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# Expedient method for acylation of amines, alcohols and thiol using Trimethylsilyl acetate

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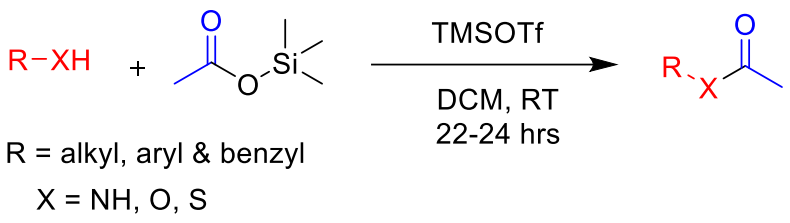
## Abstract

An efficient and convenient protocol has been developed for the acylation of amines, alcohols and thiol using Trimethylsilyl acetate (TMSOAc) is described. The method is simple, mild and economical with good substrate scope. Hence, widely useful for academia and industrial synthetic applications.

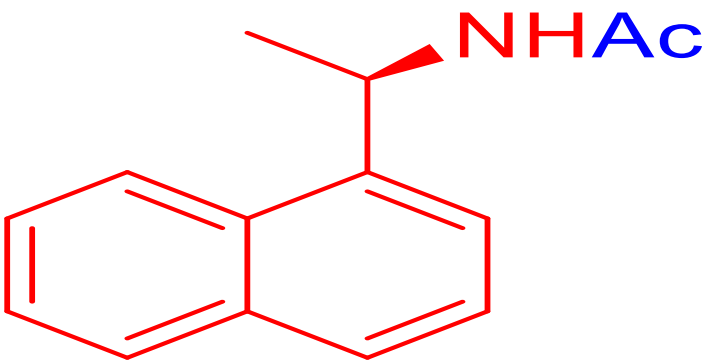



**Keywords**: Acylation, trimethylsilyl trifluoromethanesulfonate, trimethylsilyl acetate

## Introduction

Acylation of alcohols, amines and thiols are often found essential during various key transformations in organic synthesis, especially the synthesis of multifunctional molecules such as nucleosides, carbohydrates, steroids, and natural products. Varieties of synthetic procedures are routinely performed for the conversion of hydroxy, amine and thiol groups to corresponding esters, amides and thioesters including homogeneous and heterogeneous catalyst. There are numerous approaches precedented for acylation of amines, alcohols, and thiols, but most of the them require the use of excess equivalents of acetic anhydride or acetyl chloride in presence of base[1-4] (Scheme 1). Moreover, acetylation of bioactive molecules, hetero atom constituted compounds such as natural phenols, confers enhanced lipophilicity, eventually leading to an improved bioactivity.[5-12]

There are several other methods have been reported for acylation of amines, alcohols and thiols which include 4-(Dimethyl amino)pyridine (DMAP) [13-14], $CoCl_{21}$[15], $ZnCl_2$[16], ZnO[17,18], $CeCl_3$[19], $ZrOCl_2.8H_2O$[20], molecular iodine[21-22], 3-nitrobenzeneboronic acid,[23] $La(NO_3)_3.6H_2O$,[24] $P_2O_5/Al_2O_3$,[25] $NiCl_2$,[26] Co(II)salen-complex,[27] melamine trisulfonic acid,[28] $Sn(TPP)(BF_4)_2$,[29] alkylorthoformate–$ZnCl_2$–$Ac_2O$,[30] vanadium (IV) tetraphenylporphyrin,[31] $[Ti^{IV}(salophen)(OTf)_2]$,[32] *N*-acyl 1,5-diazabicyclo[4.3.0]non-5-ene (DBN) tetraphenylborate salts[33], iron(III) tosylate,[34] $Al(HSO_4)_3$,[35] $NbCl_5$,[36,] $Zn(Otf)_2$[37.] Recently, metal triflates, other metal salts, [38-42] and ionic liquids etc. [43-48] (Scheme 1).




