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A mild and efficient method for the deprotection of trimethyl silyl alkynes using sodium ascorbate and copper sulphate

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ABSTRACT

A competent and fast method for the deprotection of trimethyl silyl group was attained by using cheap, easily accessible, and nontoxic sodium ascorbate in combination with copper sulphate. The method labored was simple and effective for the cleavage of trimethyl silyl group from the protected trimethyl silyl alkynes to their corresponding alkyne derivatives. Wide functional group tolerance, shorter time period, simple procedure and high yields are the striking features of this protocol.

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1. Introduction

Development of mild, efficient and selective route for the protection of variety of functional groups and then de-protection of the protected derivatives continues to be a great challenge in synthetic organic chemistry of polyfunctional molecules including total synthesis of significant natural products [1]. Thus, a number of protecting groups have been developed along with numerous methods for their removal.

Protection and de-protection protocols using silyl protecting groups are among the most widely used methods of temporarily masking alkynes, alcohols and phenols [2]. A variety of methods are available for the conversion of alkynes to their trimethyl silyl alkynes derivatives and to de-protect these derivatives to the parent alkyne [3,4]. In the course of the studies on the synthesis of interesting biological natural products, a mild, fast and efficient method was required for the selective removal of protecting groups without affecting the other functional group. The rate of hydrolysis of trimethyl silyl (TMS) group is influenced by both steric and electronic outcome [5]. Electron withdrawing group increases the rate of basic hydrolysis, and decreases the rate of acidic hydrolysis [6,7]. Many reports says that TMS acetylenes can also be cleaved very conveniently and selectively with fluoride ions especially tetrabutylammonium fluoride [8-14], potassium fluoride in dimethyl formamide [15-19], trimethylamine hydro fluoride in pyridine [20-24] and hydrogen fluoride in methanol or acetonitrile [25-29]. Using these methods, the TMS group can often be removed selectively in the presence of other more bulky trialkyl silyl groups [30,31]. In this article, we wish to report the highly efficient and mild procedure for the de-protection of TMS group attached alkynes using sodium ascorbate and copper sulphate without affecting other functional groups.

2. Experimental

2.1. Instrumentation

Melting points stated were determined in open capillary and are uncorrected.

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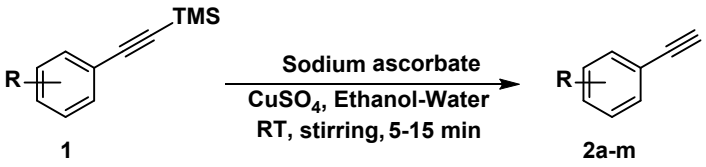


Scheme 1. Synthesis of silyl alkynes.

The structures of the newly synthesized compounds were established using ^{1}H NMR, ^{13}C NMR and LC-MS data. FTIR Spectra were recorded on Jasco FT-IR Spectrometer, ^{1}H NMR and ^{13}C NMR were recorded in $CDCl_3$ at 399.65 MHz and 100.50 MHz, respectively, on Bruker model Avance II Spectrometer. All the chemical shifts were stated in parts per million (ppm). LC-MS were documented using Waters Alliance 2795 separations module and Waters Micromass LCT mass detector. The purity of the compound was confirmed by TLC on pre-coated silica gel plate and additional purification was done using column chromatography.

2.2. Synthesis

Trimethyl silyl acetylene derivatives (0.1 mol) were dissolved in ethanol:water (5:5, *v*:*v*) system, to this mixture sodium ascorbate (0.3 mol) and copper sulphate (0.1 mol) were added at room temperature. Reaction mixture was stirred at room temperature for 5-15 min. Completion of the reaction was monitored by TLC. Ethyl acetate was added and extracted twice with ethyl acetate. Organic layer was washed with brine, dried over $MgSO_4$, filtered and concentrated in rotatory evaporator under vacuum to get crude compound which was purified by performing flash column chromatography using silica gel and 6-10 % ethyl acetate in hexane (Scheme 1, Table 1)

4-Ethynylbenzaldehyde (**2a**): Color: Off white solid. Yield: 91%. M.p.: 90-91 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 3.30 (s, 1H, CH), 7.64 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.85 (d, 2H, *J* = 8.8 Hz, Ar-H), 10.02 (s, 1H, Ar-CHO). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 191.6 (1C, CHO), 136.0 (1C, Ar-C), 132.8 (1C, Ar-C), 129.6 (1C, Ar-C), 128.4 (1C, Ar-C), 82.7 (1C, C-CH), 81.2 (1C, C-CH). MS (*m/z*): 130.9 (M^+). Anal. calcd. for C_9H_6O: C, 83.06; H, 4.65; O, 12.29. Found: C, 83.1; H, 4.60; O, 12.20%.

