Supporting Information for:

The First Total Synthesis of a Highly Branched Arabinofuranosyl Hexasaccharide Found at the Nonreducing Termini of Mycobacterial Arabinogalactan and Lipoarabinomannan

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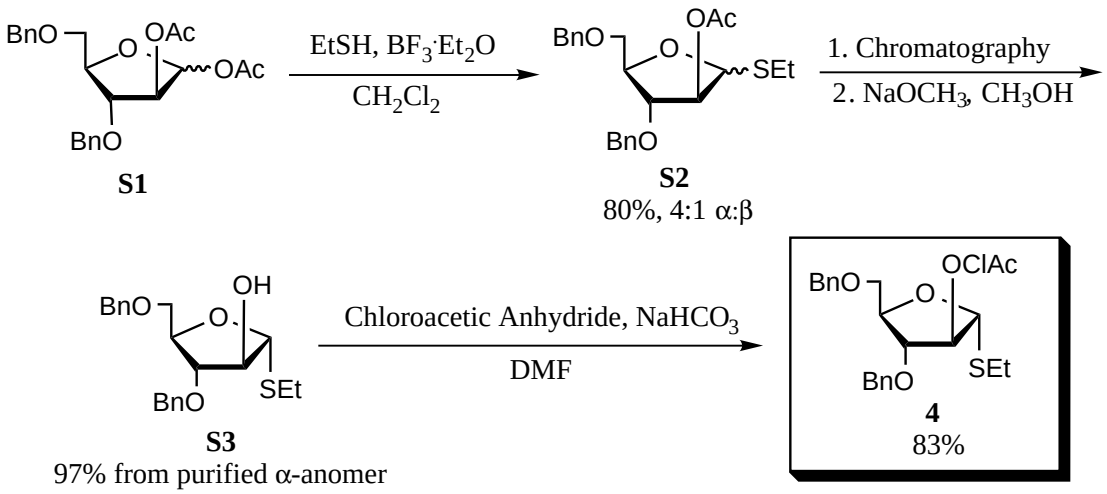
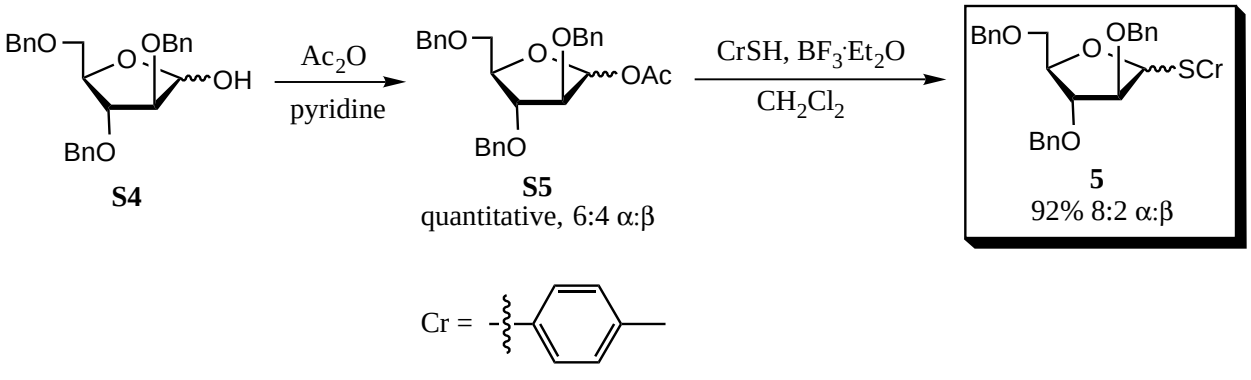
Scheme S1. Synthesis of **4** and **5**.





