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Syntheses of branched-chain sugars with push-pull functionality *

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ABSTRACT

The reaction of methyl 4,6-*O*-benzylidene-3(2)-deoxy-α-D-*erythro*-hexopyranosid-2(3)-ulose with carbon disulfide, alkyl iodide, and sodium hydride gave methyl 4,6-*O*-benzylidene-3(2)-[bis(alkylthio)methylene]-3(2)-deoxy-α-D-*erythro*-hexopyranosid-2(3)-uloses. Methyl 4,6-*O*-benzylidene-2-[bis(methylthio)-methylene]-2-deoxy-α-D-*erythro*-hexopyranosid-3-ulose (**5**) reacted with aromatic amines to give, in a rearrangement process, *N*-aryl-2-aryliminomethyl-4,6-*O*-benzylidene-2-deoxy-α-D-*erythro*-hex-1-enopyranosylamin-3-uloses. The reaction of **5** with hydrazine hydrate afforded 5-methylthio-(methyl 4,6-*O*-benzylidene-2,3-dideoxy-α-D-*erythro*-hexopyranosido)[3,2-*c*]pyrazole.

INTRODUCTION

Branched-chain monosaccharides have been found in many antibiotics[1]. Syntheses of these compounds and their analogues have mainly involved uloses, oxiranes, and Michael addition of carbanions to unsaturated sugars. Carbanions of deoxy sugars also react with carbonyl compounds to give branched-chain sugars[2]. L-Vancosamine[3] (3-amino-2,3,6-trideoxy-3-*C*-methyl-L-*lyxo*-hexose), 3-amino-2,3,6-trideoxy-3-*C*-methyl-L-*xylo*-hexose[4], and garosamine[5] (3-deoxy-4-*C*-methyl-3-methylamino-L-arabinose) have biological importance, and analogues of such monosaccharides have been described[6].

Following investigations on the formation of C–C bonds by reaction of carbanions of monosaccharides with carbon disulfide[7], we now describe a new synthesis of branched-chain sugars with push-pull functionality[8,9]. Such compounds may serve as precursors for the synthesis of sugars with one-carbon branches and with an

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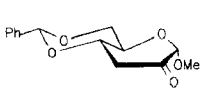
amino group at the branchpoint. Few examples of such branched-chain sugars are known[10–12].

RESULTS AND DISCUSSION

α-Oxoketene dithioacetals can be prepared[13–15] by treating acyclic or cyclic ketones with carbon disulfide and an alkylating reagent in the presence of sodium alkoxide. In this type of reaction of methyl 4,6-*O*-benzylidene-3-deoxy-α-D-*erythro*-hexopyranosid-2-ulose[16] (**1**) with carbon disulfide and an alkyl halide, the generation of the carbanion was best achieved by treatment with sodium hydride in *N*,*N*-dimethylformamide, and **2** (52%) and **3** (64%) were isolated as crystals when methyl iodide and ethyl iodide, respectively, were used. When tetrahydrofuran was used as the solvent[7], partial decomposition of the products occurred.



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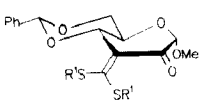
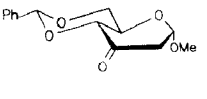


2 R^1 = Me
3 R^1 = Et



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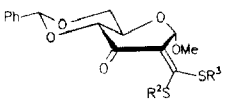
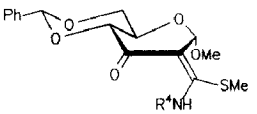


5 $R^2 = R^3$ = Me
6 $R^2 = R^3$ = Et
7 R^2, R^3 = CH_2CH_2

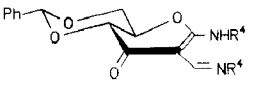
Likewise, methyl 4,6-*O*-benzylidene-2-deoxy-α-D-*erythro*-hexopyranosid-3-ulose[17] (**4**) reacted with sodium hydride, carbon disulfide, and methyl iodide, ethyl iodide, or 1,2-dibromoethane to give the crystalline bis(alkylthio)methylene derivatives **5** (49%), **6** (46%), and **7** (53%), respectively.