1-(4-Ethynylphenyl) ethanone (**2b**): Color: Pale yellow solid. Yield: 95%. M.p: 69-70 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 2.61 (s, 3H, CH_3), 3.26 (s, 1H, CH), 7.58 (d, 2H, *J* = 8.8 Hz, Ar-H), 7.91 (d, 2H, *J* = 8.8 Hz, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 197.4 (1C, $COCH_3$), 136.8 (1C, Ar-C), 132.4 (1C, Ar-C), 128.3 (1C, Ar-C), 127.0 (1C, Ar-C), 82.9 (1C, C-CH), 80.5 (1C, C-CH), 26.8 (3C, CH_3). MS (*m/z*): 144.9 (M^+). Anal. calcd. for $C_{10}H_8O$: C, 83.31; H, 5.59; O, 11.10. Found: C, 83.28; H, 5.51; O, 11.2%.

4-Ethynylnitrobenzene (**2c**): Color: Pale yellow solid. Yield: 96%. M.p.: 149-150 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 3.37 (s, 1H, CH), 7.64 (d, 2H, *J* = 8.8 Hz, Ar-H), 8.20 (d, 2H, *J* = 9.2 Hz, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 133.1 (1C, Ar-C), 129.0 (1C, Ar-C), 123.7 (1C, Ar-C), 82.5 (1C, C-CH), 81.7 (1C, C-CH). MS (*m/z*): 147.9 (M^+). Anal. calcd. for $C_8H_5NO_2$: C, 65.3; H, 3.43; N, 9.52. Found: C, 65.0; H, 3.45; N, 9.55%.

4-Ethynylbenzonitrile (**2d**): Color: Off white solid. Yield: 93%. M.p.: 155-156 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 3.31 (s, 1H, CH), 7.57 (d, 2H, *J* = 8.8 Hz, Ar-H), 7.63 (d, 2H, *J* = 8.4 Hz, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 132.8 (1C, Ar-C), 132.1 (1C, Ar-C), 127.1 (1C, Ar-C), 118.4 (1C, CN), 112.4 (1C, Ar-C), 82.0(1C, C-CH), 81.7(1C, C-CH). MS (*m/z*): 127.8 (M^+). Anal. calcd. for C_9H_5N: C, 85.02; H, 3.96; N, 11.02. Found: C, 83.5; H, 3.8; N, 10.9%.

4-Ethynylpyridine (**2e**): Color: Off white solid. Yield: 91%. M.p: 95-96 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 3.31 (s, 1H, CH), 7.35 (dd, *J* = 4.4 Hz, *J* = 1.6 Hz, 2H, Ar-H), 8.60 (dd, *J* = 4.4 Hz, *J* =1.6 Hz, 2H, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 149.9 (1C, Ar-C), 130.4 (1C, Ar-C), 126.2 (1C, Ar-C), 82.0 (1C, C-CH), 81.0 (1C, C-CH). MS (*m/z*): 103.8 (M^+). Anal. calcd. for C_7H_5N: C, 81.53; H, 4.89; N, 13.58. Found: C, 83.5; H, 4.9; N, 13.3%.

2-Ethynylpyridine (**2f**): Color: Brown solid. Yield: 93%. M.p.: 123-124 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 3.17 (s, 1H, CH), 7.27 (dd, *J* = 7.6 Hz, *J* = 4.8 Hz, 1H, Ar-H), 7.49 (d, 1H, *J* = 7.6 Hz, *J* =0.8 Hz, Ar-H), 7.67 (dd, 1H, *J* = 7.6 Hz, *J* = 1.6 Hz, Ar-H), 8.60 (dd, 1H, *J* = 4.8 Hz, *J* = 0.8 Hz, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 150.1 (1C, Ar-C), 142.4 (1C, Ar-C), 136.3 (1C, Ar-C), 127.6 (1C, Ar-C), 123.5 (1C, Ar-C), 82.8 (1C, C-CH), 77.2 (1C, C-CH). MS (*m/z*): 103.9 (M^+). Anal. calcd. for C_7H_5N: C, 81.53; H, 4.89; N, 13.58. Found: C, 83.0; H, 4.55; N, 13.20%.

