STEREOSPECIFIC SYNTHESIS OF (–)-*allo*-MUSCARINE FROM D-GLUCOSE: NOVEL ROUTES TO THE KEY CHIRAL SYNTHON

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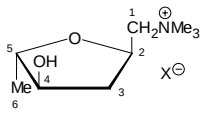
The key chiral synthon in a novel synthesis of (–)-*allo*-muscarine from D-glucose has been prepared by three independent routes. The most efficient one includes a four-step conversion *via* the 4-*O*-benzoyl derivatives of starting 2,5-anhydro-3,5-di-*O*-methanesulfonyl-L-idose ethylene acetal (**2a**) into 2,5-anhydro-3,6-dideoxy-L-*lyxo*-hexose ethylene acetal (**4b**). The intermediate **4b** was efficiently converted into the chiral synthon 2,5-anhydro-4-*O*-benzoyl-3,6-dideoxy-L-*arabino*-hexose (**4c**) by Mitsunobu reaction.

Key words: 2,5-Anhydro sugars; D-Glucose; (–)-*allo*-Muscarine; 3,6-Thioanhydro sugars.

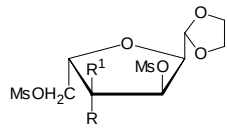
(–)-*allo*-Muscarine (**1**) is a C-2 epimer of (+)-muscarine which occurs in the mushroom *Amanita muscaria*[1], and shows cholinomimetic activity[2]. There is a renewed interest in the muscarinic field due to the discovery of a relationship between cholinergic deficits and the pathology of Alzheimer's disease[3]. The synthesis of muscarine and many of its analogues have been reviewed[4], but only few syntheses of (–)-*allo*-muscarine (**1**) have been achieved so far[5]. Recently we have completed a stereospecific synthesis of **1** based on D-glucose as a chiral precursor[6]. However, due to some relatively low-yield steps in the reported route[6], new efforts have been made in order to improve the preparation of the key chiral intermediate **4c** and (–)-*allo*-muscarine (**1**) itself. We now report two independent routes, one *via* intermediate **3b** and second *via* **4b** with three alternative in **4b** preparation, towards the key chiral synthon in an alternative synthesis of (–)-*allo*-muscarine from D-glucose.

Ethylene acetal of 2,5-anhydro-3,5-di-*O*-methanesulfonyl-L-idose (**2a**) which is readily available from D-glucose[7] was treated with triflic anhydride in pyridine and dichloromethane whereupon the corresponding 4-triflate **2b** was obtained in 93% yield. The earlier findings[8] that some sugar triflates when reacted with sodium nitrite in *N*,*N*-dimethylformamide gave the corresponding *epi*-hydroxy compounds prompted us to investigate the possible use of this reagent for conversion of **2b** into the corresponding

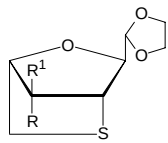
alcohol with inverted configuration. A treatment of **2b** with sodium nitrite in *N*,*N*-dimethylformamide afforded the corresponding 2,5-anhydro-L-altrose derivative **2c** in a yield of only 29%. Reaction of **2c** with benzoyl chloride in pyridine gave the corresponding 4-*O*-benzoyl derivative **2d**, which was further treated with sodium hydrogen sulfide in *N*,*N*-dimethylformamide to afford the bicyclic oxathiane derivative **3b** (25% from **2c**). The intermediate **3b** was alternatively also prepared by direct sodium hydrogen sulfide mediated cyclization of **2c** to alcohol **3a** which was subsequently benzoylated to give **3b** (38% from **2c**). Raney nickel desulfurization of **3b** afforded the chiral synthon **4c** (67%) with all chiral centers corresponding to (–)-*allo*-muscarine (**1**).



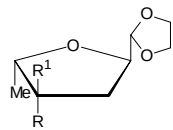
(-)-*allo*-Muscarine (**1**)
X = OH or Halogen



2	R	R^1
a	OH	H
b	OTf	H
c	H	OH
d	H	OBz
e	OMs	H
f	OBz	H



3	R	R^1
a	H	OH
b	H	OBz
c	OMs	H
d	OH	H
e	OBz	H



4	R	R^1
a	OBz	H
b	OH	H
c	H	OBz

Ms = CH_3SO_2, Tf = CF_3SO_2

Although the ^1H and ^{13}C NMR data as well as the optical rotation of **4c** were in good agreement with those already reported[6], the relatively low overall yield of the present route (6.9% from **2a**) prompted a further study directed towards preparation of **4c** by an alternative synthetic sequence *via* the 3,6-dideoxy derivative **4b** as a key intermediate.

