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Use of 1,2-dichloro 4,5-dicyanoquinone (DDQ) for cleavage of the 2-naphthylmethyl (NAP) group

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Abstract

The 2-naphthylmethyl (NAP) group is a versatile group for protection of hydroxyl functions. It is stable to 4% TFA in $CHCl_3$, hot 80% HOAc–H_2O, $SnCl_2$–AgOTf and HCl–EtOH, but it can readily be removed with DDQ in CH_2Cl_2. © 1999 Elsevier Science Ltd. All rights reserved.

Over the past decade, we have witnessed tremendous progress in the synthesis of complex carbohydrates.[1] A wide variety of glycosyl donors carrying different functionalities at their anomeric centers, for example, thio,[2] fluoride,[3] imidate,[4] sulfoxide,[5] and pentenyl, [6] together with an array of highly efficient catalysts[1] have proven to be the driving force behind such rapid and sustained progress in saccharide synthesis. At the same time, the availability of diverse protection for hydroxy groups also played an important role,[7] particularly during sequential syntheses of both linear and branched higher saccharides. Nonetheless, there is always the need for novel protecting groups for hydroxy functions that are compatible with anticipated reaction conditions. The NAP group was recently introduced by Esko et al.[8a] and Spencer et al.[8b] as a protecting group for polyhydroxy systems. Therefore, it was of interest to further explore the utility of the NAP group in the synthesis of target oligosaccharide structures. Interestingly, the NAP group, unlike its *p*-methoxybenzyl (PMB) counterpart which can be cleaved in hot acetic acid[9], was remarkably stable under those conditions usually employed for acetal cleavage. Alkylation[10] of diacetone glucose with naphthyl bromide (NAPBr) gave the 3-*O*-NAP derivative **1**, which upon treatment with HCl in ethanol[11] provided 3-*O*-NAP glucose **2** in good yield. The latter compound on acetylation gave **3**. However, when 1,2:5,6-di-*O*-isopropylidene-3-*O*-PMB-α-D-glucose was similarly treated, its PMB protecting group was partially cleaved. Similarly, compound **12** was obtained by treatment of compound **11** with a 4% solution of TFA in $CHCl_3$. By contrast, the PMB protecting group was cleaved with a 0.5% solution of TFA in dichloromethane (Table 1).[12,13]

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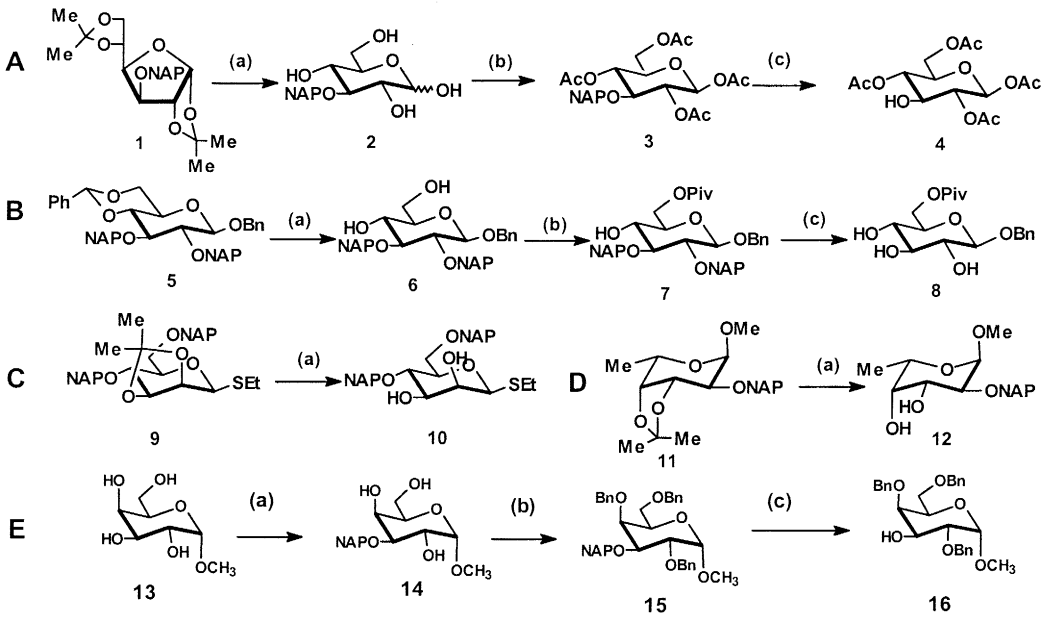
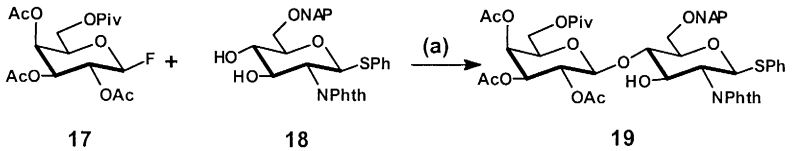
Table 1
Demonstration of NAP removal by DDQ



