N-glycosidic bonds. Several alternative procedures for sides, treatment with protic acids led to hydrolysis of the pounds starting from non-halogenated materials, and BB) allowed us to generate simple organolithium com-
triorised trityl ethers. This methodology represents a new and successfully be extended to several hydroxy, alkoxy and amino func-
tionalised trityl ethers. This methodology represents a new and very mild reaction conditions. The detritylation process could suc-

The trityl (triphenylmethyl) group is often employed for
the selective protection of primary alcohols and amines in carbohydrate, peptide and nucleotide chemistry, due to its high steric demand. This protecting group can easily be removed by acid hydrolysis, but some acid-sensitive functional groups cannot survive under the rather harsh reaction conditions. For instance, when the trityl group was used for the protection of the 5'-OH of purine nucleo-
sides, treatment with protic acids led to hydrolysis of the N-glycosidic bonds. Several alternative procedures for the removal of a trityl group have been developed involving, for instance, Lewis acids, electrolytic reduction, cata-
ytic hydrogenation, or reduction with sodium in liquid ammonia, but some of these methods are not applicable to a wide range of unsaturated substrates or other reduc-
gible groups. In the search for better methods for selective removal of the trityl group, some reductive cleavages of the trityl-oxygen bond have recently been published, which use triethylsilane or low-valent titanium reagents. Catalytic amounts of cerium (IV) ammonium nitrate ad-
sorbed on silica gel are able to oxidatively cleave trityl-oxygen bonds in nucleosides and nucleotides very effi-
ciently.

In the last few years, we have been using an arene-cata-
ysed lithiation to prepare organolithium compounds under very mild reaction conditions. The use of an excess of lithium powder and a catalytic amount of an arene [mainly naphthalene or 4,4'-di-tert-butylbiphenyl (DT-
BB)] allowed us to generate simple organolithium com-

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for the difference in the behaviour of these two substrates possessing both a secondary alkyl group.

The trityl group could chemoselectively be removed in the presence of a benzyl or a geranyl group, which are also prone to undergo reductive cleavage by reaction with lithium. The naphthalene-catalysed lithiation of benzyl trityl ether 1d and geranyl trityl ether 1e afforded the corresponding alcohols 2d and 2e, respectively, in almost quantitative yield (Table 1, entries 4 and 5).

The detritylation procedure could successfully be extended to several hydroxy, alkoxy and amino functionalised trityl ethers (Table 1, entries 6–15). 9-Trityloxy-1-nonanol (1f) was treated with n-butyllithium in order to remove the acidic proton of the hydroxy group, which could decompose the naphthalene radical-anion and/or di-anion that act as electron carriers in the process. The generated lithium alkoxide did not undergo the expected cleavage of the trityl group at –78 ºC. However, when the reaction temperature was raised to –30 ºC, a 88% yield of diol 2f was obtained, together with 12% of unreacted starting material (Table 1, entry 6). Stirring the reaction for a longer time at –30 ºC did not improve the yield. A complete conversion of 1f was achieved when the reaction was carried out at 0 ºC and was stirred overnight allowing the temperature to rise to room temperature (Table 1, entry 7). Following the same reaction sequence, that is, deprotonation with n-butyllithium followed by naphthalene-catalysed lithiation at –78 ºC, monoprotected 1,2-diol 1i led to 1,2-octanediol 2i in 89% yield (Table 1, entry 12). The ease of the cleavage in this case, in comparison with compound 1f, could be attributed to the stabilisation of the primary lithium alkoxide by the secondary lithium alkoxide formed after deprotonation via complex induced proximity effect (CIPE).17

The methoxy functionalised trityl ether 1g was quantitatively transformed into 9-methoxy-1-nonanol 2g (Table 1, entry 8). When ditritylated 1,9-nonanediol 1h was used as starting material, it was possible to remove one or both trityl groups depending on the reaction conditions. The naphthalene-catalysed lithiation of 1h at –78 ºC gave in 7 hours a 74% yield of the monoprotected diol 1f, together with 26% of unreacted starting material (Table 1, entry 9). However, when the reaction was performed at –30 ºC, both trityl groups were removed and 1,9-nonanediol 2f was obtained in quantitative yield (Table 1, entry 10). The double detritylation of compound 1h could also be achieved without any loss of yield by carrying the reaction out at 0 ºC and stirring it overnight allowing the temperature to rise to room temperature (Table 1, entry 11).