Successive inter- and intramolecular attack of hydrogen sulfide anion on trimesylate[6] **2e** led to the bicyclic oxathiane derivative **3c**, which was immediately treated with sodium methoxide in methanol to afford the corresponding alcohol **3d** (33% from **2e**). Raney nickel desulfurization of **3d** gave the expected 3,6-dideoxy derivative **4b** in an overall yield of 27% with respect to trimesylate **2e**. However, when the intermediate **3d**

was prepared directly from **2a** (by its treatment with NaSH in DMF), the same intermediate **4b** was obtained in an overall yield of 35% related to starting compound **2a**.

Finally, the best overall yield of desired intermediate **4b** has been achieved by alternative chemical transformations of dimesylate **2a** under the conditions similar to those already reported[7]. Treatment of **2a** with benzoyl chloride in pyridine gave the corresponding 4-*O*-benzoyl derivative **2f**, which was immediately treated with sodium hydrogen sulfide in *N*,*N*-dimethylformamide to give the expected oxathiane derivative **3e**. Raney nickel desulfurization of **3e** to the corresponding 3,6-dideoxy derivative **4a** was followed by the subsequent debenzoylation of **4a** to **4b**. All four steps concerning the conversion of **2a** to **4b** were carried out successively, whereupon the intermediates **2f**, **3e**, and **4a** were used in the subsequent steps without any purification. Pure product **4b** was isolated by column chromatography in an overall yield of 63% related to the starting compound **2a**.

Alcohol **4b** readily reacted with triphenylphosphine, diethyl azodicarboxylate and benzoic acid, under standard Mitsunobu[9] conditions, to afford the chiral synthon **4c** in 82% yield.

In conclusion, the sequence which uses the crude 4-*O*-benzoyl derivatives (**2f**, **3e**, and **4a**) as intermediates is the most convenient route towards the key chiral intermediate **4c** since it provides the highest overall yield of the final product (52% from **2a**) achieved by a simple preparative procedure. This result represents a significant improvement in respect to the reported route[6] which afforded the chiral synthon **4c** in a much lower overall yield[6] (23% in respect to the same starting compound **2a**).

Since (–)-*allo*-muscarine has already been obtained[6] from 4-*O*-benzoyl derivative **4c**, the chemical transformations described in this paper formally represent an alternative synthesis of (–)-*allo*-muscarine from D-glucose.

EXPERIMENTAL

Melting points were determined on a Büchi SMP 20 apparatus and were not corrected. Optical rotations were measured on a Perkin–Elmer 141 MC polarimeter at 23 °C in chloroform solutions. NMR spectra (^{1}H at 250 MHz and ^{13}C at 62.9 MHz) were recorded on a Bruker AC 250 E instrument in deuteriochloroform. Chemical shifts are expressed in ppm (δ-scale) downfield from tetramethylsilane. Coupling constants (*J*) are given in Hz. Column chromatography (Kieselgel 60 under 0.063 mm; Merck) and flash column chromatography (ICN Silica 32-63) were carried out with the following solvent mixtures: Petroleum ether–Me_2CO, 4 : 1 (S1); CH_2Cl_2–Me_2CO, 9 : 1 (S2); 49 : 1 (S3), 99 : 1 (S4), 4 : 1 (S5); Et_2O (S6); toluene–Me_2CO, 49 : 1 (S7), 4 : 1 (S8); $CHCl_3$ (S9); hexane–EtOAc, 1 : 1 (S10); toluene–EtOAc, 4 : 1 (S11); CH_2Cl_2–EtOAc, 49 : 1 (S12). All organic extracts were dried with anhydrous Na_2SO_4. Organic solutions were concentrated in a rotary evaporator under diminished pressure at bath temperature 30–35 °C.

