ChemPubSoc Europe

DOI: 10.1002/open.201500164

Open Access ChemistryOPEN Communications

Synthesis of the Heptasaccharide Repeating Unit of the Cell Wall O-Polysaccharide of Enterotoxigenic *Escherichia coli* O139

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Enterotoxigenic *Escherichia coli* (ETEC) like the O139 strain are mostly responsible for traveler's diarrhea and causes diseases in pigs, cattle, and poultry. A convenient synthetic strategy was developed for the synthesis of the heptasaccharide repeating unit of the cell wall lipopolysaccharide of the *E. coli* O139 strain. The *p*-methoxybenzyl (PMB) group was used as a temporary protecting group which was removed in situ under the glycosylation conditions by changing the reaction temperature during the synthesis of the target compound. All glycosylation steps gave high yields with good stereoselectivity. A (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO)-mediated selective oxidation of the primary hydroxyl group was carried out using a biphasic reaction condition at the late stage of the synthesis. Such synthetic oligosaccharides could later be effectively conjugated with proteins to prepare glycoconjugate derivatives as vaccine candidates.

Diarrheal diseases are quite common worldwide and are the leading cause of death in children and immunocompromised patients.[1] Even though gastrointestinal problems are more common in developing countries due to the lack of adequate sanitation and improved public health,[2] they are still important clinical problems in the developed countries.[3] The major causes of diarrheal outbreaks are food-borne infections caused by the *Salmonella* species,[4] *Campylobactor jejuni*,[5] enteropathogenic *Escherichia coli* (*E. coli*),[6] and waterborne infections by several *Giardia*[7] and *Cryptosporidium* species.[8] Among several bacterial species responsible for the enteric infections, pathogenic *E. coli* strains are predominant.[9] Although, *E. coli* colonize the gut of the host as useful commensal organisms,[10] they often acquire virulence factors and become responsible for several infectious diseases such as sepsis and meningitis,[11] urinary tract infections,[12] and gastrointestinal disorders.[9] The diarrhea-causing *E. coli* strains are divided into several subgroups based on their pathogenic mode of actions,[13] such as (a) enteropathogenic *E. coli* (EPEC), (b) enteroinvasive *E. coli* (EIEC). (c) enterohemorrhagic *E. coli* (EHEC), (d) enteroaggregative *E. coli* (EAEC), (e) enterotoxigenic *E. coli* (ETEC), etc. Among these virulent strains of *E. coli*, ETEC are mostly responsible for the traveler's diarrhea and causes diseases in pigs, cattle, and poultry.[14] After adhering to the intestine of the host, they have the ability to secrete heat-labile and heat-stable enterotoxins, which are the key factors of the diarrheal infections[15]. *E. coli* O139 strain belongs to the ETEC subgroup, and it has

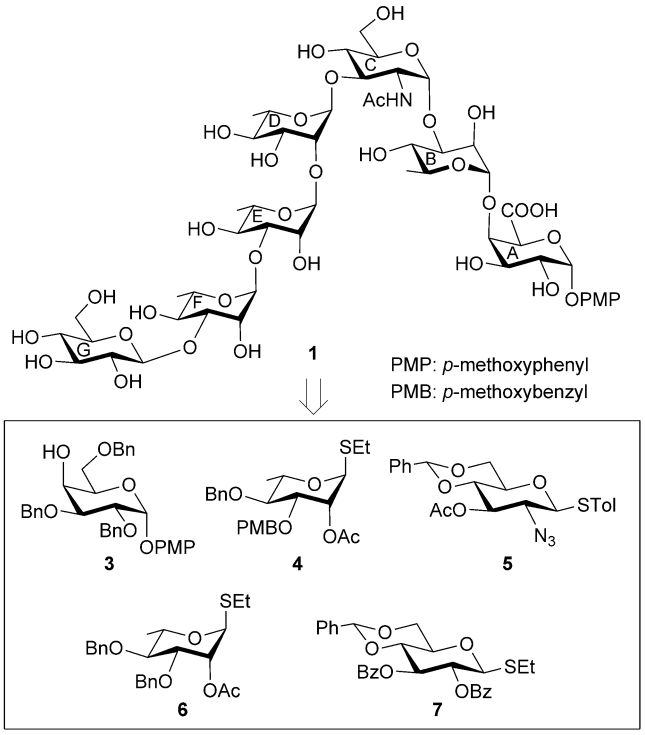
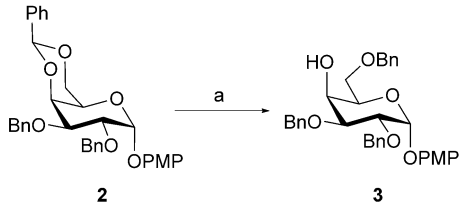