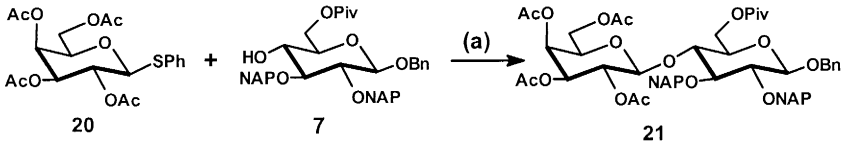
Reagents and conditions:**A**: a) HCl (0.8M)-EtOH, 55 °C 4 h ; b) Ac_2O-pyridine (1:1), 100 °C, 5 min, rt, 16 h, 60-70% ; c) DDQ (3.0eq), CH_2Cl_2 -MeOH (4:1)-H_2O (trace), 4 h, 87%. **B**: a) 60% HOAc, 70 °C,1.5 h, 80% ; b) PivCl-pyridine, 0 °C - rt, 12 h, 80%; c) DDQ (3.0eq),CH_2Cl_2-MeOH (4:1)-H_2O (trace), rt, 3.5 h, 88%. **C**: a) 60% HOAc, 60-65 °C, 1.5 h, 70%. **D**: a) TFA (4%) in $CHCl_3$, rt, 2 h, 75%; **E**: a) Bu_2SnO-benzene, refluxing, 4 h, then NAPBr, n-Bu_4NI, 80 -85 °C, 16 - 18 h, 93%; b) KOH, 18-crown-6, THF, rt, 30 - 40 min, then, BnBr, rt, 12 h, 63%c) DDQ (3.0 eq), CH_2Cl_2-MeOH (4:1), rt, 2 h, 80%.

The NAP group readily undergoes oxidative cleavage under conditions analogous to those employed for the removal of PMB.[14] Removal of NAP from **3** provided the known[11] 1,2,4,6-tetra-*O*-acetyl-β-D-glucopyranose **4**. Example **E** represents a facile procedure for the preparation of methyl 2,4,6-tri-*O*-benzyl-α-D-galactopyranoside **16**. It is noteworthy that the NAP group was stable under glycosylation conditions thus far examined by us. Recently, Wong and co-worker[15] reported that the PMB protecting group was cleaved in the presence of AgOTf. However, compound **17** was coupled with phenyl 6-*O*-naphthylmethyl-2-deoxy-2-phthalimido-1-thio-β-D-glucopyranoside **18** in the presence of $SnCl_2$–AgOTf as catalyst to give disaccharide **19** (Scheme 1). Similarly, phenyl 2,3,4,6-tetra-*O*-acetyl-1-thio-galactopyranoside **20** on reaction with acceptor **7** provided the disaccharide derivative **21** (Scheme 2). Reaction of galactosaminide **22** with thioglycosyl donor **20** gave **23** in fairly good yield.