2,5-Anhydro-3,6-di-*O*-methanesulfonyl-4-*O*-trifluoromethanesulfonyl-L-idose Ethylene Acetal (**2b**)

To a stirred and ice-cooled solution of dimesylate **2a** (2.6 g; 7.2 mmol) in dry pyridine (3.6 ml; 44.7 mmol) and dichloromethane (60 ml) was added trifluoromethanesulfonic anhydride (2.6 ml; 15.5 mmol) in

portions. The mixture was stirred at 0 °C for 1 h then diluted with dichloromethane (60 ml), washed successively with aqueous 10% hydrochloric acid (50 ml) and water (4 × 100 ml), dried and evaporated. Flash chromatography (S1) of the residue gave pure **2b** (3.3 g; 93%) as a white solid. Recrystallization from methanol afforded an analytical sample **2b** as colourless needles, m.p. 94 °C, $[\alpha]_D$ –1.2° (*c* 0.5). ^{1}H NMR spectrum: 3.10 s and 3.17 s, 2 × 3 H (2 × CH_3SO_2); 3.89–4.1 m, 4 H (dioxolane CH_2); 4.25 dd, 1 H, *J*(1,2) = 5.3, *J*(2,3) = 4.7 (H-2); 4.34 dd, 1 H, *J*(6a,6b) = 11.1, *J*(5,6a) = 6.3 (H-6a); 4.49 dd, 1 H, *J*(5,6b) = 5.5 (H-6b); 4.74 ddd, 1 H, *J*(4,5) = 4.2, *J*(5,6a) = 6.3, *J*(5,6b) = 5.5 (H-5); 5.14 d, 1 H (H-1); 5.3 dd, 1 H, *J*(2,3) = 4.7, *J*(3,4) = 2.5 (H-3); 5.62 dd, 1 H (H-4). ^{13}C NMR spectrum: 37.39 and 38.03 (2 × CH_3SO_2), 64.97 (C-6), 65.24 and 65.37 (dioxolane CH_2), 76.38 (C-5), 78.71 (C-2), 80.12 (C-3), 86.6 (C-4), 100.99 (C-1), 118.11 q, ^1J(C,F) = 320 (CF_3SO_2). For $C_{11}H_{17}F_3O_{12}S_3$. CH_3OH (526.5) calculated: 27.38% C, 4.02% H, 18.27% S; found: 27.23% C, 3.76% H, 18.23% S.

2,5-Anhydro-3,6-di-*O*-methanesulfonyl-L-altrose Ethylene Acetal (**2c**)

To a solution of triflate **2b** (1.8 g; 3.6 mmol) in *N*,*N*-dimethylformamide (20 ml) was added finely powdered sodium nitrite (4.0 g; 58.0 mmol). The mixture was stirred for 24 h at 35 °C, then filtered and concentrated *in vacuo*. Column chromatography (S2) of the residue yielded pure **2c** (0.38 g; 29%) as colorless oil. The oily residue was crystallized from dichloromethane–hexane to give an analytical sample of **2c** as colorless needles, m.p. 116 °C, $[\alpha]_D$ +0.8° (*c* 0.2). ^{1}H NMR spectrum: 3.08 s and 3.18 s, 2 × 3 H (2 × CH_3SO_2); 3.46 d, 1 H, *J*(4,OH) = 7.4 (OH); 3.88–4.08 m, 4 H (dioxolane CH_2); 4.11 dd, 1 H, *J*(1,2) = 5.5, *J*(2,3) = 4.2 (H-2); 4.18 m, 1 H, *J*(5,6a) = 3.4, *J*(5,6b) = 2.6 (H-5); 4.37 dd, 1 H, *J*(5,6a) = 3.4, *J*(6a,6b) = 11.6 (H-6a); 4.4 m, 1 H, *J*(3,4) = 4.5 (H-4); 4.47 dd, 1 H, *J*(5,6b) = 2.6, *J*(6a,6b) = 11.6 (H-6b); 5.13 d, 1 H, *J*(1,2) = 5.5 (H-1); 5.21 dd, 1 H, *J*(2,3) = 4.2, *J*(3,4) = 4.5 (H-3). ^{13}C NMR spectrum: 37.52 and 38.89 (2 × CH_3SO_2), 65.27 and 65.46 (dioxolane CH_2), 68.34 (C-6), 70.92 (C-4), 79.07 (C-2), 80.29 (C-5), 80.91 (C-3), 101.60 (C-1). For $C_{10}H_{18}O_{10}S_2$ (362.4) calculated: 33.15% C, 5.01% H, 17.66% S; found: 32.78% C, 4.71% H, 17.23% S.

