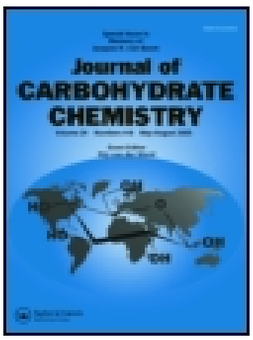

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SHORT COMMUNICATION

Renewable amberlyst-15 catalyzed highly regioselective tritylation and deprotection of sugar-based diols

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ABSTRACT

Amberlyst-15 catalyzed highly regioselective tritylation of sugar-based diols was achieved under mild condition using 4,4′-dimethoxytrityl alcohol (DMTrOH). Deprotection of the corresponding DMTr group was also established by the variation to protic solvent. Meanwhile, the heterogeneous catalyst Amberlyst-15 was recycled 3 times with satisfactory retention of catalytic activity and proved its potential application in industry.

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KEYWORDS
4,4′-dimethoxytrityl group; amberlyst-15; nucleoside; selective protection

Introduction

Hydroxyl group is one of the most common functional groups in organic compounds that are intermediates in the total synthesis of carbohydrates,[1] peptides,[2] proteins and other drug moieties. Even though there are several protecting groups[3] available for hydroxy groups, new protecting groups are still in high demand, which should be easily introduced and removed without altering the actual synthetic target.[4] For the protection of primary alcohols in carbohydrates, nucleosides,[5] peptides and steroids, several protecting groups such as trityl, methoxy-substituted trityl,[6–8] such as 4,4′-dimethoxytrityl (DMTr), and 9-phenylxanthen-9-yl or pixyl[9–12] have been used. Dimethoxytrityl protected derivatives are usually precipitated as solids in the reaction mixture which facilitated product isolation. Deprotection of DMTr ethers are generally carried out under mild acidic conditions while the use of strong acidic media usually leads to the depurination or other undesired byproducts.

The adoption of DMTr group to protect hydroxyl functionalities is conventionally carried out with 4,4′-dimethoxytrityl chloride (DMTrCl)[13] in the presence of different bases.[14] Other methods such as using AgOTf–TrCl,[15] TrODT–TrATCl$_5$,[16] TrCl/MClx,[17] TrOAc/ZnCl$_2$,[18] BnOTr–DDQ,[19] *p*-methoxybenzyl trityl ether[6] (*p*-MBTE) or prenyl trityl

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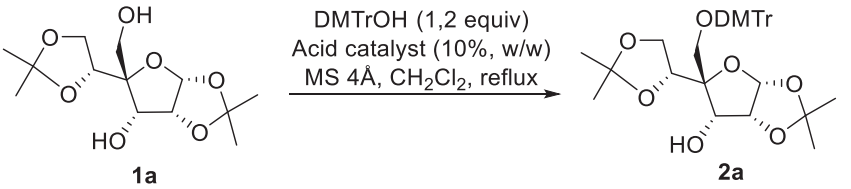
Table 1. Efficiency of various acids to catalyze regioselective DMTr protection of **1a**.



1a **2a**

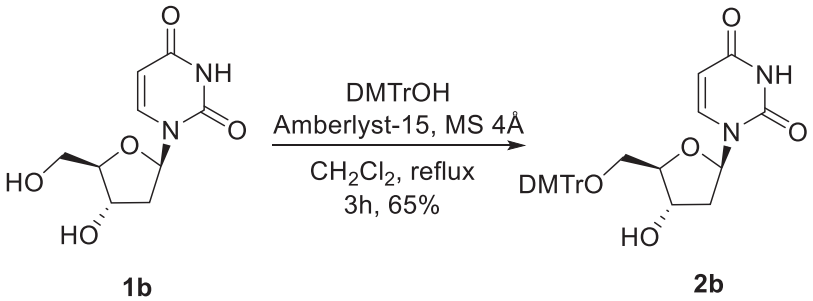
Entry	Acid catalyst	Time (h)	Isolated Yield (%)
1	*p*-TSA	2	68
2	$BF_3.OEt_2$	3	40
3	I_2	2	50
4	$ZnCl_2$	3	65
5	$AlCl_3$	3	60
6	Amberlyst-15	2	75

ether–DDQ,[7] tritylated pyridines[20] and TrOTMS–TMSOTf,[21] are also available. Most of these tritylating and dimethoxy tritylating reagents are not commercially available and have to be prepared from DMTrCl. Moreover, comparing to DMTrCl, dimethoxytriphenylmethanol (DMTrOH) usually showed higher selectivity in the protection of diols.