Figure 1. Structure of the synthesized heptasaccharide **1** as its *p*-methoxyphenyl glycoside and its synthetic intermediates.



Scheme 1. *Reagents and conditions*: a) Et_3SiH, I_2, CH_3CN, 5 °C, 1 h, 83 %.

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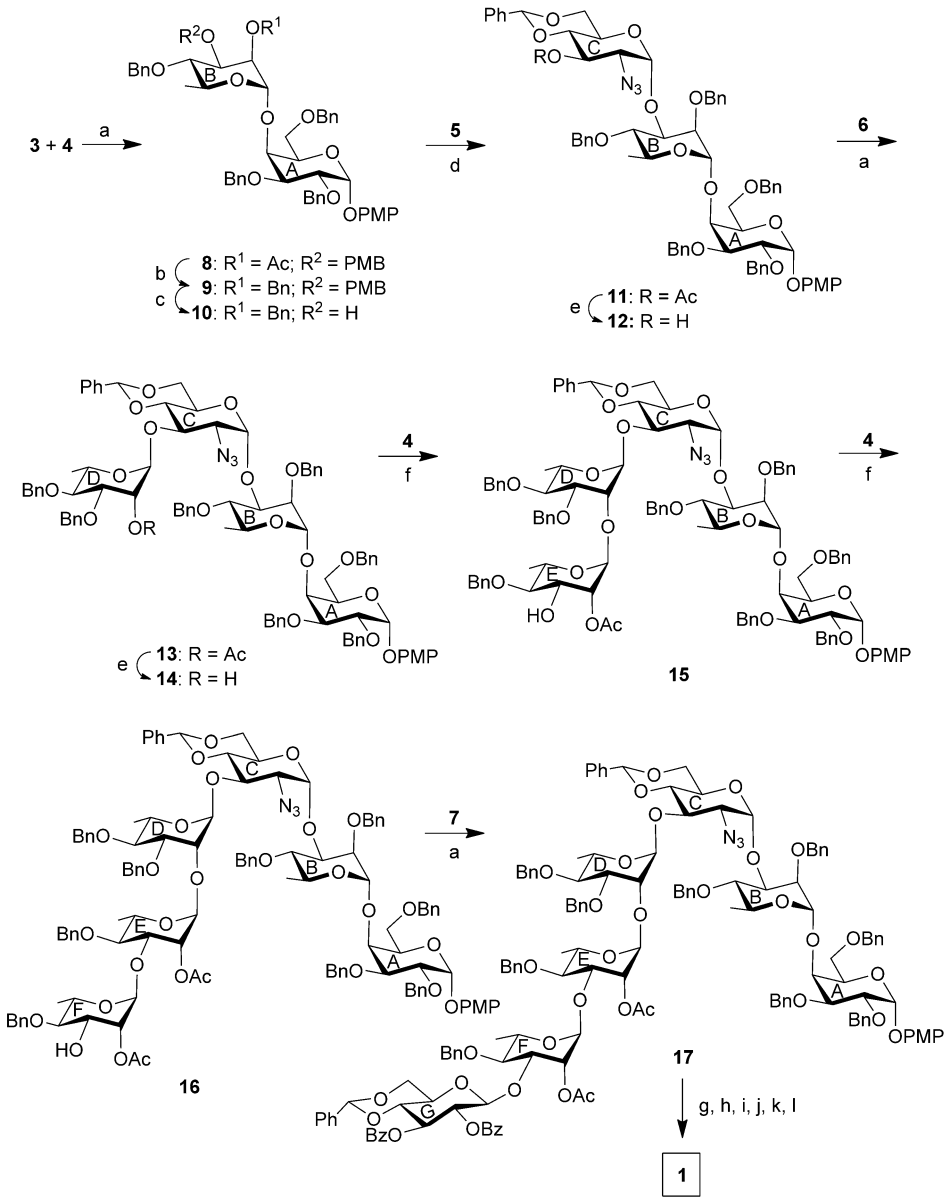
Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/open.201500164.