2,5-Anhydro-4-*O*-benzoyl-3,6-di-*O*-methanesulfonyl-L-altrose Ethylene Acetal (**2d**)

To a solution of hydroxy derivative **2c** (0.48 g; 1.3 mmol) in dry pyridine (5 ml) was added benzoyl chloride (0.50 ml; 4.3 mmol). The mixture was stored at room temperature for 24 h, then acidified with aqueous hydrochloric acid (1 : 1; 15 ml) and extracted with dichloromethane (4 × 10 ml). The extracts were combined, successively washed with water and saturated aqueous sodium hydrogen carbonate, dried and concentrated to an oil. Flash chromatography (S6) of the residue gave **2d** (0.5 g; 81%) as a colorless syrup, $[\alpha]_D$ –12.9° (*c* 0.2). ^{1}H NMR spectrum: 3.09 s and 3.11 s, 2 × 3 H (2 × CH_3SO_2); 3.86–4.15 m, 4 H (dioxolane CH_2); 4.16 dd, 1 H, *J*(1,2) = 6.6, *J*(2,3) = 3.2 (H-2); 4.45 dd, 1 H, *J*(6a,6b) = 11.2, *J*(5,6a) = 3.2 (H-6a); 4.49–4.69 m, 2 H, *J*(5,6b) = 2.2, *J*(4,5) = 8.4 (H-5 and H-6b); 5.21 d, 1 H (H-1); 5.44 t, 1 H, *J*(2,3) = 3.2, *J*(3,4) = 3.4 (H-3); 5.61 dd, 1 H, *J*(3,4) = 3.4, *J*(4,5) = 8.4 (H-4); 7.41–8.20 m, 5 H (ArH). ^{13}C NMR spectrum: 37.33 and 38.7 (2 × CH_3SO_2), 65.01 and 65.2 (dioxolane CH_2), 68.14 (C-6), 70.96 (C-4), 77.4 (C-5), 79.63 (C-2), 80.00 (C-3), 101.41 (C-1), 128.38, 129.96 and 133.60 (ArC), 165.50 (C=O).

2,5-Anhydro-3,6-thioanhydro-L-mannose Ethylene Acetal (**3a**)

To a solution of dimesylate **2c** (0.145 g; 0.40 mmol) in *N*,*N*-dimethylformamide (3 ml) was added NaSH monohydrate (0.15 g; 2.0 mmol). The mixture was stirred in an atmosphere of nitrogen at 90 °C for 1.5 h, then poured into aqueous 10% $NaHCO_3$ (10 ml) and extracted with CH_2Cl_2 (4 × 10 ml). The combined extracts were washed with water (2 × 20 ml), dried and evaporated to a brown oil.

Flash chromatography (S3) of the residue (0.05 g) yielded pure product **3a** (0.039 g; 48%) as a pale-yellow syrup, $[\alpha]_D$ –256.4° (*c* 0.1). ^{1}H NMR spectrum: 2.89 dd, 1 H, *J*(6a,6b) = 10.9, *J*(5,6a) = 1 (H-6a); 2.98 dd, 1 H, *J*(5,6b) = 2.4, *J*(6a,6b) = 10.9 (H-6b); 3.14 d, 1 H, *J*(3,4) = 1.3 (H-3); 3.88–4.12 m, 4 H (dioxolane CH_2); 4.38 m, 2 H (H-4 and H-5); 4.58 d, 1 H, *J*(1,2) = 2.3 (H-2); 4.78 d, 1 H, *J*(4,OH) = 9.9 (OH); 5.05 d, 1 H (H-1). ^{13}C NMR spectrum: 32.44 (C-6), 45.43 (C-3), 65.35 and 65.56 (dioxolane CH_2), 79.20 (C-4), 80.25 (C-5), 88.53 (C-2), 102.37 (C-1).

2,5-Anhydro-4-*O*-benzoyl-3,6-thioanhydro-L-mannose Ethylene Acetal (**3b**)

A) Treatment of 4-*O*-benzoyl derivative **2d** (0.44 g; 0.94 mmol) with NaSH monohydrate (0.35 g; 4.7 mmol) in *N*,*N*-dimethylformamide (10 ml) for 4 h, under the same reaction conditions as described above for **3a**, afforded crude **3b** (0.4 g). Column chromatography (40 g; S7) gave pure product **3b** (0.09 g; 31%) as a pale-yellow syrup.