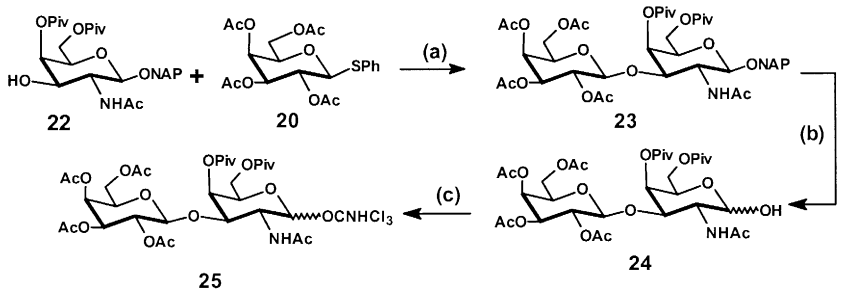
Scheme 1. (a) $SnCl_2$–AgoTf/CH_2Cl_2–toluene, −15°C to 0°C, N_2, 12 h, 74%

On removal of its anomeric NAP group, compound **23** gave **24** which has recently been reported as



Scheme 2. (a) NIS–TfOH (cat)/CH_2Cl_2, 4A-MS, −40°C to 25°C, 1 h, 67%

a precursor for glycosyl donor **25** that was utilized for the synthesis of oligosaccharides containing the Galβ1→3GalNAcβ-linkage (Scheme 3).[16]



Scheme 3. (a) NIS–TfOH/CH_2Cl_2, 4A-MS, −50°C, 2 h, 75%; (b) DDQ (3.0 equiv.)/CH_2Cl_2–MeOH (9:1), rt, 4 h, 76%; (c) Cl_3CN/DBU/CH_2Cl_2, rt, 3.5–4 h, 83%

The facile method for selective removal of the NAP group under such mild conditions is likely to encourage the use of this protecting group in synthetic carbohydrate chemistry.

1. Experimental

Typical experiment*:* Compound **15** (15.3 g, 25.0 mmol) in dichloromethane–methanol (150 ml, 4:1) was treated with DDQ (16.5 g, 73.3 mmol). The mixture was stirred at rt and the progress of reaction was monitored by TLC. After 2 h, when TLC showed that only a trace of **15** was detectable, the mixture was concentrated and the residue was taken up in dichloromethane. The solution was washed with aqueous $NaHCO_3$ (three times) and water, dried and concentrated. The concentrate was applied to a column of silica gel and eluted with hexane:ethyl acetate (1:1) to give compound **16** (9.5 g, 80%) as an oil.

The selective physical data for **1**, **3**, **7**, **15**, **16**, **18**, **19** and **21**. The structural assignment of **19** and **21** was based on 2D ^{1}H–^{1}H DQF-COSY and 2D ROESY spectroscopy. Compound **1**: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.83–7.79 (m, 4H, ArH), 7.48–7.45 (m, 3H, ArH), 5.93–5.92 (d, 1H, $J_{1,2}$=3.7 Hz, H-1), 4.83–4.80 (dd, 2H, J_{gem}=12.1 Hz, $OCH_2C_{10}H_7$, ABq), 4.63–4.61 (d, 1H, *J*=3.6 Hz), 4.51–4.35 (m, 1H), 4.20–4.00 (m, 4H), 1.49 (s, 3H, CH_3), 1.43 (s, 3H, CH_3), 1.39 (s, 3H, CH_3), 1.31 (s, 3H, CH_3); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 135.31, 133.46, 133.28, 128.40, 128.07, 127.90, 126.66, 126.35, 126.18, 125.84, 112.02, 109.24, 105.55 (C-1), 82.94, 81.90, 81.61, 72.76, 72.67, 67.67, 27.07 (CH_3), 27.03 (CH_3), 26.47 (CH_3), 25.69 (CH_3). Anal. calcd for $C_{23}H_{28}O_6$: C, 68.98, H, 7.05. Found: C, 68.71, H, 6.93. Compound **3**: (α+β): ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.82–7.80 (m, 6H, ArH), 7.72–7.68 (m, 2H, ArH), 7.52–7.44 (m, 4H, ArH), 7.40–7.32 (m, 2H, ArH), 6.33–6.32 (d, 1H, $J_{1,2}$=3.5 Hz, H-1, α-form), 5.67–5.65 (d, 1H, $J_{1,2}$=8.3 Hz, H-1, β-form), 5.25–5.09 (m, 4H), 4.88–4.78 (m, 3H, $OCH_2C_{10}H_7$), 4.25–4.19 (m, 2H), 4.18–3.99 (m, 4H), 3.84–3.78 (t, 1H, *J*=8.9 Hz), 3.78–3.72 (dq, 1H), 2.16 (s, 3H, Ac), 2.09 (s, 3H, Ac), 2.08 (s, 3H, Ac), 2.07 (s, 3H, Ac), 1.97 (s, 3H, Ac), 1.93 (s, 6H, 2Ac), 1.92 (s, 3H, Ac); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 170.91 (C=O), 170.88 (C=O), 169.76 (C=O), 169.46 (C=O), 169.38 (C=O), 169.26 (C=O), 168.93 (C=O), 135.60, 135.22, 133.43, 133.41, 133.21, 128.45,