The presence of the bis(alkylthio)methylene or 1,3-dithiolan-2-ylidene group, respectively, in **2**, **3**, and **5**–**7** was indicated by the ^{1}H-NMR data for the region of 1.23–3.64 ppm (see Experimental). The ^{13}C signals for the methylthio groups in **2** and **5** were found at ~20 ppm. In accordance with the typical push-pull behaviour[18,19], the resonances of C-3 and C-2 in **2** and **5**, respectively, were shifted upfield (**2**, 127.3 ppm; **5**, 131.9 ppm). On the other hand, the signals for the corresponding exocyclic C-1′ in each compound showed the expected downfield shift (**2**, 168.9 ppm; **5**, 161.6 ppm).

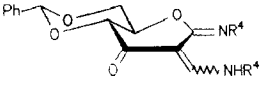
The push-pull functionality of **5** should allow reactions with *N*-nucleophiles. Simple α-oxoketene dithioacetals normally react with amines with displacement of one alkylthio group[15]. When **5** was treated with an excess of *p*-toluidine and *p*-anisidine in boiling ethanol, methanethiol was evolved and the products were not the expected ketene-*N*,*S*-acetals **8** and **9** but the crystalline branched-chain sugars **10** (84%) and **11** (82%), respectively.



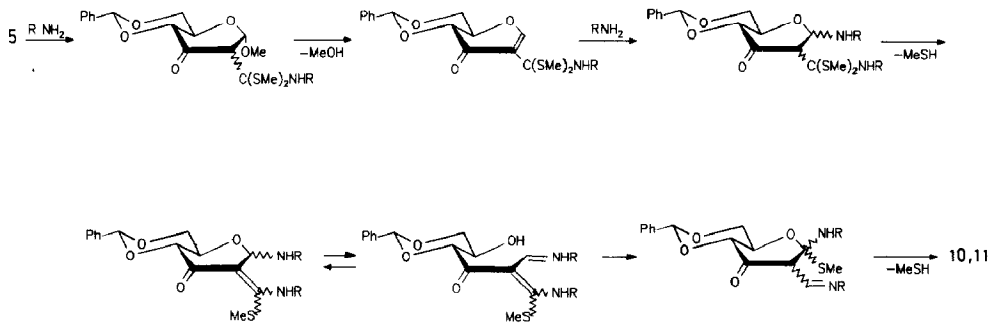
8 R^4 = *p*-MeC_6H_4
9 R^4 = *p*-$MeOC_6H_4$



10 R^4 = *p*-MeC_6H_4
11 R^4 = *p*-$MeOC_6H_4$



12 R^4 = *p*-MeC_6H_4
13 R^4 = *p*-$MeOC_6H_4$

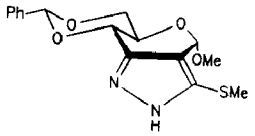


Scheme 1

The structures of **10** and **11** were determined from their NMR spectra (see Experimental). The broadened NH signals confirmed the presence of the branched-chain imino sugars. The absence of IR bands for NH of **10** and **11** indicated the strong intramolecular hydrogen-bonding. The data do not allow to exclude the tautomeric forms **12** and **13**.

One possible route for the formation of **10** and **11** is shown in Scheme 1.

Treatment of **5** with hydrazine hydrate afforded the heterocyclic derivative **14**, the mass spectrum of which contained a signal for M^+ at *m/z* 334. The $^3J_{H,H}$ value of 9.1 Hz confirmed the *trans* fusion of the 1,3-dioxane and pyran rings. The ^{1}H-NMR spectrum contained only one broad NH signal, as expected.



