844, 763, 690, 630, 568, 526, 466 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.35 (s, 9H), 5.85 (s, 2H), 7.53–7.51 (d, $J = 8.4$ Hz, 2H), 7.69 (s, 1H), 7.96–7.94 (d, $J = 8.4$ Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: −1.2 ($SiMe_3$), 54.7 (CH_2), 126.4 (C-3′), 128.7 (C-5′), 128.8 (C-2′), 129.5 (C-6′), 130.7 (C-4), 131.4 (C-5), 132.3 (C-1′), 141.2 (C-4′), 189.4 (C=O); ESI-MS m/z : 294 $[M+H]^+$; Anal. Calcd. for $C_{13}H_{16}ClN_3OSi$: C 53.14, H 5.49, N 14.30; found: C 53.20, H 5.55, N 14.39.

1-(4-Benzene-phenyl)-2-(4-(trimethylsilyl)-1H-1,2,3-triazol-1-yl)ethanone (3h). Light yellow crystals; yield: 94%; mp 138–140°C; FTIR (KBr) ν: 3107, 2956, 1944, 1690, 1598, 1487, 1404, 1355, 1230, 1112, 1056, 991, 838, 761, 696, 632, 578, 536 cm^{-1}; ^{1}H NMR (400 MHz, DMSO) δ: 0.36 (s, 9H), 5.91 (S, 2H), 7.51–7.43 (m, 3H), 7.65–7.63 (d, $J = 7.6$ Hz, 2H), 7.77–7.72 (t, $J = 8$ Hz, 3H), 8.10–8.08 (d, $J = 8$ Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: −1.1 ($SiMe_3$), 54.7 (CH_2), 127.2 (C-4a′), 127.7 (C-3′, C-5′), 128.6 (C-2a′, C-6a′), 128.7 (C-2′, C-6′), 130.7 (C-3a′, C-5a′), 132.7 (C-4), 139.3 (C-5), 147.1 (C-1′), 147.2 (C-1a′), 190.1 (C=O); ESI-MS m/z : 335 $[M]^+$; Anal. Calcd. for $C_{19}H_{21}N_3OSi$: C 68.02, H 6.31, N 12.53; found: C 68.10, H 6.36, N 12.58.

1-(3-Chlorophenyl)-2-(4-(trimethylsilyl)-1H-1,2,3-triazol-1-yl)ethanone (3i). Brown color viscous oil; yield: 91%; FTIR (KBr) ν: 3120, 2960, 1697, 1589, 1488, 1402, 1352, 1251, 1093, 993, 844, 763, 690, 630, 568, 526, 466 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.35 (s, 9H), 5.88 (s, 2H), 7.65–7.37 (m, 2H), 7.70 (s, 1H), 7.97–7.87 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: −1.2 ($SiMe_3$), 54.8 (CH_2), 126.2 (C-6′), 128.2 (C-2′), 129.6 (C-5′), 130.0 (C-5), 130.4 (C-4′), 130.9 (C-5), 132.9 (C-3′), 134.4 (C-1′), 189.5 (C=O); ESI-MS m/z : 294 $[M+H]^+$; Anal. Calcd. for $C_{13}H_{16}ClN_3OSi$: C 53.14, H 5.49, N 14.30; found: C 53.30, H 5.58, N 14.50.

1-(4-(Triflouromethoxy)phenyl)-2-(4-trimethylsilyl)-1H-1,2,3-triazol-1-yl)ethanone (3j). Light green solid; yield: 93%; mp 110–112°C; FTIR (KBr) ν: 3110, 2927, 1701, 1589, 1490, 1253, 1168, 995, 842, 757, 570, 424 cm^{-1}; ^{1}H NMR (300 MHz,

$CDCl_3$) δ: 0.34 (s, 9H), 5.86 (s, 2H), −7.38–7.35 (d, J = 11.2 Hz, 2H), 7.68 (s, 1H), 8.09–8.06 (d, J = 11.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: −1.1 ($SiMe_3$), 54.6 (CH_2), 119.0 (C-3′, C-5′), 120.7 (CF_3), 123.9 (C-1′), 124.4 (C-2′), 130.3 (C-6′), 130.6 (C-4), 132.1 (C-5), 147.3 (C-4′), 189.2 (C=O); ESI-MS m/z : 344 $[M+H]^+$; Anal. Calcd. for $C_{14}H_{16}F_3N_3O_2Si$: C 48.97, H 4.70, N 12.24; found: C 48.95, H 4.75, N 12.30.

1-(4-Methoxyphenyl)-2-(4-trimethylsilyl)-1H-1,2,3-triazol-1-yl)ethanone (3k). White crystals; yield: 95%; mp 139–141°C; FTIR (KBr) ν: 3357, 3128, 2954, 2837, 1689, 1600, 1512, 1423, 1355, 1245, 1184, 1112, 1031, 993, 842, 757, 632, 570, 505 cm^{-1}; ^{1}H NMR (400 MHz, DMSO) δ: 0.27 (s, 9H), 3.87 (s, 3H), 6.10 (s, 2H), 7.12–7.10 (dd, J = 5.12, 1.92 Hz, 2H), 8.05–8.03 (dd, J = 5.08, 1.92 Hz, 2H), 8.07 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: −1.1 ($SiMe_3$), 54.4 (CH_2), 55.6 (CH_3), 114.3 (C-3′, C-5′), 127.0 (C-1′), 130.5 (C-3′, C-6′), 130.6 (C-4), 146.9 (C-5), 164.5 (C-4′), 188.9 (C=O); ESI-MS m/z : 290 $[M+H]^+$; Anal. Calcd. for $C_{14}H_{19}N_3OSi$: C 58.10, H 6.62, N 14.52; found: C 58.12, H 6.65, N 14.58.

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SUPPLEMENTAL MATERIAL

Supplementary data for this article can be accessed on the publisher's website at http://dx.doi.org/10.1080/10426507.2014.947405

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