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Experimental

General Methods. Solvents were distilled from the appropriate drying agents before use. Unless stated otherwise all reactions were carried out at room temperature and under a positive pressure of argon and were monitored by TLC on silica gel 60 F_{254} (0.25 mm, E. Merck). Spots were detected under UV light or by charring with 10% H_2SO_4 in EtOH. Solvents were evaporated under reduced pressure and below 40 ˚C (bath). Solutions of crude products were dried over anhydrous Na_2SO_4. Column chromatography was performed on silica gel 60 (40-60 μM). The ratio between silica gel and crude product ranged from 100 to 50:1 (w/w). Optical rotations were measured at 22±2 ˚C. ^{1}H NMR spectra were recorded at 400 or 500 MHz, and chemical shifts are referenced to either to TMS (0.0, $CDCl_3$) or HOD (4.78, D_2O). ^{13}C NMR spectra were recorded at 100 or 125 MHz and ^{13}C chemical shifts are referenced to internal $CDCl_3$ (77.00, $CDCl_3$) or external dioxane $CDCl_3$ (67.40, D_2O). Elemental analyses were performed by Atlantic Microlab Inc., Norcross, GA. MALDI mass spectra were recorded on samples in an α-cyano-4-hydroxycinnamic acid matrix. ESI mass spectra were recorded on samples dissolved in CH_3OH/H_2O, 3:1.

Ethyl 2-O-*acetyl-3,5-di*-O-*benzyl-1-thio-D-arabinofuranoside* (**S2**)**.** To a solution of **S1**[1] (3.80 g, 9.17 mmol) in dry CH_2Cl_2 (10 mL) at 0 °C was added dropwise ethanethiol (0.75 mL, 10.13 mmol). After 10 min of stirring, boron trifluoride etherate (1.39 mL, 10.97 mmol) was added dropwise. After 2 h of stirring, Et_3N (3 mL) was added and the reaction mixture was concentrated. The residue was then purified by column chromatography on silica gel (hexane/EtOAc, 5:1) to provide the product **S2** (3.07 g, 80%) as two separable anomers in a 4:1 α:β ratio. **α-Isomer**: R_f 0.37 (hexane/EtOAc, 4:1); $[\alpha]_D$ + 170 ^{o} (*c* 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 7.34–7.26 (m, 10 H), 5.35 (s, 1 H), 5.13 (t, 1 H, *J* = 1.6 Hz), 4.72 (d, 1 H, *J* = 12.2 Hz), 4.56 (d, 1 H, *J* = 12.2 Hz), 4.54 (d, 1 H, *J* = 12.2 Hz), 4.49 (d, 1 H, *J* = 12.0 Hz), 4.39 (m, 1 H), 3.92 (dd, 1 H, *J* = 1.1, 4.6 Hz), 3.65 (dd, 1 H, *J* = 3.7, 10.9 Hz), 3.59 (dd, 1 H, *J* = 4.7, 10.8 Hz), 2.72 (m, 1 H), 2.65 (m, 1 H), 2.00 (s, 3 H), 1.30 (t, 3 H, *J* = 7.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$, δ) 169.93, 138.00, 137.61, 128.38, 128.34, 127.92, 127.82, 127.71, 127.66, 87.80, 83.25, 82.34, 81.52, 73.41, 72.21, 68.88, 25.17, 20.91, 14.74. Anal. Calcd for $C_{23}H_{28}O_5S$: C, 66.32; H, 6.78. Found: C, 66.35; H, 6.74. **β-Isomer**: R_f 0.43 (hexane/EtOAc, 4:1); $[\alpha]_D$ - 48 ^{o} (*c* 1.2, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 7.33–7.24 (m, 10 H), 5.36 (d, 1 H, *J* = 4.4 Hz), 5.31 (dd 1 H, *J* = 2.6, 4.4 Hz), 4.69 (d, 1 H, *J* = 12.1 Hz), 4.60 (d, 1 H, *J* = 12.1 Hz), 4.57 (d, 1 H, *J* = 12.1 Hz), 4.52 (d, 1 H, *J* = 12.1 Hz), 4.12 (m, 1 H), 3.97 (dd, 1 H, *J* = 2.7, 3.8 Hz), 3.66 (dd, 1 H, *J* = 6.0, 10.3 Hz), 3.57 (dd, 1 H, *J* = 6.3, 10.1 Hz), 2.68 (m, 2 H), 2.00 (s, 3 H), 1.27 (t, 3 H, *J* = 7.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$, δ) 169.96, 138.02, 137.53, 128.41, 128.34, 127.84, 127.76, 127.66, 85.85, 83.60, 82.44, 78.68, 73.33, 72.05,