5-Ethynylpyrimidine (**2g**): Color: Off white solid. Yield: 71%. M.p.: 75-76 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 3.42 (s, 1H, CH), 8.83 (s, 2H, Ar-H), 9.18 (s, 1H, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 159.4 (2C, Ar-C), 157.4 (1C, Ar-C), 118.9 (1C, Ar-C), 84.6 (1C, C-CH), 77.0 (1C, C-CH). MS (*m/z*): 104.9 (M^+). Anal. calcd. for $C_6H_4N_2$: C, 69.22; H, 3.87; N, 26.91. Found: C, 69.25; H, 3.85; N, 26.5%.

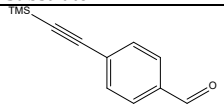
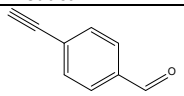
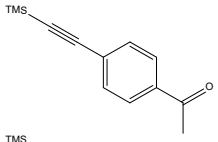
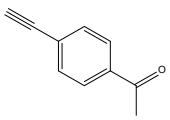
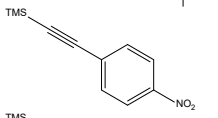
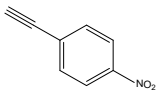
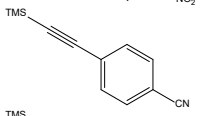
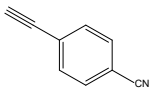
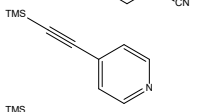
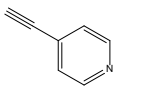
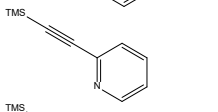
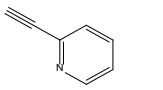
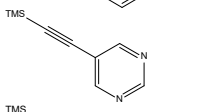
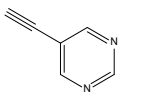
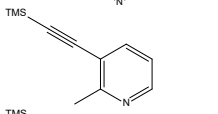
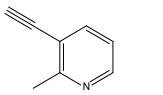
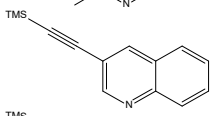
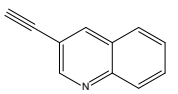
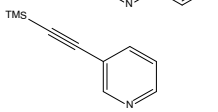
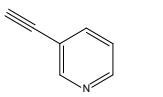
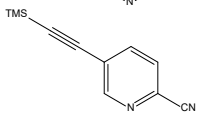
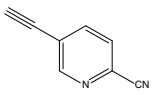
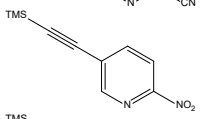
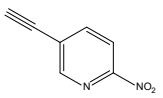
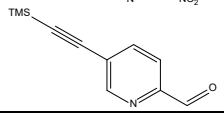
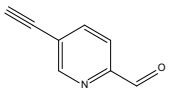
3-Ethynyl-2-methylpyridine (**2h**): Color: Off white solid. Yield: 87%. M.p.: 126-127 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 2.67 (s, 3H, CH_3), 3.28 (s, 1H, CH), 7.08-7.05 (m, 1H, Ar-H), 7.69 (d, 1H, *J* = 7.6Hz, Ar-H), 8.41 (d, 1H, *J* = 4.2 Hz, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 160.3 (1C, Ar-C), 148.3 (1C, Ar-C), 138.9 (1C, Ar-C), 128.8 (1C, Ar-C), 116.8 (1C, Ar-C), 75.0 (1C, C-CH), 69.6 (1C, C-CH), 26.0 (1C, CH_3). MS (*m/z*): 117.8 (M^+). Anal. calcd. for C_8H_7N: C, 82.02; H, 6.02; N, 11.96. Found: C, 81.50; H, 6.20; N, 11.50%.