To date, there are few examples known in the literature describing the protection of alcohols as trityl ethers with DMTrOH[13] in the presence of reusable catalyst. In addition to the limitation on substrate scope and chemoselectivity, the reusability of catalyst such as $FeCl_3$/[bmim]BF_3[22] and MCM-41-SO_3[23] remained a formidable problem. Thus, it is highly desirable to develop effictive method for the selective protection of diols with an eye on the facilitation during the deprotection process. Here we would like to reveal our findings that Amberlyst-15[24] is a mild and reusable catalyst for the selective tritylation and deprotection of sugar-based diols.[25–27]

Results and discussion

To develop a mild, eco-friendly, economical, and efficient protocol for dimethoxytritylation of alcohols, our initial investigation started with selective protection of the primary alcohol of diol sugar using DMTrOH. The evaluation of different acids as the catalyst[15–21] was conducted and the Brönsted acid *p*-toluenesulfonic acid (*p*-TSA) was found to furnish the regioselective DMTr protection of diol sugar **1a** in a 68% yield as shown in Table 1. Lewis acids such as BF_3, $ZnCl_2$ and $AlCl_3$ exhibited lower catalytic performance to give the desired product in moderate yields. However, the renewable Amberlyst-15 catalyst was identified to deliver higher efficiency than other acids affording the desired product in a 75% yield. Encouraged by this result, we further probed the strategy using uridine **1 b** (Scheme 1). Delightfully, in this reaction, regioselectively protected product **2 b** was



Scheme 1. Amberlyst-15 catalyzed regioselective 4,4′-dimethoxytrityl protection of uridine.

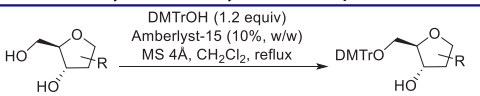
obtained with satisfactory yield considering its low solubility, which demonstrated the potential generality of this protection protocol.

Next, the utility of Amberlyst-15 as a catalyst for DMTr-protection of alcohols was further tested with nine nucleoside substrates **1c**–**1n** that had diverse structures, furnishing **2c**–**2n** in good yields (65–90%; Table 2). It is worthy mentioning that the solubility of substrates showed siginifcant influence in the reaction. The 3′-OH protected substrate **1c,** which is highely soluble in DCM, proceeded well with yields up to 90%. While **1d** with lower solubility afforded the desired product in significantly dropped yield. Interestingly, the DMTr-protection of acetonide protected sugars **1k**–**1n** furnished **2k**–**2n** with reasonable yields (65–72%) without affecting the acetonide protecting group.

One of the benefits of heterogeneous catalysts is their easy separation from a reaction mixture and their reusability. The recyclability of the Amberlyst-15 catalyst was then investigated in the protection reaction of **1j**. The reactions were carried out according to the above procedure for the synthesis of trityl ethers. After the first run, the catalyst was washed with EtOAc, dried at room temperature, and then subjected to a second run. This procedure was repeated three more times, and the average reaction yield for three repeated runs was 92.5%, as shown in Table 3. We cannot rule out the possibility that some unreacted starting materials or even the product could be absorbed on the surface or in the pores of the catalyst which will be incorporated in the next run (comparing the results runs 2 and 3). Nevertheless, it was found that Amberlyst-15 can be used at least for 3 cycles without any apparent loss in activity (Table 3) and proved its potential application in industry.

The deprotection of trityl groups is usually conducted under acidic conditions, which is usually a cause for depurination. We examined the Amberlyst-15 catalyzed deprotection of DMTr ether in protic solvent and discovered that methanol as a solvent served the purpose of deprotecting the DMTr group to afforded the desired products in good to excellent yields (Table 4).

