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Nucleophilic addition of 2-(trimethylsilyloxy)furan to a D-glucopyranose oxocarbenium ion

Anne Jégou and Alain Veyrières *

Laboratoire de Synthèses et Activations de Biomolécules, CNRS ESA 6052, Ecole Nationale Supérieure de Chimie de Rennes, Campus de Beaulieu, 35700 Rennes, France

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Abstract

$Cp_2Zr(ClO_4)_2$ was used as a promoter for nucleophilic addition of 2-(trimethylsilyloxy) furan (TMSOF) to 2,3,4,6-tetra-*O*-benzyl-D-glucopyranosyl fluoride to give a 2:1:1 mixture of adducts in 80% yield. NMR data and postulated transition states are consistent with a 3:1 ratio of regioisomers at C-5 and C-3 of TMSOF and a 2:1 ratio of *threo* and *erythro* α-adducts at C-5.

1. Introduction

The preparation of *C*-glycosides[1] as *O*-glycoside mimics or building blocks in total synthesis would be more advantageous if an exocyclic stereocentre could be created together with the formation of a carbon–carbon bond at the anomeric centre of the reacting carbohydrate species. Asymmetric functionalization of the added appendage would then follow more readily, giving access to extended carbon chains with a large number of stereogenic centres. One obvious approach is the addition of carbonyl compounds to anomeric carbanions. Because of easy β-elimination of C-2 substituents this method was restricted for a time to 2-deoxy sugars.[2] This difficulty was recently overcome[3] by an outstanding application of samarium chemistry; 1,2-*trans*-*C*-glycosides were usually obtained, unless internal chelation of the intermediate glycosyl samarium(III) compound became possible. An elegant synthesis of α-*C*-galactosamine derivatives has thus been recently reported.[4] Variable diastereoselectivities, often reaching a 5:1 ratio, have been observed at the newly created acyclic stereocentre.

2-(Trimethylsilyloxy)furan (TMSOF) usually undergoes electrophilic attack at C-5 and is therefore viewed as a stable synthon for the γ-anion of a butenolide.[5] Several reports concern its reactivity with five-membered ring oxocarbenium,[6] *N*-acyliminium[7] and oxazolidinium species.[8] Stereodifferentiation of one face in such cyclic ions is more or less achieved by the steric effect of a ring substituent located

* Corresponding author. Fax: +33 (2) 99 87 13 48.

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across the heteroatom. A low facial differentiation of TMSOF often results from a thermodynamic equilibrium between *erythro* and *threo* products where Lewis acid promoter, solvent and temperature play crucial roles. Six-membered ring oxocarbenium ions are known to add *C*-nucleophiles under efficient stereoelectronic control of the oxygen atom, D-glucopyranosyl derivatives affording α-*C*-glycosides with an excellent stereoselectivity.[9]

Herein we report our preliminary studies concerning the addition of TMSOF to a D-glucopyranosyl fluoride.

