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Efficient tetrahydropyranyl and tetrahydrofuranyl protection/deprotection of alcohols and phenols with $Al(OTf)_3$ as catalyst

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ABSTRACT

A simple and efficient method for the conversion of alcohols and phenols into their corresponding THP and THF ethers at room temperature has been developed using 1 mol % aluminium triflate as catalyst. The deprotection reaction in the presence of methanol using $Al(OTf)_3$ was equally successful and could be performed at ambient temperature in high yields.

1. Introduction

Protection and deprotection reactions of hydroxyl groups are common transformations in organic synthesis. Tetrahydropyranylation (using 3,4-dihydro-2*H*-pyran, DHP) is one of the most frequently used protocols in organic synthesis for the protection of hydroxyl groups.[1] The structurally similar but less explored tetrahydrofuranyl ethers (using 2,3-dihydrofuran, DHF) are also important and have been employed in hydroxyl group protections.[2] Tetrahydropyranyl (THP) and tetrahydrofuranyl (THF) ethers make ideal protecting groups due to their ease of preparation and stability to a range of reaction conditions including to hydrides, alkylating reagents, Grignard reagents and organometallic reagents. Their sensitivity to acidic deprotection allows orthogonal protecting group strategies to be employed[3] but their co-use with other acid-sensitive groups remains problematic.

Various reagents, including bismuth triflate,[4] *p*-TsOH,[5] Amberlyst H-15[6] and others have been reported to effect tetrahydropyranylation, whereas tetrahydrofuranylation was achieved using the *p*-TsCl/NaH/THF system,[7] $CrCl_2$,[8] ceric ammonium nitrate[9] and recently with alkylperoxy-λ^3-iodane.[10] However, many of these methods suffer from the use of expensive, sometimes exotic reagents, elevated temperatures, strongly acidic conditions, toxic reagents, high catalyst loading and incompatibility with other functional groups. To circumvent some of these problems associated with the tetrahydropyranylation reaction, Kamal et al. reported recently on the application of aluminium triflate as a catalyst in tetrahydropyranylation of several alcohols under solvent-free conditions.[11] Although the method is superior to previously reported protocols, in our hands the protocol was not effective for alcohols such as propargyl alcohols and larger scale reactions were significantly exothermic resulting in many by-products and thus poorer yields than those reported for other systems. We accordingly reinvestigated this reaction making use of $Al(OTf)_3$ as catalyst, which we have shown to be highly efficient for various other transformations.[12] We particularly wished to establish more general conditions for the preparation of THP ethers and also to include THF ethers, which have received little attention in the literature. We report our results on the tetrahydropyranylation and tetrahydrofuranylation of various alcohols and phenols as well as efforts to establish conditions for the mild deprotection of such groups at ambient temperature.

2. Results and discussion

2.1. Protection of alcohols and phenols

In our first attempts at direct tetrahydropyranylation we treated propargyl alcohol **1** with DHP and 1 mol % of $Al(OTf)_3$, using the solvent-free conditions reported by Kamal et al.[11] The desired THP ether was formed in low yield but was accompanied by significant amounts of by-products. However, the use of DCM as a solvent for the reaction at room temperature (Scheme 1) ensured clean conversion to the desired THP ether in quantitative yield. Reaction of but-2-yn-1,4-diol under similar conditions also provided the protected diol in quantitative yield. Treatment of various other alcohols

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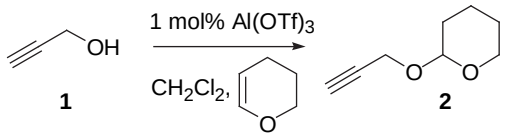
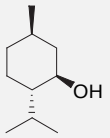
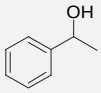
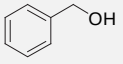
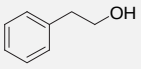
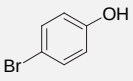
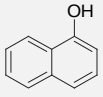
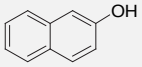
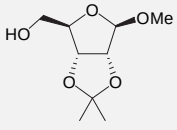


Scheme 1.

and phenols in this way (Table 1) provided efficient access to the desired THP ethers. The protection is useful for primary and secondary alkanols as well as a range of phenols and is highly efficient in DHP and DHF (see below) since only 1.5 equiv of these reagents are used per OH group.