128.38, 128.09, 128.02, 127.86, 126.73, 126.47, 126.37, 126.31, 126.25, 125.82, 125.64, 92.22, 89.72, 80.20, 77.23, 74.99, 74.46, 73.23, 71.83, 71.77, 70.50, 69.34, (2C), 62.02 (2C), 21.05 (Ac), 21.03 (Ac), 20.89 (Ac), 20.85 (Ac), 20.77 (Ac). Anal. calcd for $C_{25}H_{28}O_{10}$: C, 61.47, H, 5.78. Found: C, 61.43, H, 5.75. Compound **7**: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.90–7.70 (m, 8H, ArH), 7.50–7.20 (m, 11H, ArH), 5.20–5.10 (dd, 2H, OCH_2Ar), 5.00–4.90 (m, 3H, OCH_2Ar), 4.71–4.68 (d, 1H, J_{gem}=11.9 Hz, OCH_2Ar), 4.59–4.57 (d, 1H, $J_{1,2}$=7.0 Hz, H-1), 4.40–4.30 (m, 2H), 3.56–3.49 (m, 4H), 1.25 (s, 9H, *t*-Bu); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 174.30 (C=O), 136.06, 136.02, 128.71, 128.64, 128.34, 128.30, 128.15, 127.92, 127.89, 127.04, 127.02, 126.43, 126.35, 126.24, 126.19, 126.08, 126.03, 102.63 (C-1), 83.99, 82.07, 75.65, 75.61, 75.02, 73.89, 71.22, 70.61, 70.56, 63.66, 27.45 (CH_3). Anal. calcd for $C_{40}H_{42}O_7$: C, 75.69, H, 6.67. Found: C, 75.52, H, 6.60. Compound **11**: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.90–7.70 (m, 4H, ArH), 7.60–7.40 (m, 3H, ArH), 4.99–4.96 (d, 1H, J_{gem}=12.5 Hz, $OCH_AC_{10}H_7$, ABq), 4.90–4.86 (d, 1H, J_{gem}=13.2 Hz, $OCH_BC_{10}H_7$, ABq), 4.65–4.64 (d, 1H, $J_{1,2}$=3.9 Hz, H-1), 4.37–4.34 (dd, 1H), 4.11–4.00 (m, 3H), 3.57–3.55 (dd, 1H), 3.57 (s, 3H, OCH_3), 1.38 (s, 3H, CH_3), 1.34 (s, 3H, CH_3), 1.31–1.29 (d, 3H, *J*=6.7 Hz, CH_3); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 135.57, 133.03, 132.89, 127.89, 127.93, 127.63, 127.45, 126.60, 125.85, 125.74, 125.63, 108.55 (ketal carbon), 98.21 (C-1), 76.12, 75.99, 75.91, 72.25, 62.67, 55.24, 28.00 (CH_3), 26.21 (CH_3), 16.06 (CH_3). Compound **12**: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.88–7.80 (m, 4H, ArH), 7.56–7.40 (m, 3H, ArH), 5.00–4.70 (dd, 2H, $OCH_2C_{10}H_7$), 4.63–4.62 (d, 1H, $J_{1,2}$=3.6 Hz, H-1), 4.00–3.80 (m, 4H), 3.80–3.65 (m, 2H), 3.33 (s, 3H, OCH_3), 1.25–1.20 (d, 3H, *J*=6.7 Hz, CH_3); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 135.44, 133.00, 132.86, 127.95, 127.60, 127.35, 126.68, 125.90, 125.73, 125.71, 98.35, 76.31, 72.94, 71.80, 69.28, 65.33, 54.88, 15.65 (CH_3). Compound **15**: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.81–7.61 (m, 7H, ArH), 7.52–7.20 (m, 15H, ArH), 5.00–4.94 (d, 1H, J_{gem}=12.4 Hz, OCHAr), 4.94–4.80 (dd, 2H, OCH_2Ar), 4.80–4.60 (dd, 2H, OCH_2Ar), 4.56–4.50 (d, 1H, J_{gem}=12.6 Hz, OCHAr), 4.50–4.44 (dd, 2H, OCH_2Ar), 4.06–3.86 (m, 4H), 3.64–3.50 (m, 3H), 3.34 (s, 3H, OCH_3); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 133.50, 133.10, 128.55, 128.50, 128.38, 128.36, 128.25, 128.23, 128.08, 127.91, 127.85, 127.72, 126.30, 126.20, 126.11, 125.96, 125.87, 98.97, 79.21, 76.91, 75.50, 74.98, 73.69, 73.66, 73.56, 69.47, 69.29, 55.51. Anal calcd for $C_{39}H_{38}O_6$: C, 77.45, H, 6.67. Found: C, 77.37, H, 6.39. Compound **16**: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.40–7.20 (m, 15H, ArH), 4.86–4.76 (dd, 1H), 4.74–4.56 (m, 4H, $2OCH_2Ph$), 4.58–4.36 (dd, 2H, OCH_2Ph), 4.10–4.00 (m, 1H), 4.00–3.90 (m, 2H), 3.85–3.75 (m, 1H), 3.60–3.50 (m, 2H), 3.34 (s, 3H, OCH_3), 2.40–2.30 (br, 1H, OH); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 138.75, 138.39, 138.19, 128.69, 128.58, 128.55, 128.34, 128.32, 128.16, 127.90, 98.21 (C-1), 77.61, 76.90, 75.33, 73.65, 73.14, 70.48, 69.32, 69.26, 55.54. Compound **18**: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.85–7.75 (m, 4H, ArH), 7.37–7.35 (m, 5H, ArH), 7.25–7.24 (m, 7H, ArH), 5.64–5.61 (d, 1H, $J_{1,2}$=10.4 Hz, H-1), 4.78–4.60 (d, 1H, J_{gem}=12.4 Hz, $OCHC_{10}H_7$, ABq), 4.60–4.54 (d, 1H, J_{gem}=12.6 Hz, $OCHC_{10}H_7$, ABq), 4.26–4.24 (t, 1H, *J*=8.4 Hz, H-3), 4.15–4.13 (t, 1H, *J*=10.8 Hz, H-2), 3.90–3.87 (dd, 1H, *J*=11.6, 3.6 Hz, H-6a,), 3.81–3.80 (dd, 1H, *J*=11.6, 3.6 Hz, H-6b), 3.54–3.52 (m, 2H, H-4, H-5); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 135.49, 134.27, 133.41, 133.16, 132.66, 132.32, 133.22, 131.72, 129.01, 128.91, 128.35, 128.10, 127.84, 126.63, 126.25, 126.03, 125.87, 83.73 (C-1), 78.96, 73.79, 72.92, 72.72, 70.10, 55.79. Anal. calcd for $C_{31}H_{27}O_6NS$: C, 68.74, H, 5.02, N, 2.59, S, 5.92. Found: C, 68.61, H, 5.11, N, 2.37, S, 6.08. Compound **19**: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.86–7.80 (m, 6H, ArH), 7.68–7.64 (m, 2H, ArH), 7.52–7.40 (m, 5H, ArH), 7.28–7.16 (m, 3H, ArH), 5.65–5.63 (d, 1H, $J_{1,2}$=10.4 Hz, H-1), 5.32–5.31 (d, 1H, *J*=2.0 Hz, H′-4), 5.20–5.16 (t, 1H, *J*=10.0, 8.8 Hz, H′-2), 4.94–4.90 (dd, 1H, H′-3), 4.89–4.86 (d, 1H, *J*=12.0 Hz, $OCH_AC_{10}H_7$, ABq), 4.72–4.69 (d, 1H, *J*=12.0 Hz, $OCH_BC_{10}H_7$, ABq), 4.53–4.51 (d, 1H, *J*=8.0 Hz), 4.47–4.42 (t, 1H), 4.05–4.03 (m, 4H), 3.87–3.71 (m, 4H), 2.14–2.10 (s, 3H, Ac), 1.96 (s, 3H, Ac), 1.94 (2s, 6H, 2Ac); ^{13}C NMR ($CDCl_3$, 100.6 Hz) δ 178.50 (C=O), 170.50 (C=O), 170.00 (C=O), 169.50 (C=O), 169.20 (C=O), 134.50, 132.75, 129.00, 128.50, 128.20, 128.00, 127.80, 126.58, 126.30, 126.00, 123.58, 123.50, 100.50, 83.50, 81.00,