70.31, 25.70, 20.69, 15.13. Anal. Calcd for $C_{23}H_{28}O_5S$: C, 66.32; H, 6.78. Found: C, 65.93; H, 6.71.

Ethyl 3,5-di-O-*benzyl-1-thio-α-D-arabinofuranoside* (**S3**)**.** To a solution of **S2** (α-isomer) (1.85 g, 4.44 mmol) in dry CH_3OH (10 mL) was added dropwise 0.1 M methanolic sodium methoxide (4 mL). After 2 h of stirring at room temperature, the reaction mixture was neutralized with Amberlite IR-120 (H^+) resin, filtered, and concentrated. The residue was purified by column chromatography on silica gel (hexane/EtOAc, 4:1) to give **S3** (1.61 g, 97%) as a syrup: R_f 0.28 (hexane/EtOAc, 4:1); $[\alpha]_D$ +199° (*c* 0.4, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 7.35–7.26 (m, 10 H), 5.28 (s, 1 H), 4.69 (d, 1 H, *J* = 12.1 Hz), 4.63 (d, 1 H, *J* = 11.9 Hz), 4.51 (d, 1 H, *J* = 12.4 Hz), 4.49 (d, 1 H, *J* = 12.3 Hz), 4.35 (m, 1 H), 4.20 (dt, 1 H, *J* = 1.2, 10.2 Hz), 3.91 (d, 1 H, *J* = 3.0 Hz), 3.69 (dd, 1 H, *J* = 2.4, 10.5 Hz), 3.55 (dd, 1 H, *J* = 2.4, 10.5 Hz), 3.54 (d, 1 H, *J* = 10.2 Hz), 2.75–2.63 (m, 2 H), 1.31 (t, 3 H, *J* = 7.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$, δ) 137.63, 137.07, 128.55, 128.44, 128.05, 127.85, 127.83, 91.71, 85.27, 82.74, 79.52, 73.75, 72.06, 69.66, 25.90, 15.04. Anal. Calcd for $C_{21}H_{26}O_4S$: C, 67.35; H, 7.00. Found: C, 67.45; H, 6.95.

Ethyl 3,5-di-O-*benzyl-2*-O-*chloroacetyl-1-thio-α-D-arabinofuranoside* (**4**). Alcohol **S3** (150 mg, 0.40 mmol) was dissolved in dry DMF (5 mL) and then $NaHCO_3$ (50 mg, 0.60 mmol) and chloroacetic anhydride (103 mg, 0.60 mmol) were added. The reaction mixture was stirred for 2 h at room temperature and was then diluted with CH_2Cl_2. The organic layer was washed with water, dried (Na_2SO_4), concentrated and the residue chromatographed (hexane/EtOAc, 6:1) to give **4** (150 mg, 83%) as a syrup: R_f 0.49 (hexane/EtOAc, 3:1); $[\alpha]_D$ + 155 ° (*c* 1.0, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 7.41–7.31 (m, 10 H), 5.44 (s, 1 H), 5.24 (t, 1 H, *J* = 1.2 Hz), 4.78 (d, 1 H, *J* = 12.2 Hz), 4.62 (d, 1 H, *J* = 12.2 Hz), 4.60 (d, 1 H, *J* = 11.9 Hz), 4.52 (d, 1 H, *J* = 12.0 Hz), 4.46 (m, 1 H), 4.02 (m, 1 H), 4.00 (d, 1 H, *J* = 14.8 Hz), 3.91 (d, 1 H, *J* = 14.8 Hz), 3.72 (dd, 1 H, *J* = 3.5, 10.9 Hz), 3.64 (dd, 1 H, *J* = 4.6, 10.9 Hz), 2.82–2.67 (m, 2 H), 1.36 (t, 3 H, *J* = 7.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$, δ) 166.81, 138.34, 137.87, 128.86, 128.81, 128.42, 128.35, 128.21, 87.82, 84.31, 83.32, 82.07, 73.93, 72.81, 69.10, 40.98, 25.66, 15.17. Anal. Calcd for $C_{23}H_{27}O_5SCl$: C, 61.26; H, 6.03. Found: C, 61.32; H, 5.99.

