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Iron(III) tosylate catalyzed acylation of alcohols, phenols, and aldehydes

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ABSTRACT

Iron(III) *p*-toluenesulfonate (tosylate) is an efficient catalyst for acetylation of alcohols, phenols, and aldehydes. The acetylation of 1° and 2° alcohols, diols, and phenols proceeded smoothly with 2.0 mol % of catalyst. However, the reaction worked well with only a few 3° alcohols. The methodology was also applicable to the synthesis of a few benzoate esters but required the use of 5.0 mol % catalyst. Aldehydes could also be converted into the corresponding 1,1-diesters (acylals) under the reaction conditions. Iron(III) tosylate is an inexpensive, and easy to handle, commercially available catalyst.

The conversion of alcohols to esters is an important synthetic transformation that has received considerable attention.[1] Conversion of an alcohol to the corresponding acetate is typically carried out using acetic anhydride or acetyl chloride in the presence of pyridine or triethylamine as a catalyst.[2] 4-(Dimethylamino)pyridine (DMAP) is known to cause a remarkable rate acceleration in this reaction.[3] One problem with tertiary amines is that they are corrosive, toxic, and often highly unpleasant to work with. Lewis acids have also been reported to catalyze the acetylation of alcohols. These include $Bi(OTf)_3$,[4] $Sc(OTf)_3$,[5,6] $CoCl_2$,[7] and I_2.[8] Many of these catalysts are either corrosive (such as I_2) or very expensive (scandium salts). Some examples of acylation catalysts from the recent literature include pentafluorophenylammonium triflate,[9] silica magnesium oxide,[10] polyvinylpolypyrrolidone-bound boron trifluoride,[11] and *N*-acyl 1,5-diazabicyclo[4.3.0]non-5-ene tetraphenylborate salts.[12] Acylation methods that utilize enzymes as catalysts have also been developed.[13] With increasing environmental concerns, it is imperative that new 'environmentally friendly' reagents be developed. Our continued interest in developing environmentally friendly synthetic methodology prompted us to investigate a mild and catalytic method for the acylation of alcohols, phenols, diols, and aldehydes utilizing inexpensive, commercially available reagents. Herein we wish to report that iron(III) tosylate[14] is a mild catalyst for the acylation of a variety of alcohols, phenols, and diols (Table 1) as well as aldehydes (Table 2). As can be seen from Table 1, the reaction worked well with 1° and 2° alcohols (entries 1–11), and phenols (entries 15–17 and 20). When acetic anhydride was used as the acylating agent the reaction could be carried out under solvent-free conditions. With allylic alcohols (entries 3 and 5), the use of solvent (CH_3CN) gave fewer side products. Solvent was also necessary when the acylating agent (benzoic anhydride) was a solid, the reaction mixture solidified without solvent, or if the starting alcohol was poorly soluble in acetic anhydride. In most cases, the crude product was found to be ⩾98% pure by ^1H and ^{13}C NMR spectroscopy and further purification was deemed unnecessary. For solubility reasons, CH_2Cl_2 was used as a solvent in case of benzil (entry 11). Although the methodology was not broadly applicable to tertiary alcohols, we were able to successfully acetylate some 3° alcohols. For example, 1-ethynylcyclohexanol (entry 12) gave a moderate yield of the corresponding acetate. When 1-methylcyclohexanol (entry 13) was subjected to the reaction conditions (in CH_3CN), the crude product although colored was found to contain mostly (80%) the 3° acetate. However, chromatography yielded the pure acetate in only a low yield (38%). Any 1-methylcyclohexene that may have formed is likely to have been lost during removal of the solvent on a rotary evaporator, and hence was not seen in the ^1H spectrum of the crude product.

The hindered 3° alcohol, triphenylmethanol (entry 14), failed to yield the acetate even under reflux conditions, and the starting material was recovered unchanged. When 2-phenyl-2-propanol **1** (Scheme 1) was subjected to the reaction conditions, none of the corresponding acetate was isolated. GC analysis of the crude product, which was obtained as a dark red–brown liquid showed that it mostly (88%) contained product **3**. However, product **3** was