B) Following the procedure described above for **2d**, a solution of **3a** (0.045 g; 0.22 mmol) and benzoyl chloride (0.10 ml; 0.86 mmol) in dry pyridine (1 ml) gave crude product **3b**. Flash chromatography (S9) afforded pure product **3b** (0.054 g; 79%) as a bright-yellow syrup, $[\alpha]_D$ –39.6° (*c* 2.1). ^{1}H NMR spectrum: 3.02 dd, 1 H, *J*(6a,6b) = 10.8, *J*(5,6a) = 1 (H-6a); 3.11 dd, 1 H, *J*(5,6b) = 2.3, *J*(6a,6b) = 10.8 (H-6b); 3.64 d, 1 H, *J*(3,4) = 1.5 (H-3); 3.68–4.02 m, 4 H (dioxolane CH_2); 4.33 d, 1 H, *J*(1,2) = 7.2 (H-2); 4.79 m, 1 H (H-5); 5.09 d, 1 H, *J*(1,2) = 7.2 (H-1); 5.44 d, 1 H, *J*(3,4) = 1.5 (H-4); 7.35–8.06 m, 5 H (ArH). ^{13}C NMR spectrum: 32.82 (C-6), 44.83 (C-3), 64.91 and 65.34 (dioxolane CH_2), 78.17 (C-5), 79.77 (C-4), 89.99 (C-2), 102.96 (C-1), 128.63, 129.44, 129.77 and 133.53 (ArC), 165.84 (C=O).

2,5-Anhydro-3,6-thioanhydro-L-talose Ethylene Acetal (**3d**)

A) Treatment of trimesylate[6] **2e** (3.47 g; 7.9 mmol) with NaSH monohydrate (4.58 g; 61.9 mmol) in *N*,*N*-dimethylformamide (23 ml) for 3 h under the same reaction conditions as described above for **3a** afforded crude oxathiane **3c** (1.2 g) which was immediately treated with 1.6 M solution of NaOMe in MeOH (15 ml) for 3 h at reflux temperature. The reaction mixture was acidified with glacial AcOH to pH 6 and concentrated. The residue was partitioned between CH_2Cl_2 (15 ml) and water (20 ml). The organic layer was separated and aqueous solution extracted with CH_2Cl_2 (2 × 15 ml). The combined extracts were dried and evaporated to a yellow syrup. Column chromatography (100 g; S4) of the residue afforded pure product **3d** (0.45 g; 33%) as a colorless syrup.

B) Treatment of dimesylate **2a** (2.5 g; 6.9 mmol) with NaSH monohydrate (2.5 g; 33.78 mmol) in *N*,*N*-dimethylformamide (30 ml) for 24 h, under the same reaction conditions as described above for **3a** yielded crude product **3d**. Column chromatography (140 g; S10) of the residue (1.4 g) yielded pure product **3d** (0.6 g; 43%) as a colorless syrup, $[\alpha]_D$ –24.5° (*c* 1.2). ^{1}H NMR spectrum: 2.68 d, 1 H, *J*(4,OH) = 10.3 (OH); 3.00 m, 2 H (2 H-6); 3.38 d, 1 H, *J*(3,4) = 2.4 (H-3); 3.83–4.05 m, 4 H (dioxolane CH_2); 4.2 d, 1 H, *J*(1,2) = 4.4 (H-2); 4.34 m, 1 H (H-5); 4.74 m, 1 H (H-4); 4.80 d, 1 H (H-1). ^{13}C NMR spectrum: 34.1 (C-6), 49.6 (C-3), 64.9 and 65.1 (dioxolane CH_2), 73.5 (C-4), 77.5 (C-5), 87.5 (C-2), 102.5 (C-1).

2,5-Anhydro-3,6-dideoxy-L-*lyxo*-hexose Ethylene Acetal (**4b**)

A) A suspension of compound **3d** (1.08 g; 5.29 mmol) and Raney nickel (10 ml) in ethanol (30 ml) was hydrogenated at 85–90 °C and normal pressure of H_2 for 1 h. The mixture was diluted with a mixture of EtOAc–EtOH (1 : 1, 60 ml), filtered through a Celite pad and the catalyst was washed with mixture of EtOAc–EtOH (1 : 1, 80 ml). The filtrate and washing were combined and evaporated *in vacuo*. The residue was treated with boiling CH_2Cl_2 (20 ml), then filtered and the solvent evaporated.

Column chromatography (80 g; S5) of the residue (0.9 g) afforded pure compound **4b** (0.76 g; 82%) as a colorless syrup.