Scheme 2. *Reagents and conditions*: a) NIS, $HClO_4/SiO_2$, mol. sieves (4 Å), CH_2Cl_2, −20 °C, 45 min, 83% for compound **8**, 86% for compound **13**, 73% for compound **17**; b) benzyl bromide, NaOH, TBAB, THF, rt, 2 h, 77%; c) DDQ, CH_2Cl_2/H_2O (1:1), 5 °C, 2 h, 72%; d) NIS, $HClO_4/SiO_2$, mol. sieves (4 Å), CH_2Cl_2/Et_2O (1:3), −10 °C, 1 h, 74%; e) 0.1 M CH_3ONa, CH_3OH, rt, 1 h, 92% for compound **12**, 90% for compound **14**; f) NIS, $HClO_4/SiO_2$, mol. sieves (4 Å), CH_2Cl_2, −30 °C for 45 min, then rt for 30 min, 72% for compound **15**, 70% for compound **16**; g) AcSH, pyridine, rt, 24 h; h) 80% aq. AcOH, 80 °C, 2 h; i) acetic anhydride, pyridine, rt, 2 h; j) Et_3SiH, $Pd(OH)_2/C$ (20%), CH_3OH, rt, 18 h; k) 1) NaBr, TBAB, TEMPO, $NaHCO_3$, NaOCl, CH_2Cl_2, H_2O, 0–5 °C, 3 h, 2) *t*-butanol, 2-methyl-but-2-ene, $NaClO_2$, NaH_2PO_4, rt, 3 h; l) CH_3ONa (0.1 M), CH_3OH, rt, 5 h, 44% overall.

been isolated from pigs and poultry suffering from diarrhea.[16] Because of their well-known functions in the initial stage of bacterial infections, several structures of the bacterial cell wall polysaccharides or O-antigens have been characterized in the past.[17–19] The structure of the repeating unit of the O-polysaccharide of *E. coli* O139 has been reported by Marie et al.,[20] which is a heptasaccharide containing D-glucose, D-glucosamine, L-rhamnose, and D-galacturonic acid moieties in the ratio 1:1:4:1.

The development of effective vaccines against infectious diseases has become a thrust area of research due to the emergence of multidrug-resistant bacteria.[21] Conventionally, polysaccharides isolated from the bacterial cell wall have been used in the preparation of several vaccines[22–24] with a number of limitations, such as handling of live bacterial strains, tedious isolation of polysaccharides with adequate purity and free from biological impurities, batch-to-batch variation of the antigenic epitopes of the polysaccharides, etc. Hence, development of the chemical synthetic strategies for the preparation of oligosaccharides corresponding to the repeating unit of the polysaccharides could be the best possible approach to overcome the difficulties mentioned earlier. The synthetic oligosaccharides with precise chemical structures could be effectively conjugated with proteins to furnish glycoconjugate derivatives for their evaluation as vaccine candidates.[25,26] In an ongoing program for the synthesis of oligosaccharides corresponding to the cell wall polysaccharides of diarrheagenic *E. coli* strains,[27] a concise synthesis of the heptasaccharide repeating unit of the O-polysaccharide of *E. coli* O139 strain is reported herein.