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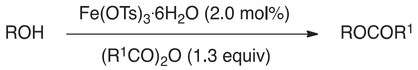
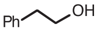
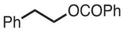
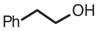
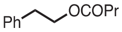
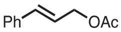
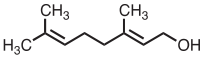
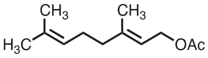
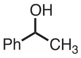
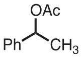
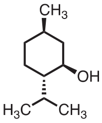
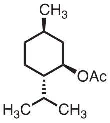
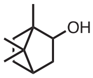
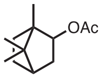
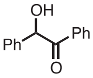
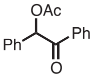
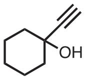
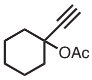
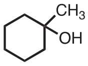
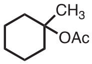
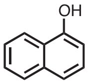
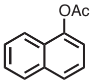
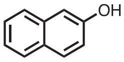
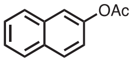
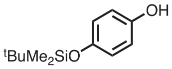
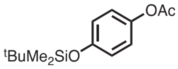
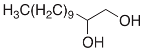
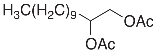
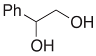
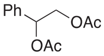
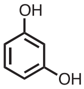
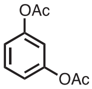
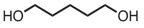
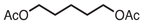
Table 1
Iron(III) tosylate catalyzed acylation of alcohols, phenols, and diols



$$ROH \xrightarrow[(R^1CO)_2O\ (1.3\ equiv)]{Fe(OTs)_3{\cdot}6H_2O\ (2.0\ mol\%)} ROCOR^1$$

Entry	Alcohol	Anhydride R^1	Solvent[a]	Time & Temp[b]	Product	Yield[c] (%)
1a		CH_3	–	10 min		94[15]
1b		Ph[d]	CH_3CN	24 h, 70 °C		73[e,16]
1c		*n*-Pr	–	1 h		85[17]
2		CH_3	–	1 h, 0 °C		92[18]
3		CH_3	–	15 min, 0 °C		95[f,15]
4a		CH_3	–	25 min, 0 °C		66[e,19]
4b		Ph[d]	CH_3CN	27 h, 70 °C		66[e,20]
5		CH_3[d]	CH_3CN	22.5 h, 0 °C		82[g,18]
6	*p*-NO2C6H4CH2OH	CH_3	CH_3CN	45 min	*p*-NO2C6H4CH2OAc	98[15]
7		CH_3	CH_3CN	10 min		92[15]
8		CH_3	CH_3CN	1 h 50 min		93[21]
9		Ph[d]	CH_3CN	44 h, 70 °C		61[22,e]
10		CH_3	CH_3CN	30 h, 50 °C		92[18]
11		CH_3	CH_2Cl_2	18 h		98[15]
12		CH_3	CH_3CN	5 h, 0 °C		95[23]
13		CH_3	CH_3CN	21 h, 0 °C to rt		38[5,e]
14	Ph_3COH	CH_3	CH_3CN	49 h, rt to 70 °C	NR[h]	
15		CH_3	–	2 h		77[15]
16		CH_3	CH_3CN	24.5 h, 50 °C		95[15]
17		CH_3	CH_3CN	1.5 h		70[24,e]
18		CH_3[i]	CH_3CN	3 h		84[25]
19		CH_3[i]	CH_3CN	50 min		89[26]
20		CH_3[i]	CH_3CN	50 min		99[15]
21		CH_3[i]	–	2 h, 0 °C		96[j]

[a] Reagent grade acetonitrile was used.

[b] All reactions were run at room temperature unless otherwise mentioned, and reaction progress was monitored by GC or TLC.

[c] Refers to yield of isolated product that was deemed to be sufficiently pure (> 98%) by ^1H & ^{13}C NMR spectroscopy, unless otherwise mentioned. All products have been previously reported in the literature or are commercially available. The superscript next to yield refers to literature reference for spectral data of the product.

[d] Reaction was carried out using 5.0 mol % catalyst.

[e] Yield of product after purification by flash chromatography.

[f] Product was determined to be 96% pure by GC.

[g] Reaction was carried out using 0.5 mol % catalyst.

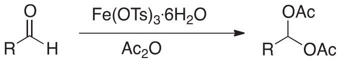
[h] No reaction was observed even when the mixture was heated at reflux for 29 h.

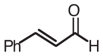
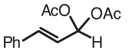
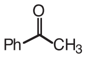
[i] Reaction was carried out with 2.6 equiv of acetic anhydride.

[j] Product is commercially available (CAS # 6963-44-6).

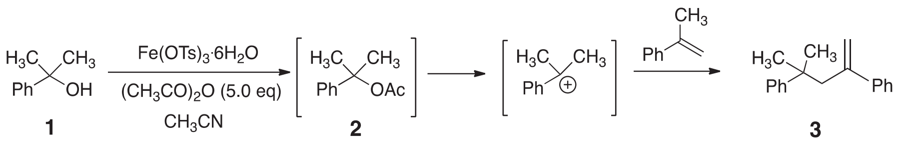
isolated only in 26% yield after chromatographic purification of the crude product. Its identity was confirmed by ^1H and ^{13}C NMR spectroscopy, and HRMS.[27] Based on the fact that the same product was obtained in the absence of acetic anhydride but at a much slower