The cleavage of both oxygen-trityl bonds in compound 1j could be performed at –78 ºC but gave only a 42% yield of the expected diol 2j (Table 1, entry 13). Neither the substrate 1j nor any mono detritylation product, were observed in the 1H NMR spectrum of the crude reaction mixture. A possible explanation for the moderate yield of diol 2j could be the participation of the initially generated lithium alkoxide as a base leading to an intra- or intermolec-

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### Table 1: Reductive Cleavage of Trityl Ethers 1 by a Naphthalene-Catalysed Lithiation Process

<table>
<thead>
<tr>
<th>Entry</th>
<th>Substrate</th>
<th>T (ºC)</th>
<th>t (h)</th>
<th>Product*</th>
<th>Yield (%)b</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>[OTr]5 (1a)</td>
<td>–78</td>
<td>3.5</td>
<td>(2a)OH</td>
<td>97</td>
</tr>
<tr>
<td>2</td>
<td>[OTr]5 (1b)</td>
<td>–65</td>
<td>3.5</td>
<td>(2b)OH</td>
<td>62</td>
</tr>
<tr>
<td>3</td>
<td>[OTr]5 (1c)</td>
<td>–78</td>
<td>2.7</td>
<td>(2e)OH</td>
<td>78c</td>
</tr>
<tr>
<td>4</td>
<td>[OTr]5 (1d)</td>
<td>–78</td>
<td>1.0</td>
<td>(2d)OH</td>
<td>&gt;99</td>
</tr>
<tr>
<td>5</td>
<td>[OTr]5 (1e)</td>
<td>–78</td>
<td>1.5</td>
<td>(2e)OH</td>
<td>&gt;99</td>
</tr>
<tr>
<td>6d</td>
<td>OH</td>
<td>–30</td>
<td>2.3</td>
<td>(2f)OH</td>
<td>88c</td>
</tr>
<tr>
<td>7d</td>
<td>1f</td>
<td>0–20</td>
<td>20.0</td>
<td>2f</td>
<td>&gt;99</td>
</tr>
<tr>
<td>8</td>
<td>OMe</td>
<td>–78</td>
<td>2.2</td>
<td>(2g)OH</td>
<td>&gt;99</td>
</tr>
<tr>
<td>9</td>
<td>[OTr]5 (1h)</td>
<td>–78</td>
<td>7.0</td>
<td>1f</td>
<td>74f</td>
</tr>
<tr>
<td>10</td>
<td>1h</td>
<td>–30</td>
<td>3.5</td>
<td>2f</td>
<td>&gt;99</td>
</tr>
<tr>
<td>11</td>
<td>1h</td>
<td>0–20</td>
<td>20.0</td>
<td>2f</td>
<td>&gt;99</td>
</tr>
<tr>
<td>12d</td>
<td>OH</td>
<td>–78</td>
<td>5.0</td>
<td>(2i)OH</td>
<td>89</td>
</tr>
<tr>
<td>13</td>
<td>[OTr]5 (1j)</td>
<td>–78</td>
<td>7.0</td>
<td>(2j)OH</td>
<td>42c</td>
</tr>
<tr>
<td>14</td>
<td>[OTr]5 (1k)</td>
<td>–78</td>
<td>4.3</td>
<td>(2k)OH</td>
<td>47c</td>
</tr>
<tr>
<td>15</td>
<td>[OTr]5 (1l)</td>
<td>–78</td>
<td>3.8</td>
<td>(2l)OH</td>
<td>60f</td>
</tr>
</tbody>
</table>

a All products 2 were ≥95% pure (GLC and/or 300 MHz 1H NMR).
b Isolated yield after column chromatography (silica gel, hexane–EtOAc) based on the starting material 1.

* Yield determined by quantitative GLC, using commercially available alcohol 2 and n-dodecane (internal standard) in the determination of response factors.

d Compounds 1f and 1i were deprotonated with n-BuLi before performing the naphthalene-catalysed lithiation step.
c 12% of unreacted starting material 1f was also obtained.
f 26% of unreacted starting material 1h was also obtained.
ular elimination of triphenylmethanol. Moderate yields of detritylated products were also obtained with the amino functionalised trityl ethers 1k and 1l (Table 1, entries 14 and 15). The occurrence of similar elimination processes of the amino functionality could be the reason for the decrease in the yields of the expected amino alcohols.

In all cases, triphenylmethane was obtained as a by-product, resulting from hydrolysis of the triphenylmethyl lithium generated during the process, but it could easily be separated from the desired detritylation products by column chromatography.

The starting trityl ethers 1 were prepared by reaction of the corresponding alcohols with trityl chloride in the presence of triethylamine and a catalytic amount of 4-(dimethylamino)pyridine.