Table 2. Substrate scope of Amberlyst-15 catalyzed DMTr-protection of alcohols.



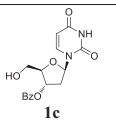
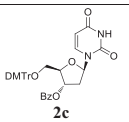
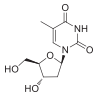
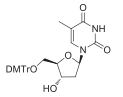
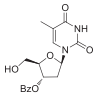
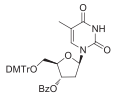
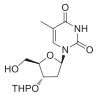
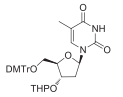
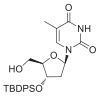
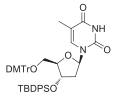
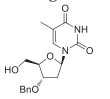
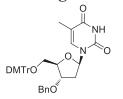
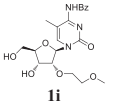
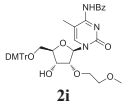
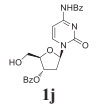
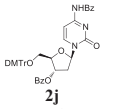
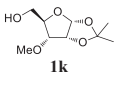
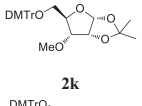
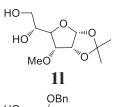
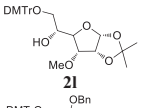
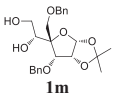
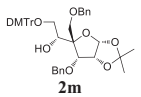
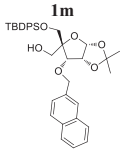
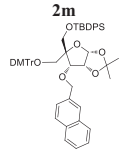
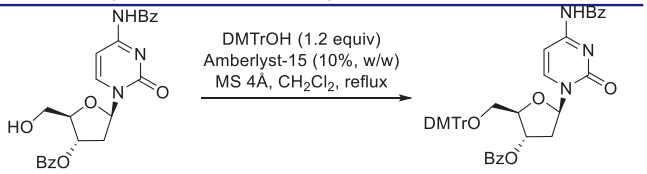
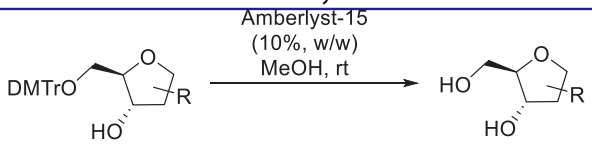
Entry	Substrate	Product	Time (h)	Isolated Yield (%)
1	 **1c**	 **2c**	5	90
2	 **1d**	 **2d**	4	70
3	 **1e**	 **2e**	4	87
4	 **1f**	 **2f**	3	82
5	 **1g**	 **2g**	5	71
6	 **1h**	 **2h**	6	65
7	 **1i**	 **2i**	7	77
8	 **1j**	 **2j**	2	68
9	 **1k**	 **2k**	2	70
10	 **1l**	 **2l**	1	65
11	 **1m**	 **2m**	2	72
12	 **1n**	 **2n**	2	68

Table 3. Recyclisation of the catalyst[a].



Use of resin	Time (h)	Isolated Yield (%)
1st run	2	93
2nd run	3	90
3rd run	4	95

Table 4. Deprotection of DMTr ether with Amberlyst-15[a].



Entry	Starting Material	Product	Time (min)	Isolated Yield (%)
1	**2a**	**1a**	45	93
2	**2e**	**1e**	60	90
3	**2i**	**1i**	30	75
4	**2m**	**1m**	30	80

Conclusion

In summary, we have developed a practical and efficient protocol for regioselective protection of diols by using DMTrOH as the reagent and Amberlyst-15 as the catalyst. The protocol is tolerant to a variety of functional groups, thus offering an additional arsenal to the repertoire of protecting groups. The use of more stable and easy to handle DMTrOH offers the convenience and advantage over conventional method using DMTrCl that is relatively less stable and more expensive. Therefore, this protocol is expected to offer an economical way for large scale synthesis of DMTr-protected molecules. Facile and efficient deprotection of DMTr-protected nucleosides was also accomplished under mild conditions.