1-O-*acetyl- 2,3,5-tri*-O-*benzyl-1-D-arabinofuranose* (S**5**). To a solution of 2,3,5-tri-*O*-benzyl-D-arabinofuranose[2] (**S4**, 5.0 g, 11.9 mmol) in dry pyridine (10 mL) at 0 °C was added acetic anhydride (1.35 mL, 14.31 mmol) dropwise. A catalytic amount of DMAP was added and the reaction was stirred for 4 h at this temperature. The reaction was then diluted with CH_2Cl_2 and then washed with 5% HCl, a saturated solution of $NaHCO_3$ and then water. The organic layer was dried (Na_2SO_4), filtered and concentrated to give the product **S5** in quantitative yield as an oil. The product was used in the next step without further purification.

Cresyl 2,3,5-tri-O-*benzyl-1-thio-D-arabinofuranoside* (**5**). Acetate **S5** (1.64 g, 3.55 mmol) was dissolved in dry CH_2Cl_2 (10 mL) and the solution was cooled to 0 °C. *p*-Thiocresol (485 mg, 3.90 mmol) was added and after stirring for 10 min, boron trifluoride etherate (540 µL, 4.26 mmol) was added dropwise. The solution was stirred for 1 h at 0 °C, before Et_3N (590 µL, 4.23 mmol) was added after which point and the reaction mixture was diluted with CH_2Cl_2. After washing with a saturated solution of $NaHCO_3$ the organic layer was dried (Na_2SO_4) and concentrated to give a residue that was chromatographed (hexane/EtOAc, 8:1) to give **5** (8:2 α:β mixture) as an oil (1.73 g, 92%): R_f 0.27 (hexane/EtOAc, 9:1); $[\alpha]_D$ + 114 ° (*c* 6.8, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$, δ) 7.09–7.44 (m, 19 H), 5.60 (d, 0.2 H, *J* = 4.9 Hz), 5.53 (d, 0.8 H, *J* = 2.8 Hz), 4.36–4.71 (m, 7 H), 4.03–4.30 (m, 2 H), 3.61–3.82 (m, 2 H), 2.34 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$, δ) 138.10, 137.73, 137.37, 137.30, 131.96, 131.53, 130.91, 129.63, 128.39, 128.35, 128.33, 128.27, 127.91, 127.86, 127.76, 127.72, 127.70, 127.66, 127.52, 90.53, 90.28, 88.37, 84.25, 83.49, 83.36, 82.35, 80.41, 73.27, 72.42, 72.20, 72.04, 71.80, 71.03, 69.10, 21.57. Anal. Calcd for $C_{33}H_{34}O_4S$: C, 75.24; H, 6.73. Found: C, 75.20; H, 6.49.