3-Ethynylquinoline (**2i**): Color: Pale brown solid. Yield: 76%. M.p.: 123-124 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 3.28 (s, 1H, CH), 7.60 (t, 1H, *J* = 7.56 Hz, Ar-H), 7.80-7.72 (m, 2H, Ar-H), 8.11 (d, 1H, *J* = 8.4 Hz, Ar-H), 8.30 (s, 1H, Ar-H), 8.95 (d, 1H, *J* = 1.4 Hz, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 153.6 (1C, Ar-C), 148.2 (1C, Ar-C), 139.3 (1C, Ar-C), 129.9 (1C, Ar-C), 128.9 (1C, Ar-C), 128.3 (1C, Ar-C), 127.0 (1C, Ar-C), 125.9 (1C, Ar-C), 114.6 (1C, Ar-C), 75.0 (1C, C-CH), 69.6 (1C, C-CH). MS (*m/z*): 154.1 (M^+). Anal. calcd. for $C_{11}H_7N$: C, 86.25; H, 4.61; N, 9.14. Found: C, 86.10; H, 4.65; N, 8.90%.

3-Ethynylpyridine (**2j**): Color: Pale yellow solid. Yield: 97 %. M.p.: 118-119 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 3.51 (s, 1H, CH), 7.07-7.04 (m, 1H, Ar-H), 7.69 (d, *J* = 7.6 Hz, 1H, Ar-H), 8.41 (d, *J* = 4.3 Hz, 1H, Ar-H), 8.69 (s, 1H, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 150.8 (1C, Ar-C), 149.3 (1C, Ar-C), 139.7 (1C, Ar-C), 123.4 (1C, Ar-C), 116.4 (1C, Ar-C), 75.0 (1C, C-CH), 69.6 (1C, C-CH). MS (*m/z*): 104.1 (M^+). Anal. calcd. for C_7H_5N: C, 81.53; H, 4.89; N, 13.58. Found: C, 83.0; H, 4.71; N, 13.2%.

5-Ethynylpicolinonitrile (**2k**): Color: Pale yellow solid. Yield: 98%. M.p.:119-120 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 8.73 (s, 1H, Ar-H), 7.85-7.83 (m, 1H, Ar-H), 7.64 (d, *J* = 8.04 Hz, 1H, Ar-H), 3.52 (s, 1H, CH). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 153.5 (1C, Ar-C), 140.3 (1C, Ar-C), 133.3 (1C, Ar-C), 128.9 (1C, Ar-C), 121.0 (1C, Ar-C), 117.1 (1C, Ar-C), 75.0 (1C, C-

Table 1. Deprotection of trimethyl silyl alkynylbenzene derivatives.

Compound	Substrate	Product	Reaction period (min)	Yield (%) *	M.p. (°C)
2a			5	91	90-91
2b			5	95	69-70
2c			10	96	149-150
2d			15	93	155-156
2e			5	87	95-96
2f			5	93	123-124
2g			10	71	75-76
2h			10	87	126-127
2i			10	76	123-124
2j			10	97	118-119
2k			15	98	119-120
2l			15	87	127-128
2m			15	79	122-123

* Isolated yield.

CH), 69.6 (1C, C-CH). MS (*m/z*): 129.1 (M^+). Anal. calcd. for $C_8H_4N_2$: C, 74.99; H, 3.15; N, 21.86. Found: C, 74.5; H, 3.20; N, 21.60%.

5-Ethynyl-2-nitropyridine (**2l**): Color: Pale yellow solid. Yield: 87%. M.p.: 127-128 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 3.56 (s, 1H, CH), 8.05-8.02 (m, 1H, Ar-H), 8.23 (d, 1H, *J* = 9.16 Hz, Ar-H), 8.66 (s, 1H, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 156.7 (1C, Ar-C), 152.5 (1C, Ar-C), 144.2 (1C, Ar-C), 124.0 (1C, Ar-C), 117.1 (1C, Ar-C), 75.0 (1C, C-CH), 69.6 (1C, C-CH). MS (*m/z*): 149.1 (M^+). Anal. calcd. for $C_7H_4N_2O_2$: C, 56.76; H, 2.72; N, 18.91. Found: C, 56.62; H, 2.80; N, 18.70%.