Table 2
Formation of acylals from aldehydes using $Fe(OTs)_3 \cdot 6H_2O$



Entry	Substrate	Solvent[a]	Temp[a] (°C)	Time[b]	Product[c]	Yield[d] (%)
1	p-ClC_6H_4CHO	–	rt	4 h	p-$ClC_6H_4CH(OAc)_2$	97[39]
2	p-$CH_3OC_6H_4CHO$	–	50	2 h	p-$CH_3OC_6H_4CH(OAc)_2$	50[d,e,39]
3	p-$CH_3C_6H_4CHO$	–	rt	6.5 h	p-$CH_3C_6H_4CH(OAc)_2$	94[40]
4	m-$CH_3OC_6H_4CHO$	–	rt	3 h 40 min	m-$CH_3OC_6H_4CH(OAc)_2$	92[d,39]
5		–		3.5 h		72[f,40]
6	p-$NO_2C_6H_4CHO$	CH_3CN	rt	4 h	p-$NO_2C_6H_4CH(OAc)_2$	90[e,29]
7	$CH_3(CH_2)_8CHO$	–	rt	26 h	$CH_3(CH_2)_8CH(OAc)_2$	71[d,f,41]
8		–	rt	27 h	NR	

[a] Reagent grade acetonitrile was used.
[b] All reactions were run at room temperature unless otherwise mentioned, and reaction progress was monitored by ^1H NMR or TLC.
[c] Refers to yield of isolated product that was deemed to be sufficiently pure (>98%) by ^1H & ^{13}C NMR spectroscopy, unless otherwise mentioned. Superscript next to yield refers to literature reference for the product.
[d] Yield of product after purification by flash chromatography.
[e] Reaction was carried out with 5.0 equiv of acetic anhydride.
[f] Crude product was purified by trituration with pentane/methanol (9:1).



Scheme 1.

rate, we propose that product **3** arises via the initially formed acetate **2**, which subsequently eliminates and dimerizes via a 3° carbocation.

Attempts to make the monoacetate from a symmetrical diol 1, 5-pentanediol (entry 21) using 1 equiv of acetic anhydride were not successful. When 1,5-pentanediol was reacted with 1.05 equiv of acetic anhydride, the product was a mixture of the monoacetate, diacetate, and unreacted starting material. However, formation of the diacetate proceeded smoothly in the presence of 2.6 equiv of acetic anhydride. With diols containing a 1° and a 2° hydroxy group, a noticeable difference in the rate of acetylation of the 1° vs 2° OH was observed in THF as the solvent. When 1-phenethane-1,2-diol (entry 19) was reacted with 1.0 equiv of acetic anhydride in THF as the solvent in the presence of 0.5 mol % of $Fe(OTs)_3 \cdot 6H_2O$, the crude product was found to be (by ^1H NMR) a mixture of the 1° monoacetate (44%), 2° monoacetate (14%), the diacetate (7%) and unreacted starting material (35%). Again, the use of 2.6 equiv of acetic anhydride afforded the diacetate in good yield. We have previously reported that aliphatic TBDMS groups can be cleaved with iron(III) tosylate in the presence of a phenolic TBDMS ether.[14c] Consistent with this observation is the fact that we were able to acetylate a phenol in the presence of a phenolic TBDMS group (entry 17).

Acylals (geminal diacetates) have often been used as protecting groups for carbonyl compounds because they are stable to neutral and basic conditions.[1] Acylals can also be converted into other functional groups, adding to their synthetic utility.[28] Some recent examples of catalysts used for their synthesis include $Al(OTf)_3$,[29] silica chloride,[30] $Fe(NO_3)_3 \cdot 9H_2O$,[31] $SnCl_2 \cdot 2H_2O$,[32] $CoCl_2$,[33] boric acid,[34] ferrous methanesulfonate,[35] $Er(OTf)_3$,[36] $Bi(NO_3)_3 \cdot 5H_2O$,[37] and $Bi(OTf)_3$.[38] Herein we report that iron(III) tosylate is also an efficient catalyst for the acylation of a range of aldehydes under mild conditions (Table 2). The reaction was carried out under solvent-free conditions in most cases. Acetophenone failed to yield any acylal.

Although detailed mechanistic studies were not carried out, a few points merit comment. The observation that a solution of iron tosylate in water is acidic (pH ~2) suggests that the p-TsOH might be an active catalyst in these reactions. Not surprisingly, p-TsOH was found to catalyze the acylation of menthol (Table 1, entry 8) under similar reaction conditions. Furthermore, the acylation of menthol with $Fe(OTs)_3$ (2.0 mol %) in the presence of proton-sponge® (N,N,N',N'-tetramethyl-1,8-naphthalenediamine)®[42] (6.0 mol %) was not successful, and the starting material was recovered. This evidence suggest that p-TsOH could be the true catalyst though the role of Fe^{3+} as a Lewis acid cannot be completely ruled out because it is possible that the Fe^{3+} loses its activity by coordinating to the nitrogen atom in the proton sponge. From a practical standpoint, the use of $Fe(OTs)_3$ is still preferable to p-TsOH because the latter compound is highly toxic and its handling poses a health hazard.[43]