Methyl 5-O-*[3,5-di*-O-*(3,5-di*-O-*benzyl-2*-O-*chloroacetyl-α-D-arabinofuranosyl)-2*-O-*benzoyl-α-D-arabinofuranosyl]-2,3-di*-O-*benzoyl-α-D-arabinofuranoside* (**8**). To a solution of disaccharide alcohol **7**[3] (533 mg, 0.88 mmol) and donor **4** (948 mg, 2.10 mmol) in dry CH_2Cl_2 (10 mL) was added powdered molecular sieves (4 Å, 1.0 g). The reaction mixture was stirred for 20 min at 0 °C and then *N*-iodosuccinimide (568 mg, 2.52 mmol) and silver triflate (162 mg, 0.63 mmol) were added. After stirring for 2 h at this temperature, Et_3N was added to quench the reaction. The reaction mixture was then diluted with CH_2Cl_2 and filtered through Celite. The filtrate was washed successively with a saturated solution of $Na_2S_2O_3$, water, and brine. The organic phase was dried (Na_2SO_4), filtered, and concentrated, and purified by chromatography on silica gel (hexane/EtOAc, 5:1→2:1) to give **8** (894 mg, 74%) as a syrup: R_f 0.36 (hexane/EtOAc, 2:1); $[\alpha]_D$ +68° (*c* 1.3, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 7.99–8.14 (m, 6 H), 7.06–7.60 (m, 29 H), 5.61 (d, 1 H, *J* = 4.9 Hz), 5.46 (d, 1 H, *J* = 1.0 Hz), 5.44 (s, 1 H, *J* = 1.1 Hz), 5.37 (s, 1 H), 5.34 (s, 1 H), 5.22 (s, 1 H), 5.15 (d, 1 H, *J* = 1.1 Hz), 5.11 (s, 1 H), 5.07 (d, 1 H, *J* = 1.0 Hz), 4.59 (d, 1 H, *J* = 12.3 Hz), 4.17–4.51 (m, 13 H), 4.00 (dd, 1 H, *J* = 4.1, 11.7 Hz), 3.89 (dd, 1 H, *J* = 2.6, 11.1 Hz), 3.73–3.88 (m, 6 H), 3.65 (d, 1 H, *J* = 15.0 Hz) 3.49–3.54 (m, 2 H), 3.44 (s, 3 H), 3.43 (dd, 1 H, *J* = 4.4, 10.9 Hz), 3.38 (dd, 1 H, *J* = 4.1, 11.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$, δ) 166.14, 165.92, 165.77, 165.52, 165.44, 138.00, 137.96, 137.66, 137.63, 133.45, 133.33, 133.30, 130.00, 129.93, 129.84, 128.58, 128.49, 128.45, 128.35, 128.30, 128.28, 128.21, 127.85, 127.79, 127.74, 127.71, 127.67, 106.72, 105.43, 105.37, 105.35, 82.87, 82.73, 82.72, 82.71, 82.66, 82.63, 82.58, 82.35, 82.17, 82.00, 81.95, 80.70,

77.56, 73.46, 73.38, 71.79, 68.95, 68.77, 65.44, 64.90, 54.87, 40.55, 40.45. HR–ESI-MS calcd for $[C_{74}H_{74}O_{22}Cl_2]Na^+$ 1407.394102, found 1407.42903.