**Scheme 1**. Traditional and novel acylation approaches.

Most of the hitherto known methods suffer from limitations such as harsh reaction conditions, use of expensive reagents, moisture sensitive and toxic catalysts, formation of side products and poor yields of desired products. Hence a highly economical, non-metallic, efficient, and high throughput method is warranted.

The use of silyl-based reagents has gained much more attention nowadays. Silyl carboxylates (acyloxysilane) were first studied by Yur ev and Belyakova in 1960.[49]

## Results and Discussion

Here we report an efficient and convenient method for acetylation of amine, alcohols and thiols with trimethylsilyl acetate (TMSOAc), due to ease of handling, room temperature stability, inexpensive and commercial availability.

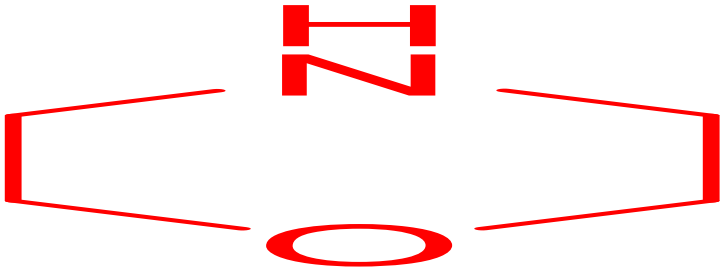
 

In our endeavor, molar equivalent of trimethylsilyl trifluoromethanesulfonate and 2 molar equivalent of trimethylsilyl acetate was opted for acylation of primary, secondary, benzylic and cyclic alcohols, phenols, amines and thiols affording corresponding acylated products in excellent yields without chromatographic purification at room temperature. The reaction of alcohols **1** with trimethylsilyl acetate **2** proceeded smoothly in dichloromethane ($CH_2Cl_2$) and offered the corresponding esters, amides, and thioester with excellent yields [(Table 1), (92-99%)] as shown in Scheme 2. In all the cases, we obtained pure products hence there was no need to perform column chromatography.

In the absence of acid, the corresponding products did not form under the same reaction conditions, even after prolonged reaction time. We attempted the reaction with lower reagent equivalents and obtained lower yields. We screened acylation reaction with various solvents, such as toluene, Dichloromethane (DCM), ethyl acetate, diethyl ether. Among all, DCM is proved to be the best in terms of solubility and, reaction conversions.

The general applicability of this method for a wide variety of compounds was studied (Scheme 2, Table 1). The key advantage of this protocol is that the reactions were found to complete at room temperature. This method is found to tolerate substrates having electron donating and withdrawing functionalities (**3a**-**t**). In addition to the above advantages, we also observed this transformation is equally effective in neat conditions. There is no racemization observed in case of chiral substrates (**3a**, **3f**, **3h**, **3p**, **3r** & **3s).**

This method proved effective even with commercial substrates like **3e, 3g, 3r, 3s.** When compared to previous literature reports, the current protocol is superior in terms of reaction yields. The reagent is inexpensive and easy to handle. The silylated by-product generated after the reaction can be removed easily without column chromatography which makes it viable for scale-up procedures and industrial use.