In summary, in this paper we have presented a very efficient method for the detritylation of trityl ethers via a naphthalene-catalysed lithiation process under very mild reaction conditions. The methodology has proved to be useful for the removal of the trityl group from tritylated primary, secondary, allylic or benzylic alcohols, as well as from hydroxy, alkoxy and amino functionalised trityl ethers. This method represents a good alternative to the commonly used detritylation procedures, which use acidic reaction conditions.

FT-IR spectra were obtained on a Nicolet Impact 400D spectrophotometer using NaCl plates (for oils) or KBr plates (for solid compounds). NMR spectra were recorded on a Bruker AC-300 (300 MHz for 1H and 75 MHz for 13C) using CDCl3 as solvent and TMS (0.00 ppm, 1H) and CDCl3 (77.0 ppm, 13C) as internal standards; chemical shifts are given in ppm and coupling constants (J) in Hz. 14C NMR assignments were made on the basis of DEPT experiments. Mass spectra (EI) were obtained at 70 eV on a Shimadzu QP-5000 spectrometer. High resolution mass spectra and elemental analyses were measured by the Technical Services at the University of Alicante. TLC was carried out on Schleicher & Schuell F1400/LS 254 plates coated with a 0.2 mm layer of silica gel; detection was done by UV254 light and staining with phosphomolybdic acid [phosphomolybdic acid (5 g) in absolute ethanol (120 mL)]; Rf values are given under these conditions. Column chromatography was performed using silica gel 60 of 35–70 mesh. All reagents used for the synthesis of trityl ethers 1 and naphthalene were commercially available (Acros, Aldrich) and were used without further purification. Lithium powder was prepared according to the procedure described in reference 18. Commercially available n-butyllithium was titrated with a 1 M solution of sec-butanol in xylene using 1,10-phenanthroline as indicator. 19 Commercially available anhydrous THF (99.9%, water content ≤ 0.006%, Acros) was used as solvent in all the lithiation reactions.

**Synthesis of Trityl Ethers 1; General Procedure**

A solution of the corresponding alcohol 2 (10.0 mmol) in CH2Cl2 (5 mL) was added to a solution of trityl chloride (3.097 g, 11.0 mmol), Et3N (2.5 mL, 17.6 mmol) and DMAP (92 mg, 0.4 mmol) in CH2Cl2 (10 mL) at r.t. and the mixture was stirred overnight. The reaction was then quenched with water (5 mL) and extracted with EtOAc (3×15 mL) and the combined organic phases were washed with brine (5 mL) and dried over sodium sulfate. After evaporation of the solvents (15 Torr), the resulting residue was purified by column chromatography (silica gel, hexane–EtOAc) affording the expected trityl ethers 1. When 1,9-nonanediol 2f was employed as starting material for the preparation of 1f, a mixture of 1f (47%) and double protected diol 1h (36%) was obtained, but they could easily be separated by column chromatography. For the preparation of the double tritylated compound 1j, the amounts of all the reagents and solvent used were double the ones indicated above except for diol 2j, from which 10 mmol were utilised again.

In some cases, the expected ethers 1 were obtained impure with some amount of triphenylmethanol (resulting from the hydrolysis of the excess of trityl chloride during the work-up) after the column chromatography. However, triphenylmethanol crystallised from the thick oil obtained upon standing for 24 h at r.t. Most of the oil was then separated from the crystals with a Pasteur pipette. The crystals of triphenylmethanol were washed with hexane (2×2 mL) and the liquid phases were mixed with the previously separated oil. Evaporation of the solvent gave pure compounds 1 without any trace of triphenylmethanol.

**n-Decyl Trityl Ether (1a)**

Colourless oil; yield: 94%; Rf 0.67 (hexane–EtOAc, 4:1).

IR (neat): 3086, 3058, 3022, 1597, 1490 (HC=C), 1088, 1071 cm⁻1 (CO).

1H NMR: δ = 0.88 (t, 3 H, J = 6.8 Hz, Me), 1.24–1.30 (30 H, CH2), 1.57–1.66 (m, 2 H, CH2,CHO), 3.03 (t, 2 H, J = 6.7 Hz, CH2O), 7.19–7.31 (m, 9 H, ArH), 7.43–7.46 (6 H, ArH).

13C NMR: δ = 14.1 (Me), 22.7, 26.5, 29.35, 29.5, 29.6 (2 C), 30.05, 31.9 [Me(CH2)3], 63.7 (CHO), 86.25 (CO), 126.75 (3 C), 127.65 (6 C), 128.7 (6 C), 144.55 (3 C, ArC).