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EXPERIMENTAL

General methods.—Melting points were determined with a Boetius apparatus and are corrected. IR spectra were recorded with a Carl Zeiss Jena Model UR 20 spectrometer. Mass spectra were obtained with a Varian-MAT 311 A spectrometer. NMR spectra ($CDCl_3$, internal Me_4Si) were recorded with a Bruker WM 300 or AC 300 spectrometer (^{1}H, 300 MHz; ^{13}C, 75.5 MHz). The ^{13}C-NMR spectra were determined by the gated decoupling method, and 2D experiments and NOE measurements were performed by means of a Bruker program AC 300. Optical rotations were measured on solutions in $CHCl_3$ or *N,N*-dimethylformamide with a Carl Zeiss Jena Polamat A polarimeter. Column chromatography was performed on Silica Gel 60 (Merck) and TLC on Silica Gel 60 F_{254} (Merck) with detection by charring with H_2SO_4.

*Methyl 4,6-O-benzylidene-3-[bis(methylthio)methylene]-3-deoxy-α-*D*-erythro-hexopyranosid-2-ulose* (**2**).—Sodium hydride (0.9 g, 55–60% dispersion in oil, ~ 20 mmol) was added under argon to hexane (10 mL), the mixture was stirred for a few min at 20° and then allowed to settle, and the hexane was decanted. The procedure was repeated with benzene (10 mL). A solution of methyl 4,6-*O*-benzylidene-3-deoxy-α-D-*erythro*-hexopyranosid-2-ulose[16] (**1**; 2.64 g, 10 mmol), CS_2 (1.2 mL, 20 mmol), and MeI (4 mL, 64 mmol) in *N*,*N*-dimethylformamide (60 mL) was added with stirring to the NaH. The mixture was stirred at 20° for 1 h, then poured into ice–water (250 g). The yellow precipitate was collected, washed with water, and recrystallised from MeOH to give yellow needles of **2** (1.92 g, 52%), mp 119–122°, $[\alpha]_D^{20}$ + 12° (*c* 1.0, $CHCl_3$); ν_{max}^{KBr} 1690 cm^{-1} (C=O). NMR data: ^1H, δ 2.41 (s, 3 H, MeS), 2.47 (s, 3 H, MeS), 3.50 (s, 3 H, MeO), 3.90 (t, 1 H, $J_{5,6ax}$ 10.2 Hz, H-6*ax*), 4.19 (m, 1 H, H-5), 4.39 (dd, 1 H, $J_{5,6eq}$ 4.6, $J_{6ax,6eq}$ 10.2 Hz, H-6*eq*), 4.69 (s, 1 H, H-1), 4.73 (d, 1 H, $J_{4,5}$ 9.7 Hz, H-4), 5.70 (s, 1 H, C*H*Ph), 7.25–7.50 (m, 5 H, Ph); ^{13}C, δ 19.4 (MeS), 19.8 (MeS), 56.0 (MeO), 64.3 (C-5), 69.0 (C-6), 77.6 (C-4), 99.8 (C-1), 100.9 (*C*HPh), 126.0, 128.0, 128.8, 137.1 (Ph), 127.3 (C-3), 168.9 (C-1′), 186.4 (C-2). Mass spectrum: *m/z* 368 (M^+).

Anal. Calcd for $C_{17}H_{20}O_5S_2$ (368.4): C, 55.4; H, 5.5; S, 17.4. Found: C, 55.2; H, 5.2; S, 16.9.

*Methyl 4,6-O-benzylidene-3-[bis(ethylthio)methylene]-3-deoxy-α-*D*-erythro-hexopyranosid-2-ulose* (**3**).—By the above procedure, **1** (2.64 g, 10 mmol) was treated with NaH (0.9 g, 55–60% dispersion in oil, ~ 20 mmol), CS_2 (1.2 mL, 20 mmol), and EtI (9.36 g, 60 mmol) in *N*,*N*-dimethylformamide (60 mL) to give yellow needles of **3** (2.54 g, 64%), mp 115–117°, $[\alpha]_D^{20}$ −33° (*c* 1.01, $CHCl_3$); ν_{max}^{Nujol} 1685 cm^{-1} (C=O). ^1H-NMR data: δ 1.89 (t, 6 H, 2 CH_3CH_2), 2.95 (q, 4 H, 2 CH_3CH_2), 3.47 (s, 3 H, MeO), 3.96 (t, 1 H, H-6*ax*), 4.17 (m, 1 H, H-5), 4.40 (dd, 1 H, H-6*eq*), 4.67 (s, 1 H, H-1), 4.76 (d, 1 H, H-4), 5.69 (s, 1 H, C*H*Ph), 7.25–7.60 (m, 5 H, Ph).