**Scheme 2**. Acetylation of various – NH, OH and SH.

**Table 1.** Acylation of amines, alcohols and thiols using trimethylsilyl acetate

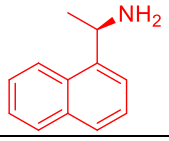
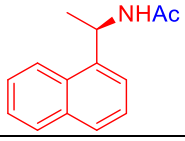
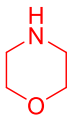
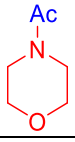
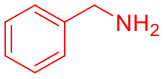
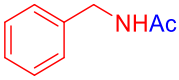
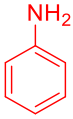
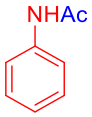
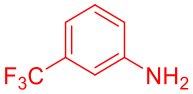
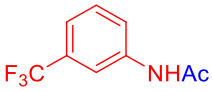
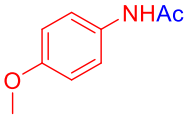
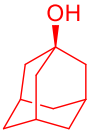
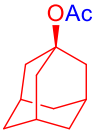
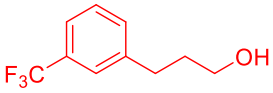
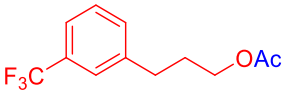
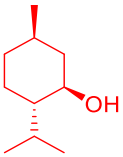
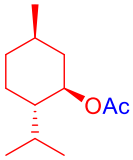
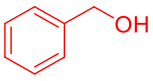
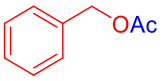
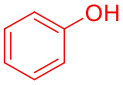
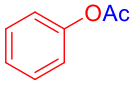
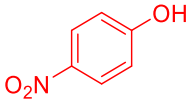
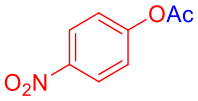
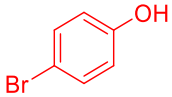
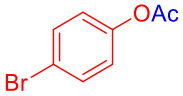
| Entry | Substrate | Product | Product number | Yield (%) |
|---|---|---|---|---|
| 1 |  |  | **3a** | 94 |
| 2 |  |  | **3b** | 95 |
| 3 |  |  | **3c** | 97 |
| 4 |  |  | **3d** | 97 |
| 5 |  |  | **3e** | 95 |
| 6 |  |  | **3f** | 96 |
| 7 |  |  | **3g** | 92 |
| 8 |  |  | **3h** | 94 |
| 9 |  |  | **3i** | 92 |
| 10 |  |  | **3j** | 94 |
| 11 |  |  | **3k** | 92 |
| 12 |  |  | **3l** | 95 |
| 13 |  |  | **3m** | 94 |
| 14 |  |  | **3n** | 94 |

**Table 1.** Continued

| Entry | Substrate | Product | Product number | Yield (%) |
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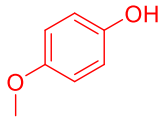
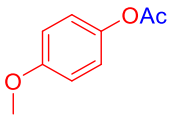
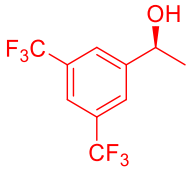
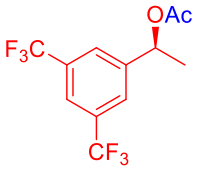
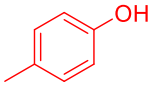
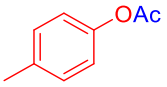
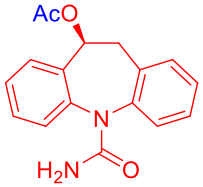
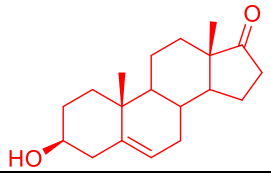
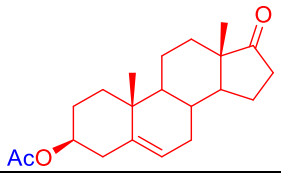
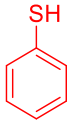
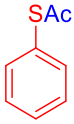
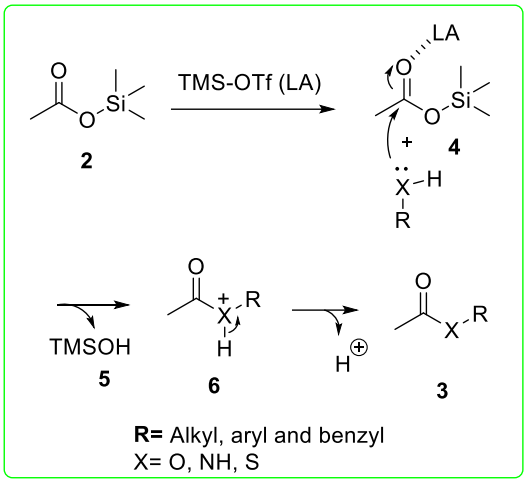
| 15 |  |  | **3o** | 96 |
| 16 |  |  | **3p** | 93 |
| 17 |  |  | **3q** | 95 |
| 18 |  |  | **3r** | 92 |
| 19 |  |  | **3s** | 92 |
| 20 |  |  | **3t** | 94 |




**Scheme 3**. Plausible mechanism for acylation.

The plausible reaction mechanism for the acetylation of amines, alcohols and thiols is shown in scheme 3. An acetyl group of trimethylsilyl acetate is activated with acid leading to transient species that reacted with nucleophiles (alcohols, amines, and thiol) to afford product **3** *via* protonated acylated nucleophiles **6**.