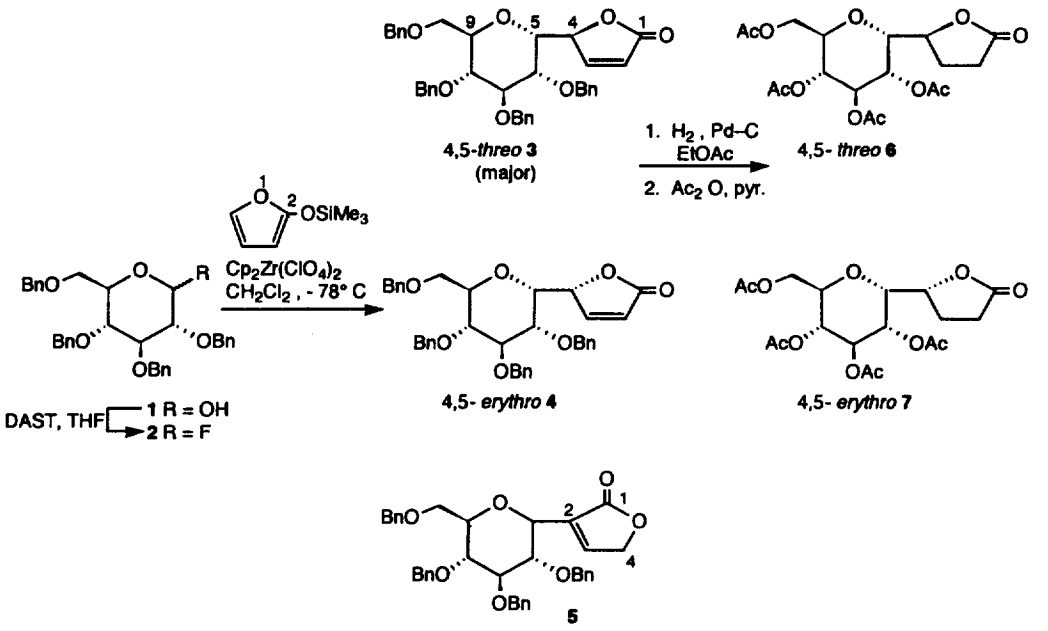
2. Results and discussion

Perbenzylated D-glucopyranosyl fluoride **2** (β:α 43:7) was quantitatively obtained by treatment of commercially available 2,3,4,6-tetra-*O*-benzyl-D-glucose **1** with (diethylamino)sulfur trifluoride (DAST) as reported by Posner and Haines.[10] It reacts with various nucleophiles such as allyl silanes or silyl enol ethers[11] usually in the presence of boron trifluoride etherate, and also aromatic rings[12] with zirconocene (or hafnocene) dichloride–silver perchlorate complexes as promoters. Zirconium and hafnium complexes are highly fluorophilic and are able to activate furanosyl and pyranosyl fluorides at low temperature to give an oxocarbenium species. The complex $[ZrCp_2(CF_3SO_3)_2$ THF] is also a fast and efficient catalyst for the Mukaiyama cross-aldol reaction involving the coupling of aldehydes and ketones with enol silanes.[13]

When fluoride **2** was allowed to react with TMSOF (3 equiv.) in dichloromethane at −78°C in the presence of Cp_2ZrCl_2–$2AgClO_4$ (1.5 equiv.), a 2:1:1 mixture of adducts **3**, **4** and **5** was obtained in 80% yield (Scheme 1). Work-up conditions had a dramatic effect upon the reaction yield; quenching with bases (triethylamine or $NaHCO_3$) induced partial decomposition of the products. Prolonged reaction times or raising the temperature above −40°C also led to transformation of the mixture components; new signals at δ 7.52 (2 dd, *J* 1.7 and 5.8 Hz) and 6.13 (2 dd, *J* 2.2 and 5.8 Hz) in the ^1H NMR spectrum were attributed to H-3 and H-2, respectively, of a 1:1 pair of *threo* and *erythro* β-adducts which were formed by thermodynamic equilibration following the opening of the pyranose ring by the Lewis acid. Unfortunately, the reaction was not clean and anomerized compounds could not be characterized. Repeated chromatography gave samples of **3** and **4** pure enough for NMR analysis. The vinylic hydrogen atom H-3 gave a signal at lower field (δ 7.71) in **4** than in **3** (δ 7.49). This downfield chemical shift (Δδ 0.22 ppm) has been observed in *erythro* butenolides.[14] Coupling constants $J_{7,8}$ 4.7 and $J_{8,9}$ 7.2 Hz are indicative of a distorted conformation of the pyranose ring in the *erythro* adduct **4**, whereas $J_{6,7}$ 9, $J_{7,8}$ 8.2 and $J_{8,9}$ 9.5 Hz correspond to a $^8C_5$ chair conformer (Fig. 1) in the major *threo* adduct **3**. The value of $J_{5,6}$ 6.2 Hz therefore confirms the α-configuration of **3**.

The third component **5** could not be isolated in a pure state, but NMR analysis revealed that it had only one vinylic hydrogen H-3 (δ 7.66), that C-2 (δ 130.18) is a disubstituted sp^2 carbon atom (C-2 gives signals at δ 121.66 and 121.35 in **3** and **4** respectively) and also that the butenolide ring contains a CH_2 group (δ 70.39). These data indicate that compound **5** arose from the coupling of an oxocarbenium ion at C-3 of the silyloxyfuran, followed by conjugation of the double bond.