MS: m/z (%) = 402 (M⁺ + 2, <1), 401 (M⁺ + 1, <4), 400 (M⁺, 12), 323 (16), 244 (36), 243 (100), 185 (35), 165 (39), 105 (40).

**2-Trityloxyoctane (1b)**

Colourless oil; yield: 59%; Rf 0.69 (hexane–EtOAc, 4:1).

IR (neat): 3083, 3058, 3032, 1598, 1501 (HC=C), 1071 cm⁻1 (CO).

1H NMR: δ = 0.84 (t, 3 H, J = 7.2 Hz, CH2CH2), 0.86 (d, 3 H, J = 5.9 Hz, CH2CH2), 1.07–1.25 [m, 10 H, (CH3)3], 3.46–3.61 (m, 1 H, CHO), 7.14–7.45 (m, 9 H, ArH), 7.46–7.55 (6 H, ArH).

13C NMR: δ = 14.05 (CH2CH2), 21.15 (CH2CHO), 22.55 (CH2), 24.95 (CH2), 29.35 (CH3), 31.75 (CH3), 37.5 (CH3), 70.05 (CHO), 88.05 (CO), 126.7 (3 C), 127.55 (6 C), 129.05 (6 C), 145.65 (3 C, ArC).

MS: m/z (%) = 373 (M⁺ + 1, <1), 372 (M⁺, 4), 244 (30), 243 (100), 183 (39), 165 (48), 105 (43).

HRMS: m/z calc for C28H54O: 372.2453; found, 372.2446.

**Cyclohexyl Trityl Ether (1c)**

White solid; yield: 50%; mp 85 °C; Rf 0.76 (hexane–EtOAc, 4:1).

IR (KBr): 3059, 3020, 1603, 1489 (HC=C), 1058 cm⁻1 (CO).

1H NMR: δ = 1.71 (m, 6 H, 3 × CH2), 2.31 (m, 4 H, 2 × CH2), 3.57 (m, 1 H, CHO), 7.23–7.54 (9 H, ArH), 7.55–7.80 (6 H, ArH).

13C NMR: δ = 24.25 (2 C), 25.7, 33.85 (2 C) (5 × CH2), 77.4 (CHO), 86.4 (CO), 126.75 (3 C), 127.55 (6 C), 120.0 (6 C), 145.65 (3 C, ArC).

MS: m/z (%) = 342 (M⁺ + 2, 100), 244 (25), 243 (100), 183 (16), 165 (39), 105 (20).

**Benzyl Trityl Ether (1d)**

White solid; yield: 90%; mp 95 °C; Rf 0.84 (hexane–EtOAc, 4:1).

IR (KBr): 3059, 3053, 3031, 3023, 1594, 1489 (HC=C), 1085, 1060 cm⁻1 (CO).

1H NMR: δ = 4.18 (s, 2 H, CH2), 7.17–7.44 (m, 14 H, ArH), 7.47–7.58 (6 H, ArH).

Geranyl Trityl Ether (1e)\textsuperscript{22}

White solid; yield: 36%; mp 91 ºC; R\textsubscript{f} 0.65 (hexane–EtOAc, 4:1).

1\textsubscript{3}C NMR: δ = 144.5 (3 C, ArC).

IR (neat): 3086, 3058, 3032, 264, 144.4 (3 C, ArC).

\[\text{HRMS: m/z (%) = 588 (M}^+ − 1), 244 (70), 243 (100), 165 (56), 105 (28).\]


1,9-Di(trityloxy)nonane (1j)

Colourless oil; yield: >99%; R\textsubscript{f} 0.60 (hexane–EtOAc, 4:1).

IR (neat): 3183, 3085, 3058, 3032, 1908, 1060 cm\textsuperscript{-1} (CO).

\[\text{HRMS: m/z (%) = 344 (M}^+ − 1), 243 (10), 165 (12), 102 (100), 58 (90).\]

Diethyl(4-trityloxypentyl)amine (1l)

Colourless oil; yield: 23%; R\textsubscript{f} 0.72 (hexane–EtOAc, 4:1).

IR (neat): 3183, 3085, 3058, 3032, 1908, 1060 cm\textsuperscript{-1} (CO).

\[\text{HRMS: m/z (%) = 401 (M}^+ − 1), 243 (15), 165 (27), 158 (60), 86 (100).\]

1-Methoxy-9-trityloxynonane (1g)

White solid; yield: 36%; mp 122 ºC; R\textsubscript{f} 0.2 (hexane–EtOAc, 1:1).