## Conclusions

In conclusion, we have developed and demonstrated method by using trimethylsilyl acetate (TMSOAc) and trimethylsilyl trifluoromethanesulfonate which can be considered as synthetically useful reagent system for efficient acylation of alcohols, phenols, amines and thiols.

## Experimental Section

**General procedure for acetylation.** In a typical experimental procedure; to a mixture of amines/alcohol/phenol/thiols (1.0 mmol) and trimethylsilyl acetate (2.0 mmol) in dichloromethane solvent (10 mL), trimethylsilyl trifluoromethanesulfonate (1.0 mmol) was added at room temperature. The reaction mixture was stirred at room temperature for 24 hrs. Completion of reaction was monitored by TLC after quenching with water, separated the bottom dichloromethane layer and further extracted product into dichloromethane (1 X 10 mL). The combined organic layer was washed with saturated $NaHCO_3$ solution, brine solution dried over anhydrous $Na_2SO_4$ and concentrated under reduced pressure.

All compounds are known and reported previously, (Reference are provided in supplemental material). We have provided the characterization data here $^1$H NMR & $^{13}$C NMR for all compounds and it is in-accordance with the previous reported literature.

***N*-Acetylnaphthylethylamine (3a).** Solid, $^1$H NMR (400 MHz, $CDCl_3$): δ 8.10 (1H, d), 7.87-7.78 (2H, br), 7.54-7.42, (4H, m),5.93(1H, m), 1.95 (3H, s), 1.66 (3H, d), $^{13}$C NMR (400 MHz, $CDCl_3$): 168.9,138.2,133.9,131.1,128.8,128.4,126.6,125.9,125.2,123.5,122.6,44.6,23.4,20.6.

***N*-Acetylmorpholine (3b).** Liquid $^1$H NMR (400 MHz, $CDCl_3$): δ 3.78-3.44 (8H, m), 2.09 (3H, s),$^{13}$C NMR (400 MHz, $CDCl_3$): 169.2,66.9,66.6,66.3,46.7,41.8,21.1.

**Benzyl acetamide (3c).** Solid ,$^1$H NMR (400 MHz, $CDCl_3$): δ 7.38-7.21 (5H, m), 5.88 (1H, br), 4.43 (2H, br), 2.01,(3H,s),$^{13}$C NMR (400 MHz, $CDCl_3$):170.0,138.2,128.7,127.9,127.5,43.8,23.2.

**Acetanilide (3d*).** Solid,$^1$H NMR (400 MHz, $CDCl_3$): δ 7.49-7.38 (2H, br), 7.29-7.18 (2H, m), 7.01-6.98 (1H, br), 2.15 (3H, s), $^{13}$C NMR (400 MHz, $CDCl_3$):168.7,137.8,129.0,124.4,120.1,24.5.

**Benzyl acetamide (3e).** Solid, $^1$H NMR (400 MHz, $CDCl_3$): δ 7.74-7.70 (2H, br), 7.61(1H,br),7.36-7.28(2H,m),2.24(3H), $^{13}$C NMR (400 MHz, $CDCl_3$):169.0,138.4,131.5,131.1,129.5,125.2,123.0, 122.5,120.9,116.7,116.6,24.4.

***N*-(4-Methoxyphenyl)acetamide (3f).** Solid,$^1$H NMR (400 MHz, $CDCl_3$): δ7.41-7.38 (2H, br), 6.96-6.80 (2H, br), 3.78 (3H, s), 2.26 (3H, s).

**(3s,5s,7s)-adamantan-1-yl acetate (3g).** Solid, $^1$H NMR (400 MHz, $CDCl_3$): δ2.08 (3H, s), 2.03 (6H, s), 1.89 (3H, s), 1.59 (6H, s).$^{13}$C NMR (400 MHz, $CDCl_3$): δ 169.3, 79.3, 40.3, 35.2, 29.8, 21.7.