Hydrogenation of the mixture of adducts, followed by *O*-acetylation allowed an easier separation of *threo*-**6** and *erythro*-**7** compounds (2:1, 61%). The major *threo* compound **6** has its pyranose ring in a $^8C_5$ chair conformation (Fig. 1) like its unsaturated precursor ($J_{6,7}$ 8.2, $J_{7,8}$ 7.7 and $J_{8,9}$ 8.6 Hz) with an α-*C*-linked butyrolactone appendage ($J_{5,6}$=5.8 Hz), whereas the *erythro* compound **7** has a flattened pyranose ring ($J_{5,6}$ 3.6, $J_{6,7}$=$J_{7,8}$ 5.7 and $J_{8,9}$ 5.4 Hz). Optical rotation values of *threo*-**6** and *erythro*-**7** are, respectively, +79 and +21, therefore following Hudson's lactone rule.[15]



Scheme 1.

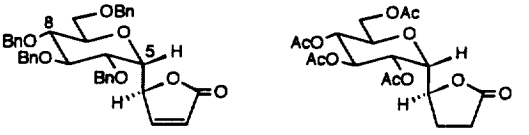


Fig. 1. Conformations of *threo* adducts **3** and **6**

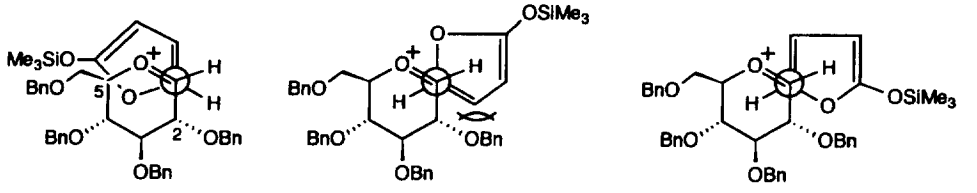


Fig. 2. Transition states in the addition of 2-(trimethylsilyloxy)furan to the 2,3,4,6-tetra-*O*-benzyl-D-glucopyranose oxocarbenium ion

As expected, addition of TMSOF occurred only on the α-face of the cyclic oxocarbenium ion because of the stereoelectronic control brought by an axial electron pair of the ring oxygen atom. The 2:1 ratio between *threo* and *erythro* adducts reflects a preferential Diels–Alder like transition state T_1 where the silyloxyfuran has favourable orbital overlap with the π-system of the oxocarbenium species.[14] Molecular models (Fig. 2) show that some steric hindrance arises from the proximity of H-5 and the trimethylsilyl group. An antiperiplanar transition state T_2 which would also lead to the *threo* adduct is less probable because of the steric influence of the 2-*O*-benzyl group. Transition state E leading to the *erythro* product shows no steric compression, but does not favour secondary orbital interactions. Formation of the C-3 substituted lactone (20%) arises from an alternative transition state less hindered than T_1. Noticeably, a similar yield of C-3 coupling has been reported[16] in the addition of TMSOF to a six-membered ring cationic species generated from a 3,5,6-trisubstituted 1,4-dioxan.

Other D-glucopyranosyl derivatives were tried: the β-pentaacetate did not react with TMSOF and

$BF_3 \cdot OEt_2$ even at room temperature. 2,3,4,6-Tetra-*O*-benzyl-D-glucose **1** was also activated without success by formation of an α,β-trifluoroacetate. Its activation by an α,β-acetate allowed coupling with TMSOF in the presence of catalytic $SnCl_4$–$AgClO_4$ in ether at 0°C to give a 3:2 mixture of *threo* and *erythro* adducts in a 60% yield and complete regioselectivity; the catalytic cycle[17] was erratic, however, even on a millimolar scale. No reaction was observed in the presence of trityl perchlorate in ether. Finally, the fluoride **2** gave a 64% yield of adducts (*threo*:*erythro*=3:2) in a $BF_3 \cdot OEt_2$ promoted reaction in dichloromethane.

Reactions with a D-mannopyranosyl fluoride where the axial substituent at C-2 will not interfere in the transition state, as well as the use of more cumbersome silyl derivatives, might lead to enhanced regio- and diastereoselectivities.