Methyl 5-O-*[3,5-di*-O-*(3,5-di*-O-*benzyl-α-D-arabinofuranosyl)-2*-O-*benzoyl-α-D-arabinofuranosyl]-2,3-di*-O-*benzoyl-α-D-arabinofuranoside* (**9**). To a stirred solution of tetrasaccharide **8** (414 mg, 0.30 mmol) in dry CH_2Cl_2 (5 mL) and dry MeOH (2 mL) were added acetic acid (171 μl, 2.99 mmol) and hydrazine monohydrate (145 μl, 2.99 mmol). The mixture was stirred at 40 °C for 3 h and then cooled to room temperature before being concentrated. The residue was taken up in CH_2Cl_2 and washed with water. The organic phase was then dried (Na_2SO_4), filtered, and concentrated, and purified by chromatography on silica gel (hexane/EtOAc, 3:1→1:1) to give **9** (335 mg, 91%): R_f 0.45 (hexane/EtOAc, 1:1)); $[\alpha]_D$ +68° (*c* 0.9, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.18–8.03 (m, 6 H), 7.63–7.20 (m, 29 H), 5.61 (dd, 1 H, *J* = 0.6, 5.1 Hz), 5.50 (d, 1 H, *J* = 1.3 Hz), 5.46 (s, 1 H), 5.39 (s, 1 H), 5.23 (s, 1 H), 5.16 (s, 1 H), 5.15 (s, 1 H), 4.59 (d, 1 H, *J* = 12.0 Hz), 4.58 (d, 1 H, *J* = 12.0 Hz), 4.53 (d, 1 H, *J* = 12.0 Hz), 4.50 (d, 1 H, *J* = 12.4 Hz), 4.48–4.38 (m, 5 H), 4.32 (dd, 1 H, *J* = 2.8, 6.3 Hz), 4.25–4.22 (m, 2 H), 4.18–4.13 (m, 2 H), 4.03 (dd, 1 H, *J* = 3.4, 11.5 Hz), 3.94 (dd, 1 H, *J* = 3.0, 11.0 Hz), 3.86–3.81 (m, 3 H), 3.59 (dd, 1 H, *J* = 2.5, 10.5 Hz), 3.56 (dd, 1 H, *J* = 2.5, 10.6 Hz), 3.49 (s, 3 H), 3.44 (t, 1 H, *J* = 3.2 Hz), 3.42 (t, 1 H, *J* = 2.9 Hz), 3.29 (d, 1 H, *J* = 9.2 Hz), 3.10 (d, 1 H, *J* = 8.9 Hz); ^{13}C NMR (125 MHz, $CDCl_3$, δ) 164.84, 164.55, 164.39, 136.90, 136.89 136.31, 136.27, 132.54, 132.32, 132.21, 128.97, 128.92, 128.83, 128.53, 128.37, 128.19, 127.62, 127.48, 127.45, 127.41, 127.36, 127.28, 127.25, 126.90, 126.84, 126.74, 126.71, 126.60, 126.56, 126.52, 126.51, 107.65, 107.28, 105.77, 104.70, 83.93, 83.78, 82.08, 81.88, 81.83, 80.98, 80.96, 80.93, 79.37, 77.41, 77.29, 76.55, 72.60, 72.54, 70.62, 70.60, 68.75, 68.65, 64.69, 64.06, 53.87. HR-ESI-MS calcd for $[C_{70}H_{72}O_{20}]Na^+$ 1255.450918, found 1255.42789.

Methyl 5-O-*{3,5-di*-O-*(2*-O-*[2,3,5-tri*-O-*benzyl-β-D-arabinofuranosyl]-3,5-di*-O-*benzyl-α-D-arabinofuranosyl)-2*-O-*benzoyl-α-D-arabinofuranosyl}-2,3-di*-O-*benzoyl-α-D-arabinofuranoside* (**10**) Tetrasaccharide diol **9** (255 mg, 0.21 mmol) and thioglycoside **5** (436 mg, 0.83 mmol) were dissolved in dry CH_2Cl_2 (6 mL). The solution was stirred under N_2 for 30 min with activated, powdered molecular sieves (4 Å, 2.0 g) at -78 °C, and then *N*-iodosuccinimide (186 mg, 0.83 mmol) and silver triflate (64 mg, 0.25 mmol) were added. After being stirred for 90 min at this temperature, Et_3N was added to quench the reaction. The reaction mixture was diluted with CH_2Cl_2 and filtered through Celite. The filtrate was washed successively with $Na_2S_2O_3$, water, and saturated NaCl solution and dried (Na_2SO_4). After removal of the solvent, chromatography of the residue on silica gel (hexane/EtOAc, 3:1→2:1) gave **10** (343 mg, 81%), R_f 0.45 (hexane/EtOAc, 2:1); $[\alpha]_D$ +1° (*c* 0.9, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$, δ) 8.12–7.97 (m, 6