Anal. Calcd for $C_{19}H_{24}O_5S_2$ (396.5): C, 57.5; H, 6.1; S, 16.2. Found: C, 57.7; H, 6.1; S, 16.1.

*Methyl 4,6-O-benzylidene-2-[bis(methylthio)methylene]-2-deoxy-α-*D*-erythro-hexopyranosid-3-ulose* (**5**).—Treatment of methyl 4,6-*O*-benzylidene-2-deoxy-α-D-*erythro*-hexopyranosid-3-ulose[17] (**4**; 2.64 g, 10 mmol), as described above for the preparation of **2**, gave yellow needles of **5** (1.81 g, 49%), mp 196–198°, $[\alpha]_D^{20}$ +42° (*c* 1.02, $CHCl_3$); ν_{max}^{Nujol} 1680 cm^{-1} (C=O). NMR data: ^1H, δ 2.42 (s, 3 H, MeS), 2.45 (s, 3 H, MeS), 3.45 (s, 3 H, MeO), 3.83 (m, 1 H, $J_{4,6ax}$ 3.0, $J_{5,6ax}$ 9.7, $J_{6ax,6eq}$ 9.7 Hz, H-6*ax*), 4.15 (dd, 1 H, $J_{4,5}$ 10.0 Hz, H-4), 4.32 (m, 1 H, $J_{5,6eq}$ 5.0 Hz, H-5), 4.36 (dd, 1 H, H-6*eq*), 5.57 (s, 1 H, C*H*Ph), 5.77 (s, 1 H, H-1), 7.30–7.60 (m, 5 H, Ph); ^{13}C, δ 18.9 (MeS), 19.2 (MeS), 54.9 (MeO), 63.1 (C-5), 69.5 (C-6), 82.2 (C-4), 101.5 (C-1), 102.3 (*C*HPh), 126.6, 128.3, 129.3, 136.9 (Ph), 131.9 (C-2), 161.6 (C-1′), 187.9 (C-3). Mass spectrum: *m/z* 368 (M^+).

Anal. Calcd for $C_{17}H_{20}O_5S_2$ (368.4): C, 55.4; H, 5.5; S, 17.4. Found: C, 55.5; H, 5.4; S, 17.5.

*Methyl 4,6-O-benzylidene-2-[bis(ethylthio)methylene]-2-deoxy-α-*D-erythro-*hexopyranosid-3-ulose* (**6**).—Treatment of **2** (2.64 g, 10 mmol), as described for the preparation of **3**, gave yellow needles of **6** (1.82 g, 46%), mp 174–176°, $[\alpha]_D^{20}$ −69.5° (*c* 1.1, $CHCl_3$). ^{1}H-NMR data: δ 1.23 (t, 6 H, 2 CH_3CH_2), 2.91 (q, 4 H, 2 CH_3CH_2), 3.40 (s, 3 H, MeO), 3.78 (t, 1 H, H-6*ax*), 4.19 (d, 1 H, H-4), 4.30 (m, 1 H, H-5), 4.39 (dd, 1 H, H-6*eq*), 5.54 (s, 1 H, CHPh), 5.81 (s, 1 H, H-1), 7.20–7.60 (m, 5 H, Ph).

Anal. Calcd for $C_{19}H_{24}O_5S_2$ (396.5): C, 57.5; H, 6.1; S, 16.2. Found: C, 57.5; H, 6.1; S, 16.0.