3. Experimental

3.1. General procedures

Optical rotations were measured at room temperature on a Perkin–Elmer 341 automatic polarimeter (concentration in g/100 mL). NMR spectra were recorded on a Bruker ARX-400 instrument. ^1H NMR were obtained at 400.13 MHz (s=singlet, d=doublet, m=multiplet). Assignments were confirmed by homonuclear 2D COSY correlated experiments. ^{13}C NMR were obtained at 100.62 MHz in the proton-decoupled mode. Heteronuclear 2D correlated spectra were recorded in order to assist in carbon resonance assignments. Chemical shifts are given in ppm relative to internal TMS (δ scale) and coupling constants (*J*) in hertz. Thin layer chromatography (TLC) was performed on Merck DC-Alufolien Kieselgel 60 F_{254} art. 5554 with detection by UV light and charring with H_2SO_4:EtOH (1:10). Flash chromatography[18] was performed on Merck Kieselgel 60 (40–63 mm). All solvents were dried and distilled according to standard laboratory procedures.[19] Elemental analyses were performed by the Service de Microanalyse du Centre National de la Recherche Scientifique (Gif-sur-Yvette, France).

3.2. 2,3,4,6-Tetra-O-benzyl-D-glucopyranosyl fluoride 2

To a solution of 2,3,4,6-tetra-*O*-benzyl-D-glucopyranose **1** (1 g, 1.85 mmol, Fluka) in dry tetrahydrofuran (5 mL) was added at −30°C under nitrogen (diethylamino)sulfur trifluoride (DAST, 290 μL, 2.21 mmol). The mixture was kept for 30 min at room temperature, then cooled to −30°C and treated with methanol (2 mL). The solution was concentrated and the residue was taken up in dichloromethane. The extract was washed with saturated aqueous $NaHCO_3$, then brine, dried ($MgSO_4$) and evaporated. The residue was purified by flash chromatography (petroleum ether:ethyl acetate=9:1) to give **2** (0.96 g, 96%) as a syrup. ^1H NMR ($CDCl_3$): δ 5.54 (dd, 0.14 H, $J_{1,2}$ 2.5, $J_{1,F}$ 53.2 Hz, H-1β) and 5.25 (dd, 0.86 H, $J_{1,2}$ 6.6, $J_{1,F}$ 52.8 Hz, H-1α).

3.3. 5,9-Anhydro-6,7,8,10-tetra-O-benzyl-2,3-dideoxy-D-erythro-L-ido- 3 and D-erythro-L-gulo- 4 dec-2-enonic acid γ-lactone

Silver perchlorate monohydrate (629 mg, 2.79 mmol) was added in one portion to a mixture of zirconocene dichloride (408 mg, 1.4 mmol) and 3 Å molecular sieves (2 g) in dichloromethane (15 mL) at −20°C under nitrogen. The mixture was stirred for 30 min at −20°C, then cooled to −78°C. A solution of fluoride **2** (507 mg, 0.93 mmol) and 2-(trimethylsilyloxy)furan (470 μL, 2.79 mmol) in dichloromethane