H), 7.58–7.15 (m, 59 H), 5.55 (dd, 1 H, *J* = 0.9, 5.2 Hz), 5.48 (d, 1 H, *J* = 1.5 Hz), 5.44 (d, 1 H, *J* = 1.4 Hz), 5.39 (s, 1 H), 5.36 (s, 1 H), 5.20 (d, 1 H, *J* = 4.0 Hz), 5.17–5.19 (m, 2 H), 5.16 (s, 1 H), 3.99-4.71 (m, 38 H), 3.95 (dd, 1 H, *J* = 3.1, 11.3 Hz), 3.86 (dd, 1 H, *J* = 1.6, 11.6 Hz), 3.48–3.60 (m, 7 H), 3.46 (s, 3 H), ^{13}C NMR (125 MHz, $CDCl_3$, δ) 165.70, 165.60, 165.53, 138.34, 138.30, 138.26, 138.24, 138.20, 138.16, 138.12, 138.10, 137.84, 137.71, 133.43, 133.27, 133.20, 129.96, 129.92, 129.82, 129.47, 129.40, 129.24, 128.55, 128.46, 128.43, 128.40, 128.35, 128.32, 128.30, 128.28, 128.26, 128.19, 128.16, 128.09, 127.97, 127.88, 127.69, 127.62, 127.59, 127.54, 127.52, 127.48, 127.41, 106.75, 106.63, 105.95, 105.84, 100.39, 100.01, 85.96, 85.37, 84.17, 83.97, 83.86, 83.80, 83.37, 83.17, 82.13, 82.04, 81.73, 81.50, 81.24, 81.08, 80.02, 79.89, 77.75, 73.28, 73.22, 73.05, 73.00, 72.34, 72.31, 72.26, 72.24, 71.97, 71.83, 69.92, 69.74, 66.05, 65.29, 54.91. MALDI-MS calcd for $[C_{122}H_{124}O_{28}]Na^+$ 2061.3, found 2061.2.

Methyl 5-O-{3,5-di-O-(2-O-[β-D-arabinofuranosyl]-α-D-arabinofuranosyl)-α-D-arabinofuranosyl}-α-D-arabinofuranoside **(3).** A solution of **10** (184 mg, 0.09 mmol) in dry CH_2Cl_2 (2 mL) and dry MeOH (6 mL) was treated with a catalytic amount of 0.1 M methanolic sodium methoxide. After 4 h of stirring at room temperature, the reaction mixture was neutralized with Amberlite IR-120 (H^+) resin, filtered, and concentrated and the resulting residue purified by chromatography on silica gel (hexane/EtOAc, 2:1→1:2). The residue was dissolved in $AcOH/H_2O$ (4:1, 5 mL) and hydrogenolyzed over 10% Pd/C (60 mg) for 3 h. The mixture was filtered through Celite, concentrated, and the residue was purified by chromatography on Iatrobeads (CH_2Cl_2:CH_3OH:H_2O, 60:35:5) to give **3** (64 mg, 86%): R_f 0.34 (CH_2Cl_2:CH_3OH:H_2O, 60:35:5); $[\alpha]_D$ +47° (*c* 0.6, H_2O); ^{1}H NMR (500 MHz, D_2O, δ) 5.21 (d, 1 H, *J* = 1.7 Hz), 5.14 (d, 1 H, *J* = 1.6 Hz), 5.11 (d, 1 H, *J* = 4.6 Hz), 5.10 (d, 1 H, *J* = 4.6 Hz), 5.08 (s, 1 H), 4.90 (d, 1 H, *J* = 1.7 Hz), 4.25–4.30 (m, 2 H), 3.97–4.18 (m, 14 H), 3.63–3.93 (m, 14 H), 3.39 (s, 3 H), ^{13}C NMR (125 MHz, D_2O, δ) 108.83, 107.86, 106.07, 105.90, 101.16, 101.05, 87.48, 87.24, 83.32, 83.24, 82.95, 82.51, 82.42, 82.41, 82.19, 81.09, 79.52, 76.86, 76.68, 76.67, 75.26, 75.14, 74.59, 74.55, 66.87, 66.79, 63.39, 63.36, 61.02, 61.00, 55.45. HR-ESI-MS calcd for $[C_{31}H_{52}O_{25}]Na^+$ 847.268993, found 847.25326.

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