5-Ethynylpicolinaldehyde (**2m**): Color: Off white solid. Yield: 79% yield. M.p.: 122-123 °C. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 3.45 (s, 1H, CH), 7.689 (d, *J* = 8.04 Hz, 1H, Ar-H), 7.91-7.88 (m, 2H, Ar-H), 8.78 (s, 1H, Ar-H). ^{13}C NMR (100 MHz, $CDCl_3$, δ, ppm): 192.6 (1C, C=O), 152.8 (1C, Ar-C), 152.2 (1C, Ar-C), 140.0 (1C, Ar-C), 121.5 (1C, Ar-C), 120.3 (1C, Ar-C), 75.0 (1C, C-CH), 69.6 (1C, C-CH). MS (*m/z*): 132.1 (M^+). Anal. calcd. for $C_8H_4N_2$: C, 73.27; H, 3.84; N, 10.68. Found: C, 73.10; H, 3.70; N, 10.50%.

3. Results and discussion

From side to side, this research paper account for the mild reaction condition to deprotect trimethyl silyl group attached to alkynes selectively without disturbing other functional groups

Table 2. Evaluation of reaction conditions and yield of product with reported methods with the present method.

Entry	Catalyst	Conditions	Time	Yield (%)*	Reference
1	AgCl or AgI catalyst	CH_2Cl_2:MeOH:H_2O (7:4:1, *v*:*v*:*v*), Room temperature	40 hr	3-5	[32]
2	$AgBF_4$ catalyst	CH_2Cl_2:MeOH:H_2O (7:4:1, *v*:*v*:*v*), Room temperature	40 hr	3	[32]
3	$AgNO_3$ catalyst	CH_2Cl_2:MeOH:H_2O (7:4:1, *v*:*v*:*v*), Room temperature	5.5-23 hr	79	[32]
4	AgOTf catalyst	CH_2Cl_2:MeOH:H_2O (7:4:1, *v*:*v*:*v*), Room temperature	2.5-9.0 hr	86	[32]
5	Na_2S	Dry methanol, 0 °C to Room temperature	30 min	77	[33]
6	K_2CO_3	MeOH:THF (3:1, *v*:*v*), Room temperature	20 hr	55	[34]
7	DBU	MeCN:H_2O (19:1, *v*:*v*), 60 °C	0.5-6.0 hr	93-99	[35]
8	$Cu(MeCN)_4PF_6$ / $AgBF_4$	CH_2Cl_2:MeOH (5:5, *v*:*v*), 25-35 °C	18-72 hr	75-98	[36]
9	$KF.2H_2O$	DMF, Room temperature	8 hr	75-80	[37]
10	KF	Tetra ethylene glycol, Room temperature	5 min-20 hr	80-99	[38]

Table 3. Influence of solvent on the synthesis of 5-ethynylpicolinonitrile (**2k**) [a].

Entry	Solvent	Time	Yield (%) [b]
1	Solvent free	4 hr	0
2	Dichloromethane	4 hr	5
3	Ethanol	4 hr	66
4	Methanol	4 hr	65
5	Tetrahydrofuran	4 hr	20
6	Dimethyl formamide	4 hr	22
7	Acetonitrile	4 hr	15
8	Water:Dimethyl formamide (5:5, *v*:*v*)	30 min	65
9	Water:Tetrahyrofuran (5:5, *v*:*v*)	50 min	56
10	Water:Acetonitrile (5:5, *v*:*v*)	45 min	51
11	Water:Methanol (5:5, *v*:*v*)	25 min	85
12	Water:Ethanol (5:5, *v*:*v*)	15 min	98
13	Water	4 hr	20

[a] Sodium ascorbate (0.3 mol) and copper sulphate (0.1 mol) were used beside solvent at room temperature.
[b] Reactions were monitored by TLC and stated were isolated yield.

Table 4. Optimisation of reaction condition for the synthesis of 5-ethynylpicolinonitrile (**2k**).

Entry	Sodium ascorbate (mol)	Copper sulphate (mol)	Ethanol (mL)	Water (mL)	Time	Yield (%) *
1	0.1	0.0	5	5	4 hr	0
2	0.1	0.1	5	5	1 hr	40
3	0.2	0.1	5	5	30 min	80
4	0.3	0.1	5	5	10-15 min	98
5	0.4	0.1	5	5	10-15 min	98
6	0.4	0.2	5	5	10-15 min	98
7	0.0	0.1	5	5	4 hr	0
8	0.3	0.1	10	0	4 hr	66
9	0.3	0.1	0	10	4 hr	25
10	0.3	0.1	2	8	10-15 min	75
11	0.3	0.1	3	7	10-15 min	90
12	0.3	0.1	4	6	10-15 min	95
13	0.3	0.1	6	4	10-15 min	95

* Isolated yield.

using sodium ascorbate and copper sulphate in ethanol and water system. We have established a new protocol which looks much better than others in terms of time, cost effectiveness, selectivity and yield (Scheme 1). Series of trimethyl silyl alkynes were prepared by using standard conditions, which upon deprotection given corresponding alkynes (Table 1, Entry **2a**-**m**).