***O*-Aetyltrifloromethylphenylpropanol (3h).** Crude Oil, $^1$H NMR (400 MHz, $CDCl_3$): δ7.38-7.29 (4H,m), 4.02 (2H,q), 2.69(2H,q), 1.98-1.81 (5H, m),$^{13}$C NMR (400MHz,$CDCl_3$): 171.1,142.2,131.8,131.2,130.9,130.2,128.9,128.3,125.6,125.0,123.0,122.9,120.2,63.5,32.0,30.0,20.8.

**(1*R*,2*S*,5*R*)-2-Isopropyl-5-methylcyclohexyl acetate (3i).** Crude Oil $^1$H NMR (400 MHz, $CDCl_3$): δ 4.63-4.57 (1H, td, *J* 4.4 Hz), 195 (3H, s), 1.93-1.90 (1H, m), 1.83-1.75 (1H, m), 1.62-1.57 (2H, m), 1.45-1.37 (1H, m), 1.32-1.18 (1H, m), 1.03-0.87 (2H, m), 0.83-0.82 (6H, d, *J* 6.52 Hz), 0.70-0.68 (3H, d, *J* 6.96 Hz).$^{13}$C NMR (400 MHz, $CDCl_3$):170.6,74.1, 47.0, 40.9, 34.2, 31.3, 26.3, 23.5, 22.0, 21.3, 20.7, 16.4.

$[\alpha]_D^{25}$: + 77 (*c* 1.0, $CHCl_3$).

**Benzyl acetate (3j).** Crude Oil, $^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.35-7.29 (5H, m), 5.08 (2H, s), 2.06 (3H, s),$^{13}$C NMR (400 MHz, $CDCl_3$):170.8, 136.0, 128.6, 128.3, 66.3,21.0, EI-Mass: *m/z* 151 $[M+H]^+$.

**Allyl acetate (3k).** Crude oil, $^{1}$H NMR (400 MHz, $CDCl_3$): δ 5.95-5.87 (1H, m), 5.34-5.21 (2H, dd, *J* 17.2, 17.6 Hz), 4.58-4.56 (2H, m), 2.08 (3H, s),$^{13}$C NMR (400 MHz, $CDCl_3$): δ 170.6, 132.2, 118.1, 65.1, 20.8.

**Phenyl acetate (3l).** Crude Oil, $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.36-7.32 (2H, t, *J* 7.40 Hz), 7.20-7.16 (1H, t, *J* 7.44 Hz), 7.07-7.05 (2H, d, *J* 8.56 Hz), 2.23 (3H, s), $^{13}$C NMR (400 MHz, $CDCl_3$):169.5, 150.8, 129.5, 125.8, 121.6, 21.1, EI-Mass:*m/z* 137 [M+H].

**4-Nitrophenyl acetate (3m).** Solid, $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.20-8.17 (2H, d, *J* 9.12 Hz), 7.22-7.19 (2H, d, *J* 9.12 Hz), 2.27 (3H, s),$^{13}$C NMR (400 MHz, $CDCl_3$):168.4, 155.4, 155.3, 125.2, 122.4, 21.1, EI-Mass:*m/z* 182 $[M+H]^+$.

**4-Bromohenyl acetate (3n).** Crude Oil,$^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.48-7.46 (2H, d, *J* 8.8 Hz), 6.98-6.96 (2H, d, *J* 8.88 Hz), 2.27 (3H, s),$^{13}$C NMR (400 MHz, $CDCl_3$): 169.1, 149.7, 132.5, 123.4, 118.9, 21.1, EI-Mass:*m/z* 215 $[M+H]^+$.

**4-Methoxyphenyl acetate (3o).** Crude Oil; $^{1}$H NMR (400 MHz, $CDCl_3$): δ 6.99-6.97 (2H, d, *J* 9.16 Hz), 6.87-6.85 (2H, d, *J* 9.16 Hz), 3.75 (3H, s), 2.24 (3H, s),$^{13}$C NMR (400 MHz, $CDCl_3$):169.9, 157.3, 144.2, 122.3, 114.4, 55.5, 21.0,EI-Mass:*m/z* 167 $[M+H]^+$.