(15 mL) was cooled to −78°C under nitrogen, then added dropwise with a double tip needle to the solution of zirconocene bis(perchlorate). The mixture was slowly heated to −40°C. Water (1 mL) was added and the mixture was filtered over Celite. The filtrate was washed with brine, dried ($MgSO_4$) and concentrated. The residue was purified by flash chromatography (petroleum ether:EtOAc=7:3) to give a 2:1:1 mixture of adducts **3**, **4** and **5** (456 mg, 80%). Anal. calcd for $C_{38}H_{38}O_7$: C, 75.23; H, 6.31. Found: C, 75.36; H, 6.37. Further chromatography gave a sample of **4**, R_f 0.30; ^{1}H NMR ($CDCl_3$): δ 7.71 (dd, 1H, $J_{2,3}$ 5.7, $J_{3,4}$ 1.6 Hz, H-3), 7.35–7.18 (m, 20H, 4Ph), 6.11 (dd, 1H, $J_{2,4}$ 2 Hz, H-2), 5.34 (ddd, 1H, $J_{4,5}$ 7.4 Hz, H-4), 4.65 (s, 2H, CH_2Ph), 4.59 and 4.47 (2d, 2H, J 11.6 Hz, CH_2Ph), 4.54 and 4.46 (2d, 2H, J 11.7 Hz, CH_2Ph), 4.53 and 4.47 (2d, 2H, J 12.3 Hz, CH_2Ph), 4.07 (ddd, 1H, $J_{8,9}$ 7.2, $J_{9,10a}$ 5.3, $J_{9,10b}$ 3.8 Hz, H-9), 3.88–3.83 (m, 3H, H-5, 6, 7), 3.64 (dd, 1H, $J_{10a,10b}$ 10.5 Hz, H-10a), 3.60 (dd, 1H, H-10b) and 3.58 (dd, 1H, $J_{7,8}$ 4.7 Hz, H-8); ^{13}C NMR ($CDCl_3$): δ 173.01 (C-1), 156.52 (C-3), 138.06, 137.93, 137.65 and 137.56 (4C quat. arom.), 128.49–127.67 (20C arom.), 121.35 (C-2), 81.24, 77.25, 75.80, 75.34, 74.22 and 73.21 (C-4, 5, 6, 7, 8, 9), 73.31, 73.18, 72.90 and 72.78 (4CH_2Ph) and 68.78 (C-10).

Then was eluted a sample of **3**, R_f 0.21; ^{1}H NMR ($CDCl_3$): δ 7.49 (dd, 1H, $J_{2,3}$ 5.8, $J_{3,4}$ 1.6 Hz, H-3), 7.34–7.13 (m, 20H, 4Ph), 6.10 (dd, 1H, $J_{2,4}$ 2.2 Hz, H-2), 5.48 (ddd, 1H, $J_{4,5}$ 5.2 Hz, H-4), 4.89 and 4.84 (2d, 2H, J 11 Hz, CH_2Ph), 4.83 and 4.58 (2d, 2H, J 11.9 Hz, CH_2Ph), 4.78 and 4.49 (2d, 2H, J 11.1 Hz, CH_2Ph), 4.56 and 4.42 (2d, 2H, J 12.1 Hz, CH_2Ph), 4.09 (dd, 1H, $J_{6,7}$ 9, $J_{7,8}$ 8.2 Hz, H-7), 4.02 (dd, 1H, $J_{5,6}$ 6.2 Hz, H-5), 3.83 (dd, 1H, H-6), 3.76 (ddd, 1H, $J_{8,9}$ 9.5, $J_{9,10a}$ 3.3, $J_{9,10b}$ 2.4 Hz, H-9), 3.67 (dd, 1H, H-8), 3.63 (dd, 1H, $J_{10a,10b}$ 10.8 Hz, H-10a) and 3.58 (dd, 1H, H-10b); ^{13}C NMR ($CDCl_3$): δ 172.64 (C-1), 155.26 (C-3), 138.39, 138.12, 137.83 and 137.59 (4C quat. arom.), 128.63–127.66 (20C arom.), 121.66 (C-2), 82.12, 81.88, 78.29, 77.26, 74.81 and 74.09 (C-4, 5, 6, 7, 8, 9), 75.22, 74.60, 74.13 and 73.40 (4CH_2Ph) and 68.52 (C-10).

Overlapping fractions showed the presence of **5**; ^{1}H NMR ($CDCl_3$): δ 7.66 (dd, 1H, J 1.7 and 3.3 Hz, H-3); ^{13}C NMR ($CDCl_3$): δ 172.46 (C-1), 149.63 (C-3), 130.18 (C-2), 79.30 (C-5) and 70.39 (C-4).