Table 1
Formation of THP ethers from alcohols and phenols using $Al(OTf)_3$.[a]

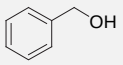
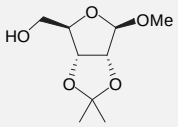
Entry	Substrate	Time (h)	Yield (%)	Ref.
1		3	100	13
2		2	100[b]	14
3		24	69, 1:1 dr	15
4		24	72, 1:3 dr	16
5		8	87[c]	15
6		3	98[c]	16
7		2	71	17
8		3	76	18
9		2	65[c]	19
10		2	67[d]	19
11		45 min	86	—

[a] Substrate (10.0 mmol), 1.5 equiv DHP, 5.0 mL CH_2Cl_2, 1 mol % $Al(OTf)_3$, rt.
[b] DHP (3.0 equiv) was used.
[c] Carried out at 0 °C.
[d] Performed at −10 °C.

2-Naphthols have been found to be difficult to protect as their THP ethers.[4] In the present instance, $Al(OTf)_3$ allowed the protection of this intractable substrate in acceptable yields (Table 1, entry 10), showing the superiority of the catalyst. Importantly, the process was found to be equally applicable to a protected D-ribose derivative (Table 1, entry 11), demonstrating tolerance of other acid-sensitive groups to this protection protocol. The yields overall compare very favourably with reported methods and the reaction conditions are mild.

Although there are several reports on the formation of THF ethers, to the best of our knowledge none of the reported protocols involve Lewis acid catalysts. The broader generality of our method was thus explored by successfully synthesising THF ethers. The alcohols listed in Table 2 were treated under the standard conditions described above to produce the corresponding THF ethers in good to excellent yields. Once again, the D-ribose derivative was readily protected under these conditions.

Table 2
Formation of THF ethers from alcohols and phenols using 1 mol % $Al(OTF)_3$[a]

Entry	Substrate	Time (h)	Yield (%)	Ref.
1		2	81	20
2		2	73[b]	—
3		4	92	8
4		75 min	81	—

[a] Substrate (10.0 mmol), 1.5 equiv DHF, 5.0 mL CH_2Cl_2, 1 mol % $Al(OTf)_3$, rt.
[b] DHF (3.0 equiv) was used.

2.2. Deprotection of THP and THF ethers

The deprotection of THP ethers by merely changing the solvent system has been effected by several reagents including $Bi(OTf)_3 \cdot 4H_2O$,[4] $BF_3 \cdot OEt_2$,[21] $InCl_3$[22] and others. We have now found $Al(OTf)_3$ also to be an efficient deprotection catalyst for THP and THF ethers in the presence of methanol at ambient temperatures to provide the corresponding free alcohols in consistently excellent yields (Table 3). Previously,[4] $Bi(OTf)_3$ has been used in DMF/CH_3OH at 110 °C to effect similar deprotections indicating the present method to be milder.

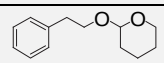
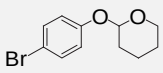
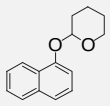
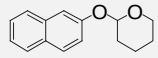
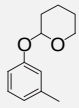
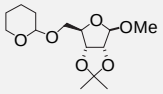
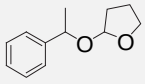
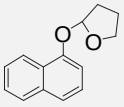
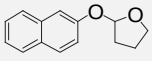
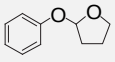
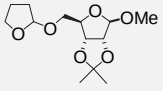
In summary we have shown that aluminium triflate is a versatile catalyst for both protection and deprotection of THP and THF ethers in what is a simple and highly efficient method for the conversion of alcohols to their tetrahydropyranyl as well as tetrahydrofuranyl ethers and vice versa. The ease of handling of the reagent, mild reaction conditions and tolerance of other acid-sensitive groups make it an attractive alternative to existing catalysts for the protection of alcohols and phenols.

3. Experimental

3.1. General

All reactions were carried out in glassware dried by heating (hot air gun) under vacuum, under an atmosphere of nitrogen. Room

Table 3
Deprotection of THP and THF ethers using 1 mol % $Al(OTF)_3$[a]

Entry	Substrate	Time (h)	Yield (%)
1		3	89
2		3	91
3		1.5	89
4		1.5	92
5		2	96
6		2	97
7		1.5	91
8		1.5	95
9		1.5	92
10		2	92
11		2	98

[a] $Al(OTf)_3$ (1 mol %), 5.0 mL MeOH, 10.0 mmol THP or THF ether substrate, rt.