Representative procedures are given here

Method A (synthesis of an acetate under solvent-free conditions): A homogenous mixture of cinnamyl alcohol (0.998 g, 7.44 mmol) and acetic anhydride (0.987 g, 0.91 mL, 9.67 mmol) was stirred as $Fe(OTs)_3 \cdot 6H_2O$ (0.101 g, 0.1487 mmol, 2.0 mol %) was added. The progress of the reaction was followed by GC. After 15 min, aqueous 10% Na_2CO_3 (10 mL) was added and the mixture was stirred for 10 min. The reaction mixture was extracted with ethyl acetate (2 × 20 mL). The combined organic layers were washed with saturated aqueous NaCl (15 mL), dried (Na_2SO_4), and concentrated on a rotary evaporator to yield 1.248 g (95%) of a clear, slightly yellow liquid that was identified as cinnamyl acetate and was determined to be >98% pure by ^1H & ^{13}C NMR spectroscopy, and 96% pure by GC.

Method B (synthesis of an acetate in solvent): A suspension of *p*-nitrobenzyl alcohol (0.486 g, 3.18 mmol) and acetic anhydride (0.421 g, 0.390 mL, 4.13 mmol) in CH_3CN (5 mL) was stirred at room temperature as $Fe(OTs)_3 \cdot 6H_2O$ (43.1 mg, 0.0636 mmol, 2.0 mol %) was added. The progress of the reaction was followed by gas chromatography. After 45 min, CH_3CN was removed on a rotary evaporator, and then aqueous 10% Na_2CO_3 (5 mL) was added to the residue and stirred for 10 min. The reaction mixture was extracted with ethyl acetate (2 × 20 mL). The combined organic layers were washed with saturated NaCl (15 mL), dried (Na_2SO_4) and concentrated on a rotary evaporator to yield 0.61 g (98%) of a yellow solid that was identified as *p*-nitrobenzyl acetate and was determined to be 97% pure by GC, ^1H NMR, and ^{13}C spectroscopy.

Method C (synthesis of a benzoate): A heterogeneous mixture of phenethyl alcohol (0.544 g, 4.45 mmol) and benzoic anhydride (1.51 g, 6.67 mmol) was dissolved in CH_3CN (5 mL) as $Fe(OTs)_3 \cdot 6H_2O$ (151.2 mg, 0.223 mmol, 5.0 mol %) was added. The reaction mixture was heated at 70 °C (temperature controlled hot plate) and the progress of the reaction was followed by TLC (EtOAc/heptane, 30/70). After 24 h, the mixture was cooled, acetonitrile was removed on a rotary evaporator and ethyl acetate (20 mL) was added to the residue. The resulting solution was washed with Na_2CO_3 (2 × 15 mL) and saturated aqueous NaCl (15 mL). The organic layer was dried (Na_2SO_4) and concentrated on a rotary evaporator to yield 1.56 g of a yellow orange liquid. NMR spectroscopy analysis showed that benzoic anhydride was still present in the crude product. The crude product was purified by flash chromatography on silica gel (70 g). A solvent gradient of EtOAc/heptane (10/90, then 20/80) was used for elution. A total of 45 fractions (8 mL-size) were collected, and fractions 15–21 were combined to yield 0.74 g (73%) of a very pale yellow clear liquid that was identified as phenethyl benzoate and was determined to be >98% pure by ^1H and ^{13}C NMR spectroscopy.

Method D (synthesis of an acylal): A homogeneous mixture of *m*-anisaldehyde (0.500 g, 3.67 mmol) and acetic anhydride (0.75 g, 0.694 mL, 7.35 mmol, 2.0 equiv) was stirred at room temperature as $Fe(OTs)_3 \cdot 6H_2O$ (0.0448 g, 0.0735 mmol, 2.0 mol %) was added. (Caution: an exothermic reaction occurs and hence due care must be exercised when scaling up this reaction). The reaction progress was followed by ^1H NMR spectroscopy. After 3 h 40 min, aqueous 10% $NaHCO_3$ (10 mL) was added to the reaction mixture and stirred for 10 min. The reaction mixture was extracted with EtOAc (2 × 20 mL) and the combined organic layers were washed with saturated NaCl (15 mL), dried (Na_2SO_4) and concentrated on a rotary evaporator to yield 0.869 g of an orange liquid. The crude product was purified by flash chromatography (5 g silica) using EtOAc/heptane (20/80) as the eluent to yield 0.801 g (92%) of the acylal as a colorless liquid that was determined to be >98% pure by ^1H and ^{13}C NMR spectroscopy.

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