Experimental section

General synthetic procedure for 4, 4′-dimethoxytrityl alcohol (1)

Anisole (21.6 g, 0.2 mol,) and benzotrichloride (19.5 g, 0.1 mol) were placed in a three necked RB flask, equipped with a reflux condenser and mechanical stirrer, and attached a trap for addition of solid. The flask was cooled in an ice-bath. Aluminium chloride (10.6 g, 0.08 mol) was added in small portions to the contents of the flask at such rate that the reaction mixture does not reflux during addition. The addition took nearly 1.5 h. The ice–bath was removed 15 min after all the solid had been added and

the reaction was allowed to proceed without further cooling. When the evolution of the gas subsided (nearly 2h). The reaction mixture was poured into a mixture of crushed ice (300 g) and conc. Sulphuric acid (300 ml) and stirred vigorously. The two layers were separated. The aqueous layer was extracted with ethylacetate (1 × 100 ml). The combined organic extracts were further washed with conc. Sulphuric acid (50 ml) and the solvent removed on rotary evaporator to give gummy substance, which was steam distilled to remove any residual anisole. The distillate was dissolved in hexane and dried over Na_2SO_4 and allow stand the solution for crystallization to give DMT-OH **1** (27.8 g, 85.0%) as white color solid.

General experimental procedure for the 4,4′-dimethoxytritylation and pixilation of diols 1a–1n

To a mixture of diol **1a–1n** (1.0 mmol) and Dimethoxy triphenylmethonol **1** (1.1 mmol) in dichloromethane (10 mL), (10% w/w) of Ambelyst-15 was added and the wine red heterogeneous reaction mixture refluxed for 3-6 h. After completion of the starting material **1a–1n,** (monitored by TLC), the catalyst was removed by fitration and the solvents were distilled out on rotary evaporator to get crude product. The crude compound was purified by column chromatography using 60-120 silica gel and (ethyl acetate in hexane) to afford **2a–2n** summarized in Table 2.

The products were confirmed by 1 H NMR, and the spectroscopic data were identical with the data reported in the literature. Analytical data for new compounds which were not reported are listed below.

4,4′-Dimethoxy trityl protection of (3aR,5R,6R,6aR)-5-((bis(4-methoxyphenyl) (phenyl)methoxy)methyl)-6-methoxy-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxole (2k)

^{1}H NMR (500 MHz, $CDCl_3$) δ:1.38 (s, 3H), 1.58 (s, 3H), 3.12 (dd, $J = 10.4$, 3.8 Hz, 1H), 3.39 (s, 3H), 3.49 (m, 2H), 3.78 (s, 6H), 3.82 (q, $J = 4.4$ Hz, 1H), 4.12 (td, $J = 5.7$, 3.0 Hz, 1H), 4.71 (t, $J = 3.8$ Hz, 1H), 5.87 (d, $J = 3.4$ Hz, 1H), 6.82 (d, $J = 8.3$ Hz, 4H), 7.19 (t, $J = 7.2$ Hz, 1H),7.28 (d, $J = 7.6$ Hz, 2H), 7.34 (dd, $J = 8.3$, 5.5 Hz, 4H), 7.36 (d, $J = 7.6$ Hz,2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ:26.4, 26.7, 55.1, 58.4, 61.9, 77.3, 78.0, 80.3, 85.7, 103.9, 113.0, 126.6, 127.7, 128.1, 130.0, 136.0, 144.9, 158.3.

MS: 529.2 (M + Na).

4,4′-Dimethoxy trityl protection of (R)-1-((3aR,5R,6R,6aR)-6-methoxy-2, 2-dimethyl tetrahydrofuro[2,3-d][1,3]dioxol-5-yl)ethane-1, 2-diol (2l)

^{1}H NMR (500 MHz, $CDCl_3$) δ:1.34 (s, 3H), 1.56 (s, 3H), 2.45 (m, 1H), 3.21 (m, 2H), 3.29 (s, 3H), 3.69 (q, $J = 4.4$ Hz, 1H), 3.78 (s, 6H), 3.80 (s, 1H),

3.99 (q, $J = 4.1$ Hz, 1H), 4.13 (m, 1H), 4.62 (t, $J = 4.1$ Hz, 1H), 5.72 (d, $J = 4.1$ Hz, 1H), 6.82 (m, 4H), 7.17 (m, 1H), 7.20 (t, $J = 7.2$ Hz, 1H), 7.28 (t, $J = 7.9$ Hz, 3H), 7.32 (m, 4H), 7.44 (d, $J = 7.6$ Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ:26.4, 26.8, 55.1, 57.9, 63.9, 70.4, 78.1, 79.8, 86.2, 103.9, 113.0, 113.1, 126.7, 127.7, 128.2, 129.1, 130.0, 136.0, 144.7, 158.4.