79.20, 72.00, 70.80, 70.20 (2C), 69.50, 69.00, 68.50, 60.50, 58.50, 20.68 (Ac), 20.62 (Ac), 20.35 (Ac), 20.40 (Ac). Anal. calcd for $C_{45}H_{45}O_{15}NS$: C, 68.54, H, 5.10, N, 2.59, S, 5.96. Found: C, 68.61, H, 5.11, N, 2.37, S, 6.08. Compound **21**: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.82–6.80 (m, 19H, ArH), 5.28–5.27 (d, 1H, J=2.7 Hz, H′-4), 5.24–5.18 (dd, 1H, H′-2), 5.16–5.00 (m, 3H, OCH_2Ph, $OCHC_{10}H_7$), 4.96–4.88 (d, 2H, H′-3, J_{gem}=12.4 Hz, $OCHC_{10}H_7$), 4.88–4.80 (d, 1H, J_{gem}=12.6 Hz, $OCHC_{10}H_7$), 4.76–4.72 (d, 1H, $J_{1,2}$=7.5 Hz, H′-1), 4.68–4.64 (d, 1H, J_{gem}=12.2 Hz, $OCHC_{10}H_7$), 4.58–4.48 (m, 2H, H-1, H-6b), 4.60–4.50 (dd, 1H, H-6a), 4.20–3.98 (dd, 1H, H′-6b), 3.90–3.78 (m, 2H, H-4, H′-6a), 3.74–3.62 (m, 2H, H-3, H′-5), 3.60–3.50 (m, 2H, H-5, H-2), 2.09 (s, 3H, Ac), 2.05 (s, 3H, Ac), 1.95 (s, 3H, Ac), 1.84 (s, 3H, Ac), 1.25 (s, 9H, *t*-Bu); ^{13}C NMR ($CDCl_3$, 100.6 Hz) δ 179.11 (C=O), 171.31 (C=O), 171.22 (C=O), 171.19 (C=O), 170.63 (C=O), 137.63, 134.52, 131.45, 129.74, 129.70, 129.32, 129.30, 129.20, 129.18, 129.14, 128.94, 128.86, 128.49, 128.42, 127.84, 127.70, 127.46, 127.42, 127.40, 127.35, 127.28, 127.16, 127.02, 126.94, 126.88, 126.82, 102.16, 101.12, 83.74, 83.65, 79.46, 76.29, 76.05, 74.07, 72.35, 72.29, 72.16, 70.94, 68.03, 67.90, 61.93, 29.80, 28.53 ($3CH_3$), 21.95 (Ac), 21.77 (Ac), 21.76 (Ac), 21.68 (Ac).

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13. In a preliminary experiment, methyl 3-*O*-(4-methoxybenzyl)-α-D-galactopyranoside **26** and its 3-*O*-naphthylmethyl analog **27** were both subjected to acetolysis conditions (Ac_2O-0.8% concd H_2SO_4). After 12 h at rt, the PMB group of **26** was completely cleaved, where the NAP group in **27** suffered only partial cleavage.
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