The heptasaccharide **1** as its *p*-methoxyphenyl (PMP) glycoside has been synthesized using a synthetic strategy containing a number of one-pot reaction conditions for the glycosylations and protective group manipulations. A number of suitably functionalized monosaccharide intermediates **3**, **4**,[28] **5**,[29] **6**,[30] and **7**[31] were prepared from the commercially available reducing sugars using the reaction conditions available in the literature (Figure 1). Similar reaction conditions for the stereoselective glycosylations have been used in several occasions in the synthetic scheme by judicious selection of the protecting groups in the glycosyl donor and acceptor components. The *p*-methoxybenzyl (PMB) group has been used as a temporary protecting group in the glycosyl donor **4**, which has been re-

moved in situ under the glycosylation conditions after the formation of the glycosidic linkages.[32] The *p*-methoxyphenyl (PMP) group present in the reducing end of the synthesized heptasaccharide **1** can be oxidatively removed[33] to get the heptasaccharide hemiacetal for its conjugation to a suitable protein through a spacer linkage using standard reaction conditions.[34] Perchloric acid supported over silica gel ($HClO_4/SiO_2$) has been used as a user-friendly, solid acid catalyst[35] in the glycosylation reactions during the synthesis of the target compound.

Treatment of the literature-known *p*-methoxyphenyl 2,3-di-*O*-benzyl-4,6-*O*-benzylidene-α-D-galactopyranoside (**2**)[36] with triethylsilane in the presence of iodine[37] furnished regioselectively ring-opened glycosyl acceptor **3** in 83% yield (Scheme 1).