Initially, we have performed reaction on our model substrate 5-ethynylpicolinonitrile (Table 1, Entry **2k**) to deprotect the trimethyl silyl group of using tetrabutyl ammonium fluoride in tetrahydrofuran. As we have expected, there was a formation of product but along with that there was a formation of other prominent side products which ultimately led to the decrease in yield of the reaction. We have suspected that the other prominent side product may be due the participation of nitrile group present in the second position (Table 1, Entry **2k**) and the characterization is under progress.

This strange result forced us to do few more reactions with other functional group containing silyl alkynes (Table 1, Entry **2a**, **2b**, **2c**, **2d**, **2l** and **2m**) under the same condition, no wonder we had same results. We have even tried the same by taking 5-ethynylpicolinonitrile (Table 1, Entry **2k**) using different procedures (Table 2, Entry 1-10), but all were in vain especially when it comes to time and yield factor.

It was a eureka moment for us, when we have tried the deprotection of 5-ethynylpicolinonitrile with our new approach using sodium ascorbate and copper sulphate in water:ethanol system. In this method, luckily functional groups remained unaffected.

To verify this procedure further and to try it out with different possible solvents first we have attempted to do reactions using 0.3 mol sodium ascorbate and 0.1 mol copper sulphate under solvent free (Table 3, Entry 1) followed by non-polar aprotic organic solvents (Table 3, Entry 2), polar aprotic organic solvents (Table 3, Entry 5, 6 and 7) and protic organic solvents (Table 3, Entry 3 and 4). In all these cases reactions were very sluggish (Table 3, Entry 1 and 2), yield was moderate (Table 3, Entry 5, 6, 7, 9 and 10) and time consuming (Table 3, Entry 3, 4 and 8). Since both the reagents used were inorganic we have tried employing water and conducted reactions using mixture of solvent (Table 3, Entry 8-13). The best results were obtained when 0.3 mol sodium ascorbate, 0.1 mol copper sulphate and ethanol-water system were used (Table 3, Entry 12).

Again to decide the effect of concentration of sodium ascorbate and copper sulphate, reaction with mere sodium ascorbate and reaction with mere copper sulphate were tried but the reaction was not successful even after 4 hr (Table 4, Entry 1 and 7). This indicated that both the reagents are important for the reaction to occur. After conducting many experiments we found that 0.3 mol of sodium ascorbate and 0.1 mol of copper sulphate given very good yield of 98% yield (Table 4, Entry 4). With further increase in sodium ascorbate

(0.4 mol) and copper sulphate (0.2 mol) yield and time remain unaltered (Table 4, Entry 5 and 6).

On the other hand, when we tried to modify the ratio of solvents, found that only water as solvent has given only 25% of yield in 4 hr (Table 4, Entry 9) while only ethanol as solvent managed to give 66% (Table 4, Entry 3). Using a mixture ethanol and water in 5:5 (*v*:*v*) ratio finest results were produced in shorter time frame (Table 4, Entry 12).

In this context, we were pleased to find that silyl alkynes were cleaved exclusively, without affecting other functionality, at room temperature. Compared to the protocols reported to date, cleavage of particular functional group in any substrate containing many functional groups leads to unwanted side products but our method is remarkably mild and selective.

4. Conclusion

In summary, we developed a mild and efficient protocol for the deprotection of silyl alkynes using sodium ascorbate in combination with copper sulphate in ethanol and water system. A wide range of silyl alkynes can be selectively cleaved with high yield even in the presence of acid and base labile functional groups. Moreover, silyl alkyne was cleaved exclusively, without affecting the other functionality, at room temperature. The advantages of this procedure over the previously reported processes include its simplicity and the clean and rapid reaction it promotes. Therefore, we believe that this protocol will find wide applications in the synthesis of complex molecules.

Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.
Author contributions: All authors contributed equally to this work.
Ethical approval: All ethical guidelines have been adhered.
Sample availability: Samples of the compounds are available from the author.

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