*3.4. 6,7,8,10-Tetra-*O*-acetyl-5,9-anhydro-2,3-dideoxy-*D*-erythro-*L*-ido-* **6** *and* D*-erythro-*L*-gulo-* **7** *deconic acid γ-lactone*

A solution of adducts **3**, **4** and **5** (360 mg, 0.59 mmol) in ethyl acetate (10 mL) was hydrogenated for 3 h at room temperature and atmospheric pressure in the presence of 10% palladium on charcoal (100 mg). The mixture was filtered over Celite and the catalyst was thoroughly washed with methanol. The combined filtrate and washings were concentrated and the residue was taken up with dichloromethane (2 mL). Acetic anhydride (1.12 mL, 12 mmol), pyridine (0.97 mL, 12 mmol) and 4-dimethylaminopyridine (a few mg) were successively added. The mixture was stirred for 5 h at room temperature, then diluted with dichloromethane, washed with aqueous 0.5 M hydrochloric acid, saturated aqueous sodium hydrogencarbonate and water, dried ($MgSO_4$) and concentrated. The residue was purified by flash chromatography (petroleum ether:EtOAc=1:1) to give a mixture of **6** and **7** (151 mg, 61%). Further chromatography gave pure **7** (47 mg); R_f 0.14; $[\alpha]_D$ +21 (*c* 0.986, $CHCl_3$); ^{1}H NMR ($CDCl_3$): δ 5.29 (dd, 1H, $J_{6,7}$=$J_{7,8}$ 5.7 Hz, H-7), 5.03 (dd, 1H, $J_{5,6}$ 3.6 Hz, H-6), 4.89 (dd, 1H, $J_{8,9}$ 5.4 Hz, H-8), 4.71 (ddd, 1H, $J_{4,5}$ 7.4 Hz, H-4), 4.49 (m, 1H, H-10a), 4.11 (dd, 1H, H-5), 4.08–4.00 (m, 2H, H-9, 10b), 2.64–2.46 (m, 2H, H-2a, 2b), 2.38–2.19 (m, 2H, H-3a, 3b), 2.12, 2.11, 2.10 and 2.08 (4s, 12H, 4OAc); ^{13}C NMR ($CDCl_3$): δ 176.17 (C-1), 170.61, 169.61, 169.46 and 169.04 (4CH_3CO), 76.98 (C-4), 72.78 (C-9), 70.89 (C-5), 68.13 (C-7), 67.92 (C-6), 66.97 (C-8), 60.89 (C-10), 27.48 (C-2), 24.08 (C-3), 20.79, 20.77, 20.75 and 20.74 (4CH_3CO). Anal. calcd for $C_{18}H_{24}O_{11}$: C, 51.92; H, 5.81. Found: C, 52.03; H, 5.96.

Then were successively eluted a mixture of **6** and **7** (24 mg) and pure **6** (80 mg); R_f 0.10; $[\alpha]_D$ +79 (*c* 1, $CHCl_3$); ^1H NMR ($CDCl_3$): δ 5.47 (dd, 1H, $J_{6,7}$ 8.2, $J_{7,8}$ 7.7 Hz, H-7), 5.23 (dd, 1H, $J_{5,6}$ 5.8 Hz, H-6), 5.00 (dd, 1H, $J_{8,9}$ 8.6 Hz, H-8), 4.72 (ddd, 1H, $J_{3a,4}$~$J_{3b,4}$~7.7, $J_{4,5}$ 3.4 Hz, H-4), 4.26 (dd, 1H, $J_{9,10a}$ 5.6, $J_{10a,10b}$ 12.1 Hz, H-10a), 4.14 (ddd, 1H, $J_{9,10b}$ 2.6 Hz, H-9), 4.10 (dd, 1H, H-5), 4.08 (dd, 1H, H-10b), 2.68–2.52 (m, 2H, H-2a, 2b), 2.35–2.21 (m, 2H, H-3a, 3b), 2.12 and 2.10 (2s, 6H, 2OAc) and 2.06 (s, 6H, 2OAc); ^{13}C NMR ($CDCl_3$): δ 175.89 (C-1), 170.65, 169.89, 169.66 and 169.50 ($4CH_3CO$), 77.98 (C-4), 72.42 (C-9), 72.10 (C-5), 70.45 (C-7), 69.08 (C-6), 68.03 (C-8), 62.07 (C-10), 28.35 (C-2), 24.24 (C-3), 20.81, 20.74, 20.72 and 20.70 ($4CH_3CO$). Anal. calcd for $C_{18}H_{24}O_{11}$: C, 51.92; H, 5.81. Found: C, 51.82; H, 5.91.

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