B) A solution of alcohol **2a** (0.50 g; 1.4 mmol) and benzoyl chloride (0.40 ml; 3.4 mmol) in dry pyridine (5 ml) was left at room temperature for 24 h. After the usual workup, the residue (0.85 g of crude **2f**) was treated with NaSH monohydrate (0.6 g; 8.1 mmol) in *N*,*N*-dimethylformamide (10 ml) for 3 h, according to the procedure described above for **3a**. The crude oxathiane **3e** (0.5 g) thus obtained was dissolved in ethanol (15 ml) and hydrogenated in the presence of Raney nickel suspension (5 ml) at 80 °C for 1 h, whereupon the corresponding 3,6-dideoxy derivative **4a** was formed. To the reaction mixture was then added NaOH (0.07 g) and the stirring continued for 0.5 h at 80 °C. After workup as described above (procedure *A*), the remaining crude **4b** (0.248 g) was purified by column chromatography (30 g; S8) to afford pure product **4b** (0.151 g; 63%) as a colorless syrup, $[\alpha]_D$ +19.5° (*c* 1.9). ^{1}H NMR spectrum: 1.22 d, 3 H, *J*(5,6) = 6.4 (Me-5); 2.05 m, 2 H (2 H-3); 2.26 bs, 1 H (OH); 3.84–4.04 m, 5 H (dioxolane CH_2 and H-5); 4.17 ddd, 1 H, *J*(4,5) = 3, *J*(3a,4) = 1.9, *J*(3b,4) = 4.2 (H-4); 4.19 ddd, 1 H, *J*(1,2) = 4.2, *J*(2,3a) = 7.5, *J*(2,3b) = 8.2 (H-2); 4.84 d, 1 H, *J*(1,2) = 4.2 (H-1). ^{13}C NMR spectrum: 14.0 (C-6), 36.2 (C-3), 65.1 and 65.4 (dioxolane CH_2), 73.6 (C-4), 77.0 (C-2), 78.9 (C-5), 104.8 (C-1).

2,5-Anhydro-4-*O*-benzoyl-3,6-dideoxy-L-*arabino*-hexose (**4c**)

A) To a solution of compound **3b** (0.054 g; 0.17 mmol) in EtOH (5 ml) was added a suspension of Raney nickel (1 ml). The mixture was hydrogenated under the same reaction conditions as described above for **4b** (procedure *A*). After the usual workup, the remaining crude **4c** was purified by column chromatography (10 g; S7) to afford pure product **4c** (0.032 g; 67%) as a colorless syrup.

B) To a stirred and ice-cooled solution of alcohol **4b** (0.20 g; 1.2 mmol), benzoic acid (0.30 g; 2.5 mmol) and triphenylphosphine (1.30 g; 5.0 mmol) in dry THF (20 ml) was added dropwise a solution of diethyl azodicarboxylate (1.0 ml; 6.4 mmol) in dry THF (5 ml). The mixture was stirred at 0 °C for 20 min and then at room temperature for 20 h. The solvent was evaporated off and the residue was column chromatographed (100 g; S11), to afford **4c** contamined with an equal amount of aromatic impurities as estimated by NMR. Repeated column chromatography (50 g; S12) of the mixture yielded pure product **4c** (0.25 g; 82%) as a colorless syrup, $[\alpha]_D$ –4.2° (*c* 2.8). ^{1}H NMR spectrum: 1.29 d, 3 H, *J*(5,6) = 6.7 (Me-5); 2.15 ddd, 1 H, *J*(3a,3b) = 14, *J*(2,3a) = 3.7, *J*(3a,4) = 3.6 (H-3a); 2.61 ddd, 1 H, *J*(2,3b) = 7, *J*(3a,3b) = 14, *J*(3b,4) = 6.8 (H-3b); 3.86–4.07 m, 4 H (dioxolane CH_2); 4.13 ddd, 1 H, *J*(1,2) = 5.4, *J*(2,3a) = 3.7, *J*(2,3b) = 7 (H-2); 4.36 m, 1 H (H-5); 5.0 d, 1 H, *J*(1,2) = 5.4 (H-1); 5.15 ddd, 1 H, *J*(3a,4) = 3.6, *J*(3b,4) = 6.8, *J*(4,5) = 3.7 (H-4); 7.30–8.10 m, 5 H (ArH). ^{13}C NMR spectrum: 18.64 (C-6), 32.57 (C-3), 65.23 and 65.4 (dioxolane CH_2), 77.95 (C-2), 79.34 (C-4), 80.02 (C-5), 104.59 (C-1), 128.36, 129.58, 129.75 and 133.11 (ArC), 166.1 (C=O).

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