1\textsubscript{3}C NMR: δ = 11.2–1.42 (m, 10 H, (CH\textsubscript{2})\textsubscript{10}OTr), 3.32 (s, 3 H, Me), 7.12–7.31 (m, 18 H, ArH), 7.34–7.53 (m, 12 H, ArH).

\[\text{HRMS: m/z (%) = 401 (M}^+ − 1), 243 (15), 165 (27), 158 (60), 86 (100).\]

HRMS: m/z calc for C\textsubscript{32}H\textsubscript{40}NO, 435.2315; found, 435.2400.

1,4-Di(trityloxy)pentane (1j)

White solid; yield: 36%; mp 122 ºC; R\textsubscript{f} 0.63 (hexane–EtOAc, 4:1).

IR (KBr): 3083, 3052, 3022, 1598, 1500 (HC=O), 1081, 1075 cm\textsuperscript{-1} (CO).

1\textsubscript{3}C NMR: δ = 1.25 (d, 3 H, J = 6.1 Hz, MeCO), 1.00–1.68 (m, 4 H, (CH\textsubscript{2})\textsubscript{2}CO), 2.70–2.94 (m, 2 H, CH\textsubscript{2}O), 3.15–3.60 (m, 1 H, CHO), 7.12–7.31 (m, 18 H, ArH), 7.34–7.53 (m, 12 H, ArH).

\[\text{HRMS: m/z (%) = 388 (M}^+ − 1), 244 (40), 243 (100), 183 (12), 165 (42), 105 (17).\]

\[\text{HRMS: m/z calc for C\textsubscript{44}H\textsubscript{50}O, 644.3654; found, 644.3574.}\]
Reductive Cleavage of Trityl Ethers 1 via Naphthalene-Catalysed Lithiation to Give Alcohols 2; General Procedure

A solution of trityl ether 1 (1.0 mmol) in THF (2 mL) was added dropwise to a green suspension of lithium powder (70 mg, 10.0 mmol) and naphthalene (26 mg, 0.2 mmol) in THF (5 mL), under Ar, at the temperature indicated in Table 1. The reaction mixture turned a dark red colour after the addition of a few drops of the solution of 1. After stirring for the time indicated in Table 1, water (5 mL) was carefully added, the cooling bath was removed, and the reaction was stirred till it reached r.t. For entries 7 and 11 in Table 1, the hydrolysis was performed at 0 °C. The mixture was extracted with EtOAc (3 × 15 mL) and the combined organic phases were washed with brine (5 mL), being then dried over sodium sulfate. After evaporation of the solvents (15 Torr), the resulting residue was passed through a short column of silica gel, affording the products in yields indicated in Table 1. Compounds 2a–2f and 2i–2l (commercially available) were characterised by comparison of their physical and spectroscopic data with authentic samples.

Compounds 1f and 1i were deprotonated with n-BuLi (0.69 mL of 1.6 M solution in hexane, 1.1 mmol) at 0 °C, before submitting them to reductive cleavage.

For the reductive cleavage of ethers 1c, 1j, 1k, and 11, the reactions were hydrolysed with MeOH (5 mL) instead of water and the yields of the detritylated products were determined by quantitative GLC, using commercially available alcohols (2c, 2j, 2k, and 2l respectively) and n-dodecane (internal standard) in the determination of response factors.

9-Methoxy-1-nonanol (2g)

Colourless oil; yield >99%; Rf 0.31 (hexane–EtOAc, 6:4).

IR (neat): 3385 (OH), 1086, 1030 cm⁻¹ (CO).

1H NMR: δ = 1.19–1.42 [m, 10 H, (CH₂)₂CO], 1.46–1.47 [m, 4H, (CH₃)₂CO], 1.70 (br s, 1 H, OH), 3.33 (s, 3 H, Me), 3.36 (t, 2 H, J = 6.6 Hz, CH₂OMe), 3.63 (t, 2 H, J = 6.6 Hz, CH₂OMe).

13C NMR: δ = 25.7, 29.3, 29.35, 29.5, 29.55, 32.7, 38.5 [(CH₃)₂CO], 58.5 (Me), 62.95 (CH₂OMe), 72.9 (CH₂OMe).

MS: m/z (%) = 124 (M⁺ – 18 – 32, <1), 68 (11), 67 (14), 55 (22), 45 (100), 41 (42).

HRMS: m/z calc'd for C₁₇H₃₆O₂: 299.2929, found, 299.2928.

Acknowledgement

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