temperature (rt) refers to 20–25 °C. All reagents were of synthetic grade and were used without further purification. $Al(OTf)_3$ was dried before use by heating under vacuum (0.01 mmHg) at 120 °C for 12 h. Dichloromethane was dried by distillation from calcium hydride under nitrogen. Nuclear magnetic resonance spectra were measured in $CDCl_3$ solutions on a Varian Gemini 2000 NMR spectrometer at 300 MHz unless otherwise stated. All chemical shift values for ^1H and ^{13}C nuclei are reported as parts per million δ-values downfield of Me_4Si. The spectral coupling constants (J values) are reported in hertz. Mass spectra were measured on a Thermo DFS magnetic sector instrument using *iso*-butane as the ionisation gas. IR Spectra were recorded on a Tensor 27 spectrophotometer using an ATR diamond fitting.

3.2. General procedure for the protection of alcohols and phenols

To a solution of propargyl alcohol (561 mg, 10.0 mmol) in CH_2Cl_2 (5.0 mL) were added DHP (1.26 g, 15.0 mmol, 1.5 equiv) and $Al(OTf)_3$ (47 mg, 0.099 mmol, 1 mol %) and the solution was stirred at ambient temperature for 2 h. Aqueous sodium bicarbonate (2 mL of a 5% solution) was then added and the mixture extracted with CH_2Cl_2 (3×5 mL). The combined organic phases were dried over anhydrous magnesium sulfate. The volatile component was removed under vacuum leaving the product as a pure material that did not require further purification (1.40 g, 10.0 mmol, 100%). Materials were subjected to column chromatography on flash silica.

3.2.1. Tetrahydro-2-(2-propyn-1yloxy)-2H-pyran.[13] Light yellow oil; R_f 0.32 (ethyl acetate/hexane, 1:7); ^1H NMR (300 MHz, $CDCl_3$) δ 4.71 (1H, t, J 3.1 Hz), 4.17 (1H, dd, J 15.7, 2.5 Hz), 4.12 (1H, dd, J 15.7, 2.5 Hz), 3.72 (1H, td, J 10, 3.8 Hz), 3.46–3.40 (1H, m), 2.34 (1H, t, J 2.5 Hz), 1.77–1.41 (6H, m).

3.2.2. 2,2′-[2-Butyne-1,4-diylbis(oxy)]bis(tetrahydro-2H-pyran).[14] Light yellow oil; R_f 0.26 (ethyl acetate/hexane, 1:7); ^1H NMR (300 MHz, $CDCl_3$) δ 4.66 (2H, t, J 3.0 Hz), 4.21–4.08 (4H, m, overlapping AB systems), 3.68 (2H, ddd, J 11.0, 9.0, 3.8 Hz), 3.39–3.35 (2H, dtd, J 11.0, 4.4, 1.1 Hz), 1.69–1.37 (12H, m).

3.2.3. Tetrahydro-2-[[(1R,2S,5R)-5-methyl-2-(1-methylethyl)cyclohexyl]oxy]-2H-pyran.[15] Colourless oil; 1:1 diastereomeric mixture; R_f 0.72 (ethyl acetate/hexane, 1:7); ^1H NMR (300 MHz, $CDCl_3$) δ 4.76 (0.55H, d, J 3.0 Hz), 4.55 (0.45H, d, J 4.8 Hz), 3.96–3.80 (1H, m), 3.50–3.21 (2H, m), 2.38–0.91 (15H, m), 0.89–0.81 (6H, m), 0.76 (3×0.55H, d, J 6.9 Hz), 0.73 (3×0.45, d, J 7.2 Hz) (fractional integrals represent two diastereomers.).

3.2.4. Tetrahydro-2-(1-phenylethoxy)-2H-pyran.[16] Colourless oil; 1:3 diastereomeric mixture; R_f 0.56 (ethyl acetate/hexane, 1:7); ^1H NMR (300 MHz, $CDCl_3$) δ 7.40–7.16 (5H, m), 4.90–4.78 (1H, m), 4.39 (1H, t, J 3.9 Hz), 3.96–3.89 (0.75H, m), 3.66 (0.25H, td, J 10.1, 3.2 Hz), 3.48–3.41 (0.75H, m), 3.61–3.32 (0.25H, m), 1.86–1.48 (6H, m), 1.46 (3×0.75H, d, J 6.3 Hz), 1.41 (3×0.25H, d, J 6.6 Hz) (fractional integrals represent two diastereomers.).