*Methyl 4,6-O-benzylidene-2-deoxy-2-(1,3-dithiolan-2-ylidene)-α-*D-erythro-*hexopyranosid-3-ulose* (**7**).—A suspension of NaH (0.45 g, 55–60% dispersion in oil, ~ 10 mmol), prepared as described above for the synthesis of **2**, **4** (1.32 g, 5 mmol), and CS_2 (0.6 mL, 10 mmol) in *N*,*N*-dimethylformamide (30 mL) was stirred at 20° for 10 min. 1,2-Dibromoethane (0.94 g, 5 mmol) was added, stirring was continued for 30 min, water (100 mL) was added gradually, and the yellow precipitate was collected, washed with water, and recrystallised from *N*,*N*-dimethylformamide–EtOH to give yellow needles of **7** (1.94 g, 53%), mp 260–265°, $[\alpha]_D^{24}$ +162° (*c* 0.4, *N*,*N*-dimethylformamide); ν_{max}^{Nujol} 1755 cm^{-1} (C=O). ^{1}H-NMR data: δ 3.46 (s, 3 H, MeO), 3.64 (m, 4 H, CH_2CH_2), 3.92 (t, 1 H, $J_{5,6ax}$ 10.0, $J_{6ax,6eq}$ 10.0 Hz, H-6*ax*), 4.15 (m, 1 H, $J_{4,5}$ 10.2, $J_{5,6eq}$ 5.0 Hz, H-5), 4.31 (d, 1 H, H-4), 4.39 (dd, 1 H, H-6*eq*), 5.42 (s, 1 H, H-1), 5.76 (s, 1 H, CHPh), 7.40–7.55 (m, 5 H, Ph). Mass spectrum: *m*/*z* 366 (M^+).

Anal. Calcd for $C_{17}H_{18}O_5S_2$ (366.4): C, 55.7; H, 4.9; S, 17.5. Found: C, 55.7; H, 4.9; S, 17.0.

*4,6-O-Benzylidene-2-deoxy-*N-p-*tolyl-2-(*p-*tolyliminomethyl)-α-*D-erythro-*hex-1-enopyranosylamin-3-ulose* (**10**).—A solution of **5** (0.74 g, 2 mmol) and *p*-toluidine (0.54 g, 5 mmol) in aq 95% EtOH (100 mL) was boiled under reflux for 8 h, then cooled. The yellow precipitate was collected and recrystallised from EtOH to give yellow needles of **10** (0.76 g, 84%), mp 212–216°, $[\alpha]_D^{23}$ −18° (*c* 1.0, $CHCl_3$). NMR data: ^{1}H, δ 2.34 (s, 3 H, $CH_3C_6H_4$), 2.36 (s, 3 H, $CH_3C_6H_4$), 3.90 (t, 1 H, $J_{5,6ax}$ 10.8, $J_{6ax,6eq}$ 9.6 Hz, H-6*ax*), 4.34 (m, 1 H, H-5), 4.38 (d, 1 H, $J_{4,5}$ 10.3 Hz, H-4), 4.43 (dd, 1 H, $J_{5,6eq}$ 4.6 Hz, H-6*eq*), 5.59 (s, 1 H, CHPh), 7.05–7.20 (m, 8 H, 2 $CH_3C_6H_4$), 7.30–7.60 (m, 5 H, Ph), 8.71 (s, 1 H, CH=N), 13.45 (b, 1 H, NH); ^{13}C, δ 20.9 ($CH_3C_6H_4$), 21.0 ($CH_3C_6H_4$), 68.4 (C-6), 68.8 (C-5), 77.4 (C-4), 97.0 (C-2), 102.3 (CHPh), 118.0, 123.5, 126.5, 128.3, 129.3, 130.4, 133.8, 135.9, 136.5, 137.0, 141.4 (2 $CH_3C_6H_4$ and Ph), 147.8 (C-1′), 155.5 (C-1), 186.0 (C-3). Mass spectrum: *m*/*z* 454 (M^+).

Anal. Calcd for $C_{28}H_{26}N_2O_4$ (454.5): C, 74.0; H, 5.8; N, 6.2. Found: C, 74.1; H, 5.6; N, 6.5.