Stereoselective glycosylation of compound **3** with L-rhamnosyl thioglycoside **4** in the presence of a combination[38] of *N*-iodosuccinimide (NIS) and $HClO_4/SiO_2$ furnished exclusively disaccharide derivative **8** in 83% yield. The stereochemistry at the glycosidic bonds in compound **8** was confirmed from the NMR spectral analysis. Treatment of compound **8** with benzyl bromide in the presence of sodium hydroxide afforded compound **9** in 77% yield after de-O-acetylation and O-benzylation in one step.[39] Removal of the PMB group in compound **9** using 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) in a biphasic reaction condition[40] furnished disaccharide acceptor **10** in 72% yield. Stereoselective glycosylation of compound **10** with 2-azido-2-deoxy-D-glucosyl thioglycoside **5** in the presence of a combination[38] of NIS and $HClO_4/SiO_2$ in a mixed solvent (CH_2Cl_2/Et_2O; 1:3) furnished trisaccharide derivative **11** in 74% yield, which was confirmed from the NMR spectral analysis. Sodium-methoxide-mediated trans-esterification[41] of compound **11** furnished the trisaccharide acceptor **12** in 92% yield. Iodonium-ion-mediated stereoselective glycosylation of compound **12** with L-rhamnosyl thioglycoside **6** in the presence of a combination[38] of NIS and $HClO_4/SiO_2$ led to the exclusive formation of the tetrasaccharide derivative **13** in 86% yield. NMR spectral analysis of compound **13** unambiguously confirmed its formation. Further trans-esterification of compound **13** using sodium methoxide[41] gave the tetrasaccharide acceptor **14** in 90% yield. Iodonium-ion-mediated stereoselective glycosylation of compound **14** with the L-rhamnosyl thioglycoside **4** in the presence of a combination[38] of NIS and $HClO_4/SiO_2$, followed by in situ removal of the PMB group[32,42] by increasing the reaction temperature, furnished the pentasaccharide acceptor **15** in 72% yield. The formation of compound **15** was unambiguously supported by its NMR spectral analysis. The presence of an O-acetoxy group at C-2 position of the thioglycoside donor **4** directed the formation of exclusively 1,2-*trans* glycosylation product by the neighboring group participation. Repetition of the stereoselective glycosylation of compound **15** with compound **4** followed by the in situ removal of PMB group in one pot[32,42] in the presence of a combination[38] of NIS and $HClO_4/SiO_2$ furnished hexasaccharide acceptor **16** in 70% yield. Finally, stereoselective glycosylation of compound **16** with D-glucosyl thioglycoside donor **7**, in the presence of a combination[38] of NIS and $HClO_4/SiO_2$, exclusively furnished heptasaccharide derivative **17** in 73% yield. NMR spectral analysis of compound **17** supported its formation: signals at $\delta = 5.50$ (br s, H-1_B), 5.43 (d, $J = 3.5$ Hz, H-1_A), 5.17 (br s, H-1_D), 5.12 (d, $J = 8.0$ Hz, H-1_G), 5.11 (br s, H-1_E), 5.10 (br s, H-1_F), 4.90 ppm (d, $J = 3.5$ Hz, H-1_C) in the ^{1}H NMR and $\delta = 101.8$ (PhCH), 101.4 (2 C, C-1_G, PhCH), 99.2 (2 C, C-1_E, C-1_F), 99.1 (C-1_D), 98.5 (C-1_B), 97.4 (C-1_A), 93.5 ppm (C-1_C) in the ^{13}C NMR spectra. The stereochemical configurations of the glycosidic linkages in compound **17** were further confirmed from the gated ^{1}H-coupled ^{13}C NMR spectrum. The coupling constants between C-1 and H-1 ($J_{C1/H1}$) of the anomeric centers of the monosaccharide units unambiguously confirmed the stereochemistry of the glycosidic linkages: $J_{C-1A/H-1A} = 172.0$ Hz (C-1_A, α-D-Gal*p*), $J_{C-1B/H-1B} = 174.0$ Hz (C-1_B, α-L-Rha*p*), $J_{C-1D/H-1D} = 174.0$ Hz (C-1_D, α-L-Rha*p*), $J_{C-1E/H-1E} = 174.0$ Hz (C-1_E, α-L-Rha*p*), $J_{C-1F/H-1F} = 174.0$ Hz (C-1_F, α-L-Rha*p*), $J_{C-1C/H-1C} = 172.0$ Hz (C-1_C, α-D-Glc*p*N), $J_{C-1G/H-1G} = 164.0$ Hz (C-1_G, β-D-Glc*p*)].[43] Compound **17** was subjected to a series of functional-group transformations involving (a) conversion of azido group to acetamido group by the treatment with thioacetic acid in pyridine,[44] (b) removal of benzylidene acetal using 80% aq. acetic acid, (c) acetylation of the hydroxy groups using acetic anhydride and pyridine, (d) removal of the benzyl groups under a catalytic transfer hydrogenation condition using triethylsilane in the presence of Pearlman's catalyst [20% $Pd(OH)_2/C$],[45] (e) 2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO)-mediated selective oxidation of the primary hydroxyl group at the D-galactosyl moiety using $NaOCl/NaClO_2$ in a biphasic reaction condition,[46] and (f) saponification of benzoyl and acetyl groups using sodium methoxide[41] to give the heptasaccharide **1** as its *p*-methoxyphenyl glycoside in overall 44% yield. The structure of compound **1** has been confirmed by its NMR spectral analysis (Scheme 2).

In summary, the heptasaccharide repeating unit of the cell wall polysaccharide of *E. coli* O139 has been synthesized in an efficient manner with satisfactory yield. A number of one-pot reaction conditions have been applied during the synthesis for the glycosylation and in-situ removal of the PMB group. Similar reaction conditions for the glycosylations have been used during the synthesis by the judicious functionalization of the glycosyl donor and acceptors. Most of the glycosylation steps gave high yields with satisfactory stereoselectivity.

Experimental Section

For details of the experimental conditions and characterization data for the compounds reported, see the Supporting Information.

Acknowledgements

T. G. thanks the Council of Scientific and Industrial Research (CSIR), India for providing a Senior Research Fellowship. This work was supported by the CSIR, New Delhi, India (AKM; Project No. 02(0038)/11/EMR-II).

Keywords: carbohydrates · glycosylation · oligosaccharides · polysaccharides · stereoselectivity

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Received: June 18, 2015
Published online on August 12, 2015