MS: 559.2 (M + Na).

4,4′-Dimethoxy trityl protection of (3aR,5R,6S,6aR)-5-((bis(4-methoxyphenyl) (phenyl)methoxy)methyl)-5-((R)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-6-ol (2m)

^{1}H NMR (500 MHz, $CDCl_3$) δ:1.46 (s, 3H), 1.64 (s, 3H), 2.95 (d, $J = 6.9$ Hz, 1H), 3.03 (d, $J = 10.3$ Hz, 1H), 3.36 (m,1H), 3.64 (m, 1H), 3.78 (s, 6H), 4.00 (dd, $J = 8.6$, 7.2 Hz, 1H), 4.31 (t, $J = 6.9$ Hz, 1H), 4.37 (t, $J = 6.9$ Hz, 1H), 4.85 (q, $J = 3.4$ Hz,1H), 6.16 (d, $J = 4.1$ Hz, 1H),6.83 (d, $J = 9.0$ Hz, 4H),7.17 (td, $J = 6.0$, 3.7 Hz,1H), 7.25 (m, 8H), 7.36 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ:20.20, 25.34, 50.8, 68.2, 73.4, 77.0, 82.6, 86.3, 101.9, 104.5, 108.8, 110.3, 123.7, 125.6, 131.1, 140.0, 154.2.

MS: 615.3 (M + Na).

4,4′-Dimethoxy trityl protection of (R)-1-((3aR,5S,6S,6aR)-6-(benzyloxy) ((benzyloxy) methyl)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-5-yl)-2-(bis(4-methoxyphenyl) (phenyl)methoxy)ethanol (2n)

^{1}H NMR (500 MHz, $CDCl_3$) δ:1.33 (s, 3H), 1.60 (s, 3H), 3.04 (d, $J = 1.4$ Hz, 1H), 3.15 (dd, $J = 9.6$, 6.2 Hz, 3H), 3.27 (d, $J = 10.3$ Hz, 1H), 3.35 (dd, $J = 10.10$, 3.8 Hz, 1H), 3.49 (d, $J = 10.3$ Hz, 1H), 3.74 (s,6H), 3.78 (d, $J = 4.1$ Hz, 1H), 4.12 (q, $J = 7.1$ Hz, 1H), 4.16 (d, $J = 5.5$ Hz, 1H), 4.26 (t, $J = 11.4$ Hz, 2H), 4.37 (m, 2H), 4.57 (d, $J = 12.4$ Hz, 1H), 4.61 (dd, $J = 5.5$, 4.1 Hz, 1H), 5.76 (d, $J = 3.4$ Hz, 1H),7.11 (q, $J = 3.2$ Hz,2H), 7.16 (m, 3H), 7.23 (t, $J = 7.6$ Hz,2H), 7.26 (m,6H), 7.32(m, 5H), 7.44(d, $J = 6.9$ Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ:22.6, 55.1, 60.3, 64.3, 71.3, 73.5, 79.7, 86.0, 88.8, 104.6, 112.9, 114.1, 127.5, 130.1, 137.4, 145.2, 158.2.

MS: 755.3 (M + Na).

General procedure for the deprotection of DMTr-ether

To a mixture of 4,4'-Dimethoxy trityl ether (1.0 mmol) in MeOH (5 mL) was added (10 mol% w/w) of Ambelyst-15 and stirred the reaction mixture at room temperature for given time (see Table 3). After completion of the reaction (monitored by TLC), catalyst was removed by filtration and the

solvents were distilled out on rotary evaporator to get crude product. The crude compound was purified by column chromatography using 60–120 silica gel and (ethyl acetate in hexane) to afford unprotected nucleosides or carbohydrates summarized in Table 4.

Disclosure statement

No potential conflict of interest was reported by the authors.

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