*4,6-O-Benzylidene-2-deoxy-*N-p-*methoxyphenyl-2-(*p-*methoxyphenyliminomethyl)-α-*D-erythro-*hex-1-enopyranosylamin-3-ulose* (**11**).—Reaction of **5** (0.74 g, 2 mmol) and *p*-anisidine (0.62 g, 5 mmol), as described above, gave yellow needles of **11** (0.80 g, 82%), mp 188–191°, $[\alpha]_D^{23}$ −17° (*c* 1.0, $CHCl_3$). NMR data: ^{1}H, δ 3.78 (s, 3

H, $CH_3OC_6H_4$), 3.80 (s, 3 H, $CH_3OC_6H_4$), 3.90 (m, 1 H, $J_{4,6ax}$ 1.4, $J_{5,6ax}$ 9.9, $J_{6ax,6eq}$ 9.9 Hz, H-6*ax*), 4.33 (m, H-4,5), 4.42 (dd, 1 H, $J_{5,6eq}$ 4.2 Hz, H-6*eq*), 5.56 (s, 1 H, CHPh), 6.86–6.92 (m, 4 H, $CH_3C_6H_4$), 7.09–7.25 (m, 4 H, $CH_3OC_6H_4$), 7.30–7.60 (m, 5 H, Ph), 8.61 (s, 1 H, CH=N), 13.50 (b, 1 H, NH); ^{13}C, δ 55.4 ($CH_3OC_6H_4$), 55.5 ($CH_3OC_6H_4$), 68.3 (C-6), 68.9 (C-5), 77.2 (C-4), 96.7 (C-2), 102.2 (CHPh), 114.0, 114.9, 119.4, 124.9, 126.5, 128.2, 129.3, 132.9, 136.5, 136.9, 155.4, 156.4, (2 $CH_3OC_6H_4$ and Ph), 147.7 (C-1′), 157.7 (C-1), 185.8 (C-3). Mass spectrum: *m/z* 486 (M^+).

Anal. Calcd for $C_{28}H_{26}N_2O_6$ (486.5): C, 69.1; H, 5.4; N, 5.8. Found: C, 68.7; H, 5.9; N, 5.5.

*5-Methylthio-(methyl 4,6-O-benzylidene-2,3-dideoxy-α-*D-erythro-*hexopyranosido)[3,2-c]pyrazole* (**14**).—A solution of **5** (0.74 g, 2 mmol) and hydrazine hydrate (0.15 g, 3 mmol) in EtOH (10 mL) was boiled under reflux for 3 h, then cooled to room temperature, water (200 mL) was gradually added, and the precipitate was collected and washed with water. Column chromatography (benzene–EtOAc, 9:1) of this product and recrystallisation from EtOAc–ether–hexane (4:3:1) gave **14** (0.24 g, 36%), mp 190–193°, $[\alpha]_D^{26}$ +50° (*c* 0.2, $CHCl_3$). NMR data: ^{1}H, δ 2.40 (s, 3 H, MeS), 3.54 (s, 3 H, MeO), 3.91 (t, 1 H, $J_{5,6ax}$ 10.2 Hz, H-6*ax*), 4.08 (m, 1 H, H-5), 4.36 (dd, 1 H, $J_{5,6eq}$ 4.5, $J_{6ax,6eq}$ 10.2 Hz, H-6*eq*), 4.67 (d, 1 H, $J_{4,5}$ 9.1 Hz, H-4), 5.48 (s, 1 H, H-1), 5.53 (s, 1 H, CHPh), 7.30–7.50 (m, 5 H, Ph), 11.5 (b, 1 H, NH); ^{13}C, δ 16.8 (MeS), 55.7 (MeO), 63.9 (C-5), 69.3 (C-6), 74.1 (C-4), 96.1 (C-1), 101.9 (CHPh), 116.0 (C-2), 126.3, 128.2, 129.2, 137.0 (Ph), 139.4, 143.1 (C-3,C-5′). Mass spectrum *m/z* 334 (M^+).

Anal. Calcd for $C_{16}H_{18}N_2O_4S$ (334.4): C, 57.5; H, 5.4; N, 8.4. Found: C, 57.3; H, 5.6; N, 8.4.

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