3.2.5. Tetrahydro-2-(phenylmethoxy)-2H-pyran.[15] Colourless oil; R_f 0.54 (ethyl acetate/hexane, 1:7); ^1H NMR (300 MHz, $CDCl_3$) δ 7.36–7.23 (5H, m), 4.77 (1H, d, J 12.0 Hz), 4.69 (1H, br s), 4.47 (1H, d, J 12.0 Hz), 3.89 (1H, t, J 9.6 Hz), 3.53–3.49 (1H, m), 1.85–1.51 (6H, m).

3.2.6. Tetrahydro-2-(2-phenylethoxy)-2H-pyran.[16] Colourless oil; R_f 0.54 (ethyl acetate/hexane, 1:7); ^1H NMR (300 MHz, $CDCl_3$) δ 7.30–7.18 (5H, m), 4.60 (1H, t, J 3.4 Hz), 3.94 (1H, dt, J 9.6, 7.5 Hz), 3.74 (1H, ddd, J 11.3, 7.6, 3.6 Hz), 3.61 (1H, dt, J 9.6, 7.5 Hz), 3.47–3.40 (1H, m), 2.90 (2H, t, J 7.5 Hz), 1.83–1.45 (6H, m).

3.2.7. Tetrahydro-2-(3-methylphenoxy)-2H-pyran.[17] Cream solid; mp 35–37 °C; R_f 0.68 (ethyl acetate/hexane, 1:7); ^1H NMR (300 MHz, $CDCl_3$) δ 7.05 (1H, t, J 7.8 Hz), 6.78 (1H, s), 6.76 (1H, d, J 7.8 Hz), 6.67 (1H, d, J 7.8 Hz), 5.30 (1H, t, J 3.1 Hz), 3.81 (1H, td, J 10.4, 3.8 Hz), 3.52–3.45 (1H, m), 2.22 (3H, s), 1.95–1.43 (6H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.9, 139.1, 128.9, 122.2, 117.0, 113.3, 96.0, 61.8, 30.3, 25.1, 21.3, 18.7; CI HRMS calcd for $C_{12}H_{16}O_2$ $[M+H]^+$ 192.254, found 192.1147; IR ν_{max} 2942, 1584, 1489, 1254, 1037, 1020, 966 cm^{-1}.

3.2.8. Tetrahydro-2-(4-bromophenoxy)-2H-pyran.[18] White solid; mp 57–58 °C; R_f 0.43 (ethyl acetate/hexane, 1:10); ^1H NMR (300 MHz, $CDCl_3$) δ 7.35 (2H, d, J 8.9 Hz), 6.92 (2H, d, J 8.9 Hz), 5.35 (1H, t, J 3.0 Hz), 3.89–3.70 (1H, m), 3.60–3.55 (1H, m), 2.01–1.55 (6H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.1, 132.2, 118.3, 113.8, 96.5, 62.0, 30.2, 25.1, 18.6.

3.2.9. Tetrahydro-2-(1-naphthalenyloxy)-2H-pyran.[19] White solid; mp 32–34 °C; R_f 0.62 (ethyl acetate/hexane, 1:7); ^1H NMR

(300 MHz, $CDCl_3$) δ 8.31–8.28 (1H, m), 7.78–7.74 (1H, m), 7.48–7.30 (4H, m), 7.12 (1H, dd, *J* 7.5, 0.9 Hz), 5.60 (1H, t, *J* 2.9 Hz), 3.90 (1H, td, *J* 10.7, 2.9 Hz), 3.63–3.56 (1H, m), 2.15–1.55 (6H, m).

3.2.10. Tetrahydro-2-(2-naphthalenyloxy)-2H-pyran.[19] Light yellow oil; R_f 0.54 (ethyl acetate/hexane, 1:7); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74–7.70 (3H, m), 7.43–7.19 (4H, m), 5.55 (1H, t, *J* 3.1 Hz), 3.97–3.89 (1H, m), 3.65–3.59 (1H, m), 2.04–1.56 (6H, m).