**(S)-1-(3,5-Bis(trifluoromethyl)phenyl)ethyl acetate (3p).** Crude Oil ,$^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.81 (3H, s), 5.98-5.93 (1H, q, *J* 6.68, 6.64 Hz), 2.12 (3H, s), 1.58-1.57 (3H, d, *J* = 6.64 Hz), $^{13}$C NMR (400 MHz, $CDCl_3$): 170.0, 144.4, 132.4, 126.3, 124.5, 121.9, 7.9, 22.2, 21.0, EI-Mass: *m/z* 301 $[M+H]^+$.

***p*-Tolyl acetate (3q):** $^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.19-7.10 (2H, br), 6.98-6.90 (2H, br), 2.39 (3H, s), 2.26 (3H, s), $^{13}$C NMR (400 MHz, $CDCl_3$): 169.7,148.5,135.5,129.9,121.2,21.1,20.9, EI-Mass: *m/z* 152 $[M+H]^+$.

**(*S*)-5-Carbamoyl-10,11-dihydro-5*H*-dibenzo[*b,f*]azepin-10-yl acetate (3r).** Solid $^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.47-7.42 (2H, m), 7.32-7.23 (6H, m) 6.40-5.99 (1H, b), 5.03(2H, br), 3.61-3.58 (1H, d, *J* 13.48 Hz), 3.19-3.05 (1H, dd, *J* 13.48, 4.09 Hz), 2.09 (3H, s); $^{13}$C NMR (400 MHz, $CDCl_3$): δ 170.7, 170.2, 157.0, 156.7, 141.3, 140.6, 139.1, 134.4, 133.5, 131.0, 129.3, 128.9, 128.3, 128.1, 128.0, 127.8, 72.3, 70.1, 36.0, 35.8, 21.1. HRMS calc for $C_{17}H_{16}N_2O_3$:297.1239, found: 297.124; IR (KBr): 3476, 3361, 2934, 1726, 1653, 1411, 1254 $cm^{-1,}$ $[\alpha]_D^{25}$: 21.0 (*c* =1, pyridine).

**(3S,10R,13S)-10,13-Dimethyl-17-oxo-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl acetate (3s).** Solid, $^{1}$H NMR (400 MHz, $CDCl_3$): δ 5.34-5.33 (1H, d, *J* = 5.12 Hz), 4.56-4.49 (1H, m), 2.42-2.35 (1H, dd *J* = 8.4 Hz), 2.28-2.24 (2H, m), 2.06-1.97 (2H, m), 1.96 (3H, s), 1.91-1.85 (3H, m), 1.63-1.39 (6H, m), 1.26-1.18 (2H, m), 1.11-1.05 (1H, m), 0.98-0.93 (4H, m), 0.81 (3H, s), $^{13}$C NMR (400 MHz, $CDCl_3$): δ 221.0, 170.5, 139.9, 121.9, 73.7, 51.7, 50.1, 47.5, 38.1, 36.9, 36.7, 35.8, 31.5, 31.4,30.8, 27.7, 21.9, 21.4, 20.3, 19.3, 13.5, IR (KBr):1240.0, 1734.6, 1460.7,1434.4, 1369.7, 2949.3, 1023.7 $cm^{-1,}$ EI-Mass: *m/z* 348.4 $[M+NH_4]^+$.

***Benzenethiol* (3t).** Crude Oil $^{1}$H NMR (400 MHz, $CDCl_3$): δ 7.41-7.7.37 (5H, m), 2.38 (3H, s),$^{13}$C NMR (400 MHz, $CDCl_3$): 194.0, 134.5, 129.5, 129.2, 128.0, 30.2, EI-Mass : *m/z* 152 $[M+H]^+$.

## Supplementary Material

Copies of $^{1}$H and $^{13}$C NMR spectra are given in the supplementary material file associated with this manuscript.

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