3.2.11. Methyl 5-(tetrahydro-2H-pyran-2-yl)-(2,3-O-methylethylidene)-β-D-ribofuranoside. Colourless oil 1.33 g (86%); ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.89 (d, 1H, *J* 1.5 Hz), 4.63 (d, 1H, *J* 5.7 Hz), 4.63–4.59 (m, 1H), 4.51 (dd, 1H, *J* 5.7, 1.5 Hz), 4.30–4.20 (m, 1H), 3.80–3.54 (m, 2H), 3.48–3.18 (m, 2H), 3.20 (s, 3H), 1.81–1.44 (m, 6H), 1.38 (s, 3H), 1.27 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 112.2, 109.2, 98.9, 98.8, 85.2, 85.1, 68.3, 68.2, 62.1, 61.8, 54.8, 54.7, 30.4, 30.3, 26.4, 25.9, 24.9, 20.0, 19.2, 19.1; CIMS (*iso*-butane) *m*/*z* (relative intensity) 289 (14), 258 (26), 257 (100), 205 (17), 173 (23), 85 (98); CI HRMS calcd for $C_{14}H_{25}O_6$ $[M+H]^+$ 289.1651, found 289.1637; IR ν_{max} 2940, 2872, 1372, 1202, 1091, 1032, 869 cm^{-1}.

3.2.12. Tetrahydro-2-(2-propyn-1-yloxy)-furan.[20] Light yellow oil; R_f 0.16 (ethyl acetate/hexane, 1:7); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.16 (1H, t, *J* 2.7 Hz), 4.13–4.01 (2H, m), 3.78–3.74 (2H, m), 2.33 (1H, t, *J* 2.4 Hz), 1.93–1.68 (4H, m).

3.2.13. 2,2′-[2-Butyne-1,4-diylbis(oxy)]bis(tetrahydrofuan). Light yellow oil 1.65 g (73%); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.24 (t, 2H, *J* 2.7 Hz), 4.30–4.10 (m, 4H), 3.86–3.78 (m, 4H), 2.00–1.75 (m, 8H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 102.2, 81.8, 67.1, 54.0, 32.2, 23.2; CIMS (*iso*-butane) *m*/*z* (relative intensity) 227 (9), 157 (36), 141 (19), 71 (100); CI HRMS calcd for $C_{12}H_{19}O_4$ 227.1283, found 227.1278; IR ν_{max} 2924, 1083, 1022, 917 cm^{-1}.

3.2.14. Tetrahydro-2-(phenylmethoxy)-furan.[8] Colourless oil; R_f 0.48 (ethyl acetate/hexane, 1:7); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33–7.24 (5H, m), 5.21–5.19 (1H, m), 4.71 (1H, d, *J* 11.9 Hz), 4.46 (1H, d, *J* 11.9 Hz), 3.95–3.87 (2H, m), 2.02–1.82 (4H, m).

3.2.15. Methyl 5-(tetrahydrofuran-2-yl)-(2,3-O-methylethylidene)-β-D-ribofuranoside. Colourless oil 1.19 g (81%); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.10–5.05 (m, 1H), 4.90 (s, 1H), 4.58 (t, 1H, *J* 6.3 Hz), 4.52 (dd, 1H, *J* 5.9, 2.1 Hz), 4.30–4.18 (m, 1H), 3.90–3.75 (m, 2H), 3.60 (dd, 1H, *J* 9.9, 3.9 Hz), 3.57 (dd, 1H, *J* 10.2, 8.7 Hz), 3.42–3.18 (m, 1H), 3.25 (s, 3H), 2.03–1.70 (m, 4H), 1.42 (s, 3H), 1.27 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 112.2, 109.1, 109.1, 104.2, 103.8, 85.2, 85.1, 85.1, 82.2, 68.0, 67.3, 66.9, 54.6, 32.2, 32.2, 26.4, 25.0, 23.3; CIMS (*iso*-butane) *m*/*z* (relative intensity) 275 (3), 243 (57), 173 (11), 71 (100); CI HRMS calcd for $C_{13}H_{23}O_6$ $[M+H]^+$ 275.1495, found 275.1504; IR ν_{max} 2986, 2924, 1372, 1092, 1036, 870 cm^{-1}.

3.3. General procedure for the deprotection of THP and THF ethers

To a solution of THP-protected 2-naphthol (2.28 g, 10.0 mmol) in MeOH (5.0 mL) was added $Al(OTf)_3$ (47 mg, 0.099 mmol, 1 mol %) and the mixture was stirred at room temperature for 1.5 h after which CH_2Cl_2 (10 mL) was added along with a 5% aqueous solution of sodium bicarbonate (2 mL). The mixture was extracted with CH_2Cl_2 (3×5 mL) and the combined organic phases dried over anhydrous magnesium sulfate. The residue was subjected to column chromatography on flash silica (hexanes/EtOAc 4:1) to provide the pure 2-naphthol (1.33 g, 9.2 mmol, 92%).

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Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tet.2010.04.053. These data include MOL files and InChIKeys of the most